Microheater

A microheater is a MEMS structure which has two terminals, marked for positive and ground terminals, and a heating element connected to the heater support. The microheater is grown on a glass (silicon) substrate. First a metallic structure is grown on the silicon substrate which would represent the heater terminal, support and element. The heater terminals are used for connecting the power to the heater. This part is connected to the external circuit, hence, the temperature of this terminal must be at room temperature. To keep the terminals cooled, the terminal base are made large compared to the complete structure. The large base made of Titanium, will help drive in higher current and similarly keep the structure cooled. This current must be conveyed to the heating element, and losses in the heater support element must be minimized. This condition requires that the resistance offered by the support element must be minimized, and it can be done if the support element offers lower resistivity and also higher cross-section area. This must also offer higher thermal conductivity to keep the support structure cooled and at much lower temperature than the heater element. Thus, the heater support is constructed with a gold layer deposited on titanium layer. Finally, the titanium heating element having a zig-zag structure is constructed. The structure is such that, the electrons encounter least mean free path which will cause more heating to occur throughout the element. With time, more and more collision will cause heating of the element. Also the Titanium heating element's conductivity decreases as temperature increases, which again increases the resistance of the element and compounds the heating effect. Therefore, the temperature of the heating element rises rapidly and faster than rest of the structure.

Metallization

Physical vapor deposition (PVD) is a process by which metal, insulator can be deposited on semiconductor without undergoing any chemical reaction. PVD is used for deposition of metal on the surface of a sample and is known as metallization. It is basically a process of transferring growth species from a source or target and deposit them on a substrate to form a film. Metallization is carried on in a high vacuum chamber so that mean free path of the metal molecules increased as compared to the source-to-substrate distance and they can easily reach the wafer without any collision with unwanted species and uniform coating of metal is deposited. Vacuum coating unit is employed to carry out the entire process step of metallization. The system consists of an evaporation source that vaporizes the desired material and a substrate is located at an appropriate distance facing the evaporation source. The source metal is placed in a boat which is chemically inert and the substrate are located in a vacuum chamber. The substrate can be heated or electrically biased. Heating is necessary to clean the substrate and to promote better adhesion of metal with the substrate. Generally the source metal is placed and then evaporated by resistive heating. In addition, to evaporation of source by resistive heating, sometimes electron beam evaporation is used to minimize the impurity contamination. For multiple wafers, planetary system can be used where each wafer is placed on the surface of the sphere, rotating about its own axis and evaporating source is located at the center. The transport of atoms or molecules from the source to the growth substrate is straight forward along the line of sight, and therefore, the conformal coverage is relatively poor and a uniform film over a large area is difficult to obtain. Metallization unit mainly consists of 3 units-

- Rotary Pump
- Diffusion Pump
- Vacuum chamber

Direct Drive Rotary Vane Vacuum Chamber

Rotary vane vacuum pumps are low and medium vacuum pumps and also can be used as a backing pump for dry vacuum pumps, turbo molecular pumps, diaphragm pumps. Rotary vacuum pumps are extensively used in air conditioning and refrigeration, oil and gas industries, electrical industries, coating and deposition, vacuum furnaces, leak detection.

Operating principle:

Rotary pump is connected with the main vacuum chamber through roughing line, there is a valve called roughing valve in between this line and the vacuum chamber. Rotary pump is also connected to diffusion pump through leaking line. Baking valve is placed between diffusion pump and rotary pump. Use of this pump can attend the pressure up to 10^{-3} Torr. After that diffusion pump starts working and decreases the pressure up to 10^{-6} Torr.

Diffusion Pump

Diffusion pumps are designed and manufactured to suit high vacuum needs of both industrial and laboratory application. The superior pumping speed of diffusion pumps make them exclusively suitable for vacuum furnace, space, research, vacuum coating, thin film deposition and several other industrial applications. These pumps are capable of achieving vacuum in the range of 10^{-6} to 10^{-7} Torr.

Operating Principle

The main body is fabricated from stainless steel in the shape of a cylinder and water cooling copper coils are wrapped around the body. This unit operates on the principle of momentum transfer. It has a special type of structure within it. On the top of this structure there is a cap. Silicon oil is kept at the bottom which is heated using RF coil. The oil is vaporized and the vapor while moving through the special kind of structures attains supersonic speed until it reaches the top of the structure. Once it reaches the cap is there to present its upward motion and directs the vapor sideways. Now the vapor is free and rushes outwards.

Oil molecules being heavy in comparison with air molecules, it may easily drive the local air particles away along the diagonal direction. Here the momentum transfer takes place between oil and air molecules. The momentum of air particles has two components along horizontal and vertical direction. Due to the vertical component and also due to the pressure of air molecules coming from the chamber to fill up the vacancy, air molecules stated initially, rush go downwards where they are sucked out by a pump. Hence molecules pumped out are actually the same those were inside the main chamber where the metallization process is supposed to be performed. The oil molecules, those placed horizontally finally reaches the old surface (with respect to the cold wall process) would be condensed and again transferred to liquid phase and as a consequence, drops down to the bottom to raise the oil level there.

Remedies are taken in advance so that vaporized oil molecules can never reach the main chamber (if it would do so there is a big chance of contamination). Cold water is allowed to be circulated through the water line around the structure during the course of the process. Besides that cold trap is kept at the top that basically contains liquid nitrogen. All the cooling agents are useful in terms of condensation of oil vapor. Diffusion pump is effective if the main chamber is already partially evacuated. Employing this pump pressure up to 10^{-6} Torr may be achieved inside the chamber.

Vacuum Chamber

It is nothing but a large volume closed by an envelope made of the material having considerably high temperature resistance along with high pressure resistance. This is the place where the entire process is performed. This region of the entire unit is connected directly with the diffusion and rotary pump through baffle and roughing valve respectively.

Steps for Metallization on the Sample

Cleaning Technique

A glass substrate is taken as a sample. The surface of the sample may have some organic and non-metallic impurities, which must be cleaning using cleaning techniques. The steps for cleaning are as follows:

1. TCE (Trichloroethylene) – To remove grease, organic substances from the glass substrate we use TCE. It is slightly poisonous in nature, so care should be taken.
2. Acetone – To remove the traces of TCE, the substrate is to be cleaned with acetone.
3. Isopropyl Alcohol – IPA is again used to remove the acetone on the substrate.
4. De-ionized water – It is used to remove the IPA because of its high resistivity.

Loading of the Sample

After the completion of proper cleaning of the sample, it is ready for metallization. The vacuum coating unit is used for the purpose. Sample is generally introduced inside the vacuum chamber and a substrate holder is already there to hold the sample above the chamber. Titanium and Gold is loaded into individual boats and heated using RF heater so that they melt and start to evaporate. There is a shutter which controls which metal vapor is directed towards the sample. First the Titanium vapor is directed towards the sample and thus, titanium layer of controlled thickness is formed. Then after the required thickness is achieved, then Gold vapor is directed to the substrate and thus, a layer of Gold is deposited over the Titanium layer. The entire process of metallization is performed under high vacuum condition as mentioned before.

Pressure reduction inside the chamber

1. After loading the sample and metal inside the vacuum chamber, the vacuum chamber is closed properly and the rotary pump is turned on and the roughing valve is opened, keeping the roughing valve off.
2. The rotary pump creates a vacuum about the order of 10^{-3} Torr inside the upper chamber then the roughing valve is closed and the backing valve connected to the lower chamber is opened.
3. Then the air inside of the lower vacuum chamber is pumped out and it creates vacuum in the order of 10^{-3} Torr.
4. Roughing valve and backing valve should not be opened simultaneously otherwise oil inside the diffusion pump may enter inside the upper chamber or rotary pump.
5. After that diffusion pump is turned on. It creates 10^{-6} Torr at lower chamber. Backing valve is opened to support the diffusion pump.
6. Now the middle valve (backing valve) between the upper and lower chamber is opened. It is important that before starting the diffusion pump, cold water supply from the chiller must continue.

7. After 1.5-2 hours, the total pressure decreases to 10^{-5}. Now liquid nitrogen is poured in the liquid nitrogen trap (LNT) pressure decreases to 10^{-6} Torr. Vacuum is needed to reduce the mean free path of the metal molecule to reach the substrate surface.

Metallization

The substrate holder has the heater inside which is connected to a PID controller. The PID temperature controller controls the temperature. The metal boat are heated to the melting point and the metals starts to evaporate. The evaporating metal will be directed towards the substrate where the metal will deposit. Once the required layer depth is obtained, the shutter over Titanium is closed and Gold is opened. The deposition of Gold starts. Finally, the layer of Gold over Titanium is completed. The heater over substrate is switched off. The substrate is brought to room temperature. Then the metallized substrate is taken out of the chamber.

Observations

Time(sec)	Pirani Guage (Torr)	Penning Guage(Torr)
0	580	
12	380	
23	24	
34	7.1	
45	2.7	
55	1.2	
66	0.635	
77	0.39	
88	0.27	
99	0.19	
110	0.145	
120	0.115	
131	0.095	
142	0.0815	
153	0.07	
164	0.064	
174	0.0585	
185	0.054	
196	0.051	
207	0.048	
218	0.045	
239	0.0405	
272	0.036	
294	0.0335	
305	0.032	
326	0.03	

358	0.027	
411	0.0245	
443	0.0225	
485	0.021	
539	0.019	
634	0.0165	
740	0.0145	
761	0.0135	
782	0.0139	
803	0.01275	
845	0.01205	
887	0.01185	
919	0.01125	
940	0.011	
982	0.01075	
1066	0.00995	
1150	0.00925	
1213	0.00865	
1287	0.00855	
1319	0.008	
1382	0.0078	
1491	0.0074	
1543	0.00725	
1606	0.0071	
1638	0.007	
1680	0.0068	
1785	0.00655	
1891	0.00615	
1964	0.00595	
1978	0.00605	
1978	0.00605	
2030	0.0059	
2209	0.0056	
2272	0.0055	
2504	0.00525	
2851	0.00495	
3020	0.0048	
3262	0.0053	
3283	0.2165	0.00019
3294	0.0161	0.000093
3304	0.0136	0.000075

3315	0.01255	0.000062
3325	0.01105	0.000053
3336	0.0101	0.000046
3346	0.00955	0.000041
3357	0.00905	0.000036
3378	0.00855	0.00003
3388	0.008	0.000028
3399	0.00755	0.000026
3420	0.007	0.000024
3441	0.00655	0.000023
3473	0.00625	0.000021
3494	0.00605	0.00002
3526	0.00565	0.000018
3568	0.00525	0.000016
3610	0.005	0.000014
3673	0.00485	0.000012
3768	0.0046	0.00001
3831	0.0045	0.0000099
3915	0.0044	0.0000098
3954	0.0043	0.0000097
3954	0.0043	0.0000097
3989	0.00425	0.0000092
4021	0.0042	0.0000088
4042	0.0042	0.0000084
4074	0.00415	0.0000074
4084	0.00415	0.0000072
4105	0.0041	0.0000069
4126	0.0041	0.0000066
4148	0.0041	0.0000064
4211	0.004	0.0000061
4253	0.004	0.0000059
4305	0.00395	0.0000057
4348	0.00395	0.0000055
4432	0.00385	0.0000053
4495	0.0038	0.0000051
4548	0.0038	0.000005
4642	0.00375	0.0000048
4706	0.0037	0.0000046
4821	0.0036	0.0000044
4916	0.00365	0.0000042
5042	0.00345	0.0000041

5116	0.02145	0.000004
5211	0.00345	0.0000038
5306	0.00345	0.0000037
5411	0.0034	0.0000036
5495	0.0029	0.0000035
5621	0.00345	0.0000034
5679	0.0034	0.0000034

Photolithography

After the metal deposit is completed, photolithography process must be performed so that the required geometrical structure remains and the remaining metal deposition is removed. A photoresist named Dow Electronics Materials MICROPOSIT^{TM} S1813 Positive Photoresist is applied uniformly using spin coating for 35 seconds at 3500 rpm. Then a prebake or soft-bake process is performed at 100 ^{o}C for 1 minute on a hot plate. The prebake will convert the liquid-cast resist into a solid film which prevents the mixing of exposed section with unexposed section, provides temporary adhesion of the resist, and remove excess solvent by evaporation.

Photolithography is performed in yellow room to avoid unwanted UV exposure. During this process, a printed mask representing the geometry is applied over the prebaked substrate. Then UV light is switched on for 2 minutes. This is required to provide sufficient energy needed by the polymer in photoresist layer to cross-link. The exposure time is also a crucial parameter, as power of UV light and exposure time together will give the total energy taken up by the photoresist. An imperfection in time will cause either under or over-exposure, resulting in resolution loss. Overexposure due to longer time of exposure will cause roughness of wall and changes in width of geometry. Underexposure due to shorter exposure time, the photoresist may not be properly cross-linked over all the depth and so the mask will lift off during the developmental step.

After UV exposure is completed, a development solution compatible with the photoresist is used. This is used to remove the photoresist from the substrate. The positive photoresist used here, will be washed away by the developer solution (MF26a), leaving the bare underlying material. Thus, the mask now consist of exactly the pattern which is to remain on the wafer.

Then a post bake or hard bake is performed at 120 ^{o}C for 30 minutes to harden the resist. The hard bake solidifies the remaining photoresist, to make a more durable protective layer for future processes.

Etching Process

After the photolithography is performed, etching process is performed to remove the unwanted metal deposit. First gold is etched away using a solution containing 4 g Potassium Iodide and 1 g Iodine dissolved in water. Then excess titanium is etched away using a solution of Nitric acid, Hydrogen Fluoride in water with mixing ratio 1:1:50.

Steps performed in LAB

1. A clean glass substrate was taken and using metallization process, Nickel was deposited on it. Then photoresist S1813 was applied over it using spin coating process for 35 seconds at 3500 rpm, and the resultant was soft-baked for 1 minute at 100 ^{o}C on a hot plate.
2. A photo mask is now placed over the substrate, which represents the heater pattern to be developed (the overall microheater geometry that is simulated is represented by the printed mask). Then it is exposed to the UV light (in a yellow room) for 2 minutes.
3. After the UV exposure is complete, a developer solution MF26a is used to dissolve the unmasked portion of the resist. The developer solution is poured in to a beaker and the exposed sample is placed in it. The beaker is shaken so that the reaction occurs. As the reaction progresses the pattern begins to unmask and the developer solution starts to change color towards purple. The process continues until the proper mask pattern unveils.
4. Then hard bake is performed at 120 ^{o}C for 30 minutes. This will harden the resist and drive off the excess solvent in unmasked portion.
5. Finally etching is performed to remove the polymer formed in unmasked section of the developed mask. For etching, the developed mask is shaken in a beaker containing a solution of Nitric Acid and distilled water in 1:10 ratio for 4 min 1 seconds.
6. Then the etchant must be removed completely using Acetone for few seconds. Finally, the transparent glass with patterned mask yields.