Version AJF/4

EGT3
ENGINEERING TRIPOS PART IIB

Thursday 30 April 2015 x.x0 to xx

Module 4M6

MATERIALS AND PROCESSES FOR MICROSYSTEMS (MEMS)

ANSWERS

1 (a) (i) An alternative to the gas phase deposition of metallic layers is deposition (or plating) from a liquid solution, immersion plating is the simplest of the solution based technologies. The surface to be coated is simply dipped into a solution containing a more noble (more electropositive) metal than the surface atomic species. The less electropositive surface atoms will go into solution in preference to the plating metal, and so a galvanic displacement takes place, and a thin metallic coating results. Assuming that the coating in impermeable to the solution, the plating will naturally stop once coverage is complete, and so the resulting layer is very thin.

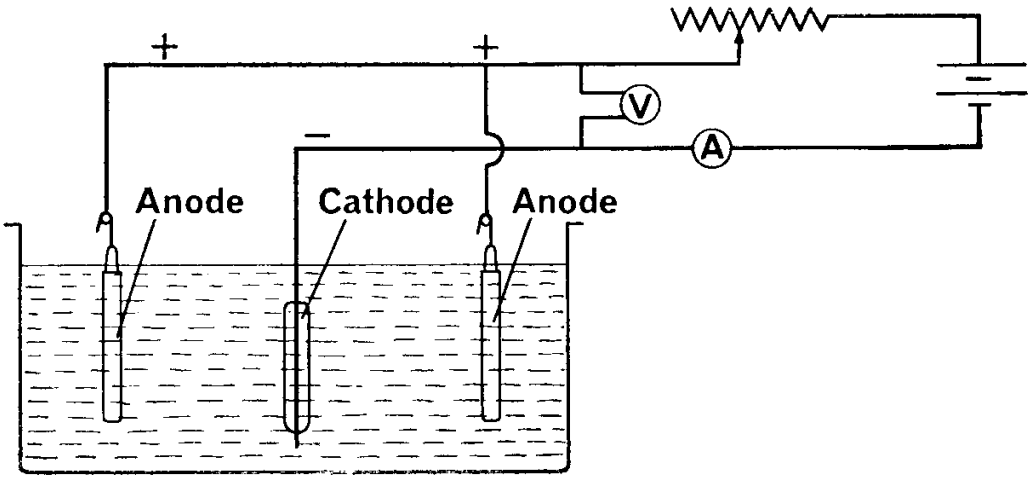
(ii) Electroless plating also involves dipping of the substrate to be covered into a solution containing ions of required metal coating. However, unlike immersion plating, this is not a displacement reaction, and so thick layers may be produced. The plating solution contains a reducing agent which is oxidised in the presence of the metal surface and catalyses the reaction, allowing reduction of the metal ions which then deposit. Non-conducting materials may also be coated by this method if first dipped in a sensitising solution which initiates the reaction. Once started, the reaction will continue by autocatalysis in the usual way. Alloys may be produced by using a mixture of metal salts. In addition to the reducing agent and metal salt, the following are also frequently added to the plating solution to improved the process:

- Stabiliser. These ensure that the reaction only occurs in the presence of the catalyst
- Buffer. A buffering agent is used to ensure that the H+ ions produced do not cause the pH of the solution to decrease too quickly
- Accelerator. This is an anion which facilitates the anodic oxidation process and yields faster deposition rates (up to ~10 nm s^{-1})

Electroless plating has the advantage over electroplating (next section) in that it yields a highly conformal coating which will only occur on surfaces where the reaction can be initiated, allowing simple patterning, but hydrogen evolution from the solution near the surface can reduce the quality of the resulting metal coating.

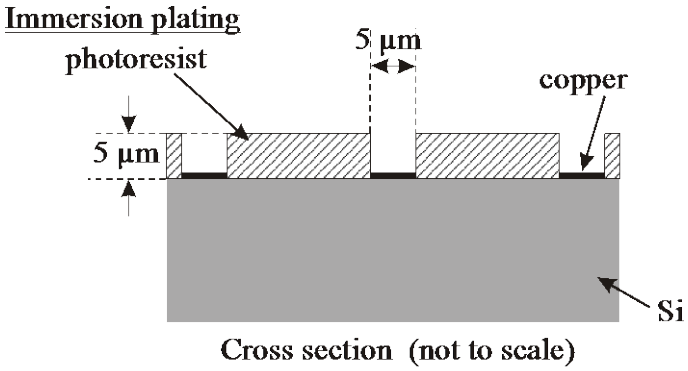
(iii) Electroplating is the application of metallic coatings to conductive surfaces by electrochemical processes. The sample to be coated, which must have a conductive surface, is placed in a solution of the metal salt to be deposited

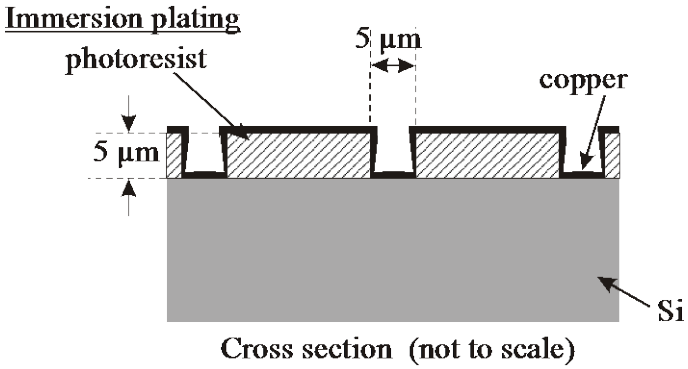
together with another conducting plate and these are connected as the cathode and anode respectively to a low voltage, dc power supply. In aqueous solution, the metal salt will dissociate, as will the water to a small extent.

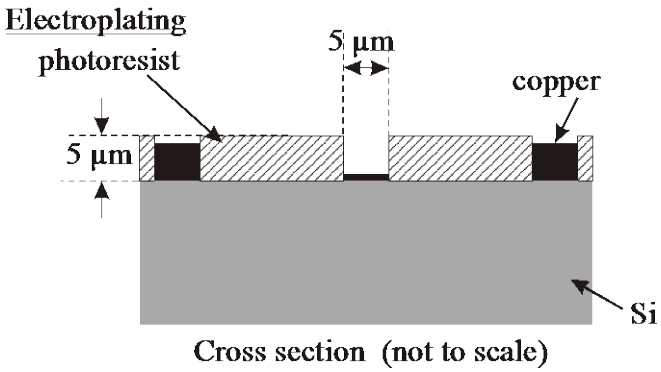


Under the application of a bias to the electrodes, the positive ions will migrate to the cathode. The metal ions will be deposited onto the electrode surface while the H+ ions will mostly react to form gaseous hydrogen, although a small quantity may be incorporated into the metal. The reaction at the anode will depend on its nature. For an inert anode, the hydroxyl ions will be discharged, $4OH^- - 4e^- \rightarrow 2H_2O + O_{2\,(gas)}$. As the anode is inert, the salt ions cannot react and so are not discharged. This has the disadvantage that the metal ion is depleted from the solution with time. Alternatively, the anode may be made from the depositing metal, in which case it will be dissolved into the solution in preference to any other electrochemical reactions at low voltages.

(b)







(c) A negative tone resist would be preferable as this means tah the mask would be mostly transparent for ease of processing, we do not require very high resolution, and pinhole count is likely to be lower.

2 (a) Materials selection in MEMS devices is affected by a number of factors. These will include the function of the device (its purpose, which might be to resonate at a particular frequency with minimum mass, or deflect under a particular load without fracture, for example). This will allow the generation of a figure of merit for comparing different materials. The geometry of the device will be important as not all materials can be produced in particular geometries. This is particularly true of microfabrication, where, for example, intrinsic stresses can limit the thickness of thin film materials. Finally, there is the process required to fabricate the device. Only certain materials can be produced by given techniques. In practice all of these factors are mixed together – photolithography might be required to pattern a given material, which limits the minimum feature size, for example.

(b) (i) The force produced by the actuator from the Young modulus expression is

$$F = \frac{AE\Delta l}{l}$$

However, the expansion die to the temperature rise is

$$\Delta l = l\alpha\Delta T$$

Therefore

$$F = A\Delta TE\alpha$$

So the key Figure of Merit is that $E\alpha$ has to be maximised.

(ii) If actuation speed is important, then the thermal mass of the metal should be mimimised, implying a low specific heat capacity, as this will allow fast heating and cooling. In addition, to assist with fast cooling, the thermal conductivity should be high [Assessor's note: few candidates appreciated this in the exam].

(c) The process flow is as follows:

1 RCA1 Boil one silicon (100) wafer in RCA Clean 1 (NH_3(aq):H_2O:H_2O_2) to remove organic contaminants.

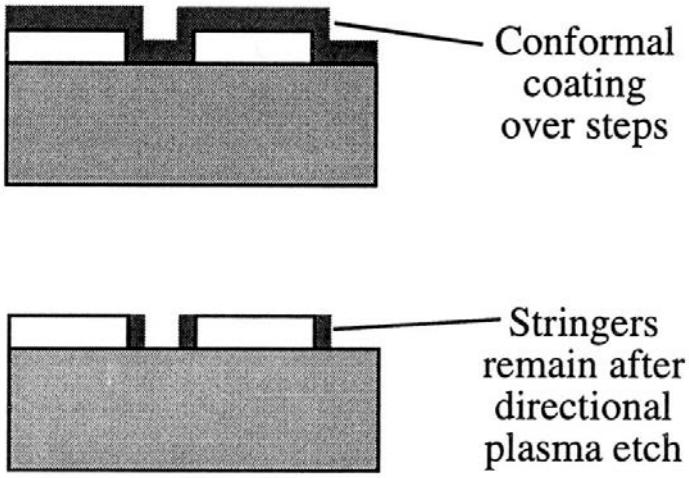
2 RCA2 Boil the wafer in RCA Clean 2 (HCl:H_2O:H_2O_2) to remove metallic ion contaminants.

3 SIN1 Use LPCVD to grow a 200 nm thick layer of silicon nitride onto the top surface of the silicon using a gas mixture of SiH_2Cl_2 and NH_3 at 800 °C and 70 Pa.

4	PHO1	Spin a layer of photoresist onto the top side of the silicon wafer.
5	BAK1	Pre-bake the photoresist.
6	EXP1	Expose the photoresist through a mask to produce a pattern in the photoresist which will form square holes where the silicon nitride is to be removed.
7	DEV1	Develop the photoresist.
8	BAK2	Post-bake the photoresist to harden it.
9	RIE1	Reactive ion etch the silicon nitride layer using a gas mixture of CF_4+CHF_3+He.
10	ACE1	Ultrasonicate the substrate in acetone to remove the photoresist.
11	IPA1	Ultrasonicate the substrate in IPA to remove the acetone.
12	DIW1	Rinse in DI water to remove the IPA.
13	NIT1	Blow dry under dry nitrogen.
14	PHO2	Spin a layer of photoresist onto the top side of the silicon wafer.
15	BAK3	Pre-bake the photoresist.
16	EXP2	Expose the photoresist through a mask to produce a pattern in the photoresist which leave holes where the metal is to be deposited.
17	DEV2	Develop the photoresist.
18	BAK4	Post-bake the photoresist to harden it.
19	SPU1	Sputter deposit the metal layer from a pure nickel target in an argon atmosphere.
20	ACE2	Ultrasonicate the substrate in acetone to remove the photoresist and lift off unwanted metal.
21	KOH1	Etch the sample in a 25% KOH solution at 80 °C to undercut the structure.
22	DIW1	Rinse in DI water.

[Assessor's Note: Several students in the exam tried to carry out infilling of the area underneath the actuator rather than simply using a silicon etch to undercut the structure which would have been much easier.]

3 (a) Stringers are regions of material left on the side walls of a structure that are left behind after an anisotropic etch, such as a plasma etch. They are often formed when a rough surface is coated conformally with another material which is subjected to the anisotropic etch. The large effective film thickness on the edge of structures leads to the stringer formation.



(b) CMP is widely employed in the fabrication of both microsystem and microelectronic devices. It provides a means of planarising a surface by removing the top layers of material. A rotating platen is pressed against the surface to be planarised and a slurry injected between the two surfaces. The slurry contains both an abrasive and a chemical etchant, and the two act in tandem to produce a faster etch rate than either can achieve alone (faster even than the algebraic sum of the two processes) whilst yielding a very flat surface. This is an extremely useful process which can also be used to produce patterned structures.

CMP does have its disadvantages. It uses very large quantities of slurry. There is an etch rate dependency on pattern density and material type. Dummy structures are frequently required to ensure a uniform etch over a whole surface. However, compared with alternatives such as resist etch back or polymer planarization, no new material is added to the surface being planarised, and there is no need to try to match the etch rate of dissimilar materials.

(c) The process flow is as follows:

1 RCA1 Boil one silicon (100) wafer in RCA Clean 1 (NH_3(aq):H_2O:H_2O_2) to remove organic contaminants.

2	RCA2	Boil the wafer in RCA Clean 2 ($HCl:H_2O:H_2O_2$) to remove metallic ion contaminants.
3	PHO1	Spin a layer of photoresist onto the top side of the silicon wafer.
4	BAK1	Pre-bake the photoresist.
5	EXP1	Expose the photoresist through a mask to produce a pattern in the photoresist which will leave photoresist where the silicon supports are to be formed, but 20 µm wider than the final width of the supports.
6	DEV1	Develop the photoresist.
7	BAK2	Post-bake the photoresist to harden it.
8	RIE1	Reactive ion etch the exposed silicon using a gas mixture of CF_4+CHF_3+He.
9	OXI1	Thermally oxidise the wafer in a wet oxygen ambient at 1200 °C until a 10 µm thickness has been oxidised.
10	CMP1	Use chemical mechanical polishing to remove the protruding silicon dioxide and leave a flat surface with 10 µm of silicon dioxide between the exposed silicon supports.
11	SIN1	Use LPCVD to grow a 500 nm thick layer of silicon nitride onto the surface of the wafer using a gas mixture of SiH_2Cl_2 and NH_3 at 800 °C and 70 Pa.
12	PHO2	Spin a layer of photoresist onto the top side of the silicon wafer.
13	BAK3	Pre-bake the photoresist.
14	EXP2	Expose the photoresist through a mask to produce a pattern in the photoresist which will leave photoresist where the silicon nitride beams supports are to be formed.
15	DEV2	Develop the photoresist.
16	BAK4	Post-bake the photoresist to harden it.
17	RIE2	Reactive ion etch the exposed silicon nitride using a gas mixture of CF_4+CHF_3+He.
18	ACE1	Ultrasonicate the substrate in acetone to remove the photoresist.
19	IPA1	Ultrasonicate the substrate in IPA to remove the acetone.
20	DIW1	Rinse in DI water to remove the IPA.
21	BHF1	Etch the silicon dioxide using a buffered hydrofluoric acid etch. This is self-terminating.
22	DIW1	Rinse in DI water to remove the acid.
23	IPA2	Rinse in isopropanol.
24	ACE2	Rinse in acetone to reduce surface tension.
25	NIT1	Blow dry gently under dry nitrogen.

4 (a) The challenge with MEMS device using microelectronic fabrication techniques is that they tend to be very costly. Historically, they have been designed to achieve very high levels of tolerance, both in minimum feature size and surface roughness, but this results in very high costs of fabrication per unit area. The challenge therefore with MEMS devices, such as biosensors, which require large areas, is that they become very costly using the traditional fabrication techniques. However, if many devices can be produced on a single substrate with a high level of functionality through integration of electronics, then, for certain markets, manufacture can become economic. A good example of a successful MEMS sensor is the accelerometer, where the large markets associated particularly with the mobile device industry have allowed significant cost reductions.

(b) Three sterilisation techniques are:

- Heat sterilisation in a autoclave (121° C, >10 psi for >30 minutes in either steam or dry heat)
- Gas sterilisation (ethylene oxide ambient temperature, 30-60% relative humidity for >16 hours) [NOTE: ETO is carcinogenic and toxic so this method is far from ideal]
- Liquid disinfection (chlorine, iodine, 50-70% aqueous ethanol or isopropanol)

(c) Full biomicrosystems need to be carefully designed and properly partitioned. We need to consider what really needs to be ‘on chip’ in the biological environment. Frequently only static systems are required in which microwells are used to hold small volumes of material in place either electrically or microfluidically and detection is performed optically. Some silicon electronics is frequently required to perform in situ pre-amplification of small electrical signals. [Assessor’s note: Few students in the exam really appreciated that the economics of manufacture makes such devices difficult to commercialise or the consequent importance of system partitioning – i.e. making sure that the disposable part of a sensor is as simple as possible.]

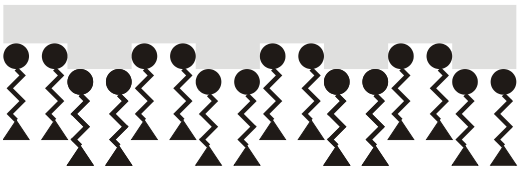
(d)

1 Photoresist is patterned onto a silicon substrate in the negative of the pattern to be printed. The surface is then treated with (tridecafluoro-1,1,2,2-tetrahydrooctyl)-1-trichlorosilane to reduce adhesion.

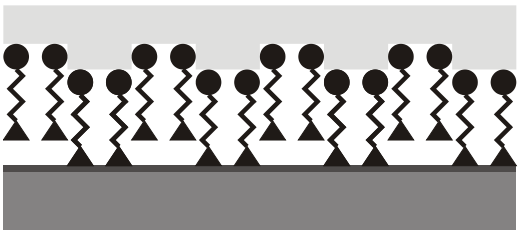
2 PDMS (polydimethylsiloxane) is spin coated over the patterned resist and cured at 60° C for one hour.

3 Finally, the PDMS may be simply peeled away from the substrate to leave the printing stamp.

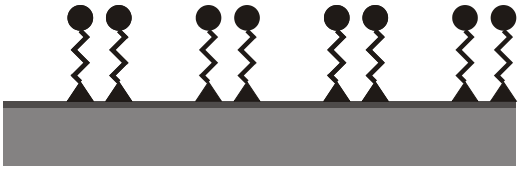
PDMS is hydrophobic in its native state, but may be made hydrophilic by exposure to an oxygen plasma which leaves OH groups on the surface.



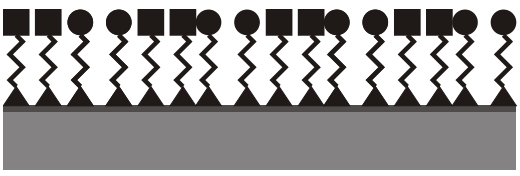
1 The PDMS stamp is coated in the hydrophilic or hydrophobic end of alkane thiol 1.



2 The coated stamp is pressed onto the substrate to be coated (such as gold coated silicon).



3 The PDMS stamp is removed, leaving alkane thiol 1 only at the contact points.



4 The sample may now be exposed to a second alkane thiol which will only be able to coat the exposed regions of the substrate.

The PDMS stamp allows the patterned transfer of molecules with an almost nanometre resolution without the need for repeated photolithography beyond the initial stamp fabrication.