# Microfabrication

## Fabrication accuracy

City (10 km)---House(10 m)----Optical fiber (1 mm)----Bacteria(1μm)----Virus(0.01μm)---1A°

## Traditional mechanical machining

- Object of size 1mm to 50 cm
- Precision of the order of 10 μm

## Special mechanical machining

- Displacement precision ~ 1 nm
- Angular precision ~ $(10^{-5})°$
- Applicable for metal alone. Not for glass, teflon or plastic
- Price tag

## Etching / lithography / deposition

- Range 0.2 μm to 500 μm
- Use glass or silicon

# Microfabrication .....contd.

## Plastic MEMS

- Range 0.5 µm to 500 µm.
- Use elastomers such as PDMS (polydimethyl siloxane) or PMMA (polymethyl methacrylate).
- Channels can be made by either direct method (laser ablation) or replication method (use of mold).
- By weight, plastics are 100 times less expensive than silicon, and can be made disposable.
- Rapid prototyping.
- Surface effects, transparency, diversity of materials can be introduced easily.

# Clean Room

- Temperature regulated at 20°C.
- Humidity regulated and traversed by flux of filtered air.
- Number of particles of size < 4 μm per cubic inch defines a clean room.
- For fabrication of microprocessor, a number ≤ 10 is suggested.
- For MEMS microfabrication, a number ≤ 10,000 is suggested.
- Wearing hair-cover, shoe-cover, gloves required in the second case also.

# Photolithography mask

- Protects certain part of a resist from illumination (spectral band 300 nm to 450 nm).
- Plates of quartz on which deposits of chrome forms a pattern.
- Deposition is made using electron beams.
- A less-precise one can be just a print-out on plastic transparency sheet.

# Photosensitive resist

- Mask is placed on top of a substrate, covered by photosensitive polymer.
- Substrate is put under uniform beam.
- The regions appear on the polymer as lit or dark as per the pattern that had been designed on the mask (pattern transfer step).
- Light sets changes in photosensitive polymer and make the polymer soluble to certain solvent. Upon dipping in the solvent, the polymer in the lit portion is dissolved.
- The exposed portions of the substrate undergo chemical attack from the etching fluid.
- Depending on the time over which the substrate is kept in etching fluid, the exposed portions are etched to a depth.

# Spin Coating

- Photosensitive polymer is deposited in a thin layer on a solid substrate of silicon or glass.
- Three stages of the coating process are Distribution, Spreading, Evaporation, Solidification.
- Use of vacuum to hold the wafer.
- Equilibrium thickness: The thickness achieved after spinning for few minutes. With evaporation of solvent, viscosity increases. With decrease in radial velocity, viscosity increases for shear-thinning fluid. The fluid stops moving beyond a distance.
- Heating of resist at 70°C for few minutes to evaporate 15% of solvent remaining to avoid formation of cracks.

equilibrium thickness (h): kC(u/w2)^(1/3)
k = constant
C = initial concentration of polymer
u = viscosity
w = angular momentum velocity

Air bubbles in dispensed resin
Dispense tip is cut unevenly or has defects

Dispense rate is too high
Spin bowl exhaust rate is too high
Resist on wafer for too long prior to spin
Spin speed / acceleration is too high

Fluid is striking substrate  off centre
Spin time too short

Insufficient dispense volume

Air bubbles / particles present in resin
Particles on substrate prior to dispense

# Exposure

- Substrate, coated with photosensitive polymer is placed in an aligner.
- Luminous flux crosses the mask and hits the coating on the substrate.
- Light of wavelength 300 – 450 nm is used.
- Luminous flux initiates reactions in the polymer.
- Positive Resist (PMMA): After exposure, the polymer becomes soluble in a solvent as the light weakens the internal bonds→ Exposed part is eliminated.
- Negative resist (KTFR from Kodak, SU8 from IBM): After exposure, the polymer becomes insoluble in a solvent, as light induces covalent bonds between chains → The unexposed part is eliminated.

# Other issues

Half-light effect during exposure

- Opaque object produces shadow as well as penumbra.
- Diffraction.
- These effects should be as small as possible.

Transparency of the resist

- Light has to penetrate and modify the resist over the entire thickness of resist layer

Choice between positive and negative resist

- Negative resists adhere better to the substrate.
- Negative resists are more chemically resistant.
- Positive resists show better contrast in photosolubility.

# Typical Protocol

- Immerse the wafer in acetone in an ultrasonic bath to dissolve organic residue and then drying.
- Repeat the previous step with alcohol.
- Dehydrate on a hot plate at 120°C for 5 minutes
- The first three steps, above ensure removal of contaminant from the wafer.
- The wafer is coated with photoresist in spin-coater.
- System is heated to 65°C for one minute, and 95°C for three minutes to ensure hardening of photoresist.
- Exposure.
- The wafer is post-baked to 65°C for one minute, and 95°C for two minutes to ensure progressive rearrangement of material during thermal deformation.
- The system dipped in developing solution for 3 minutes.
- The system is rinsed with alcohol, and dried with nitrogen.
- The system is heated to 200°C for two hours.

# Silicon

- Delivered in the form of wafer that constitute a monocrystal.
- Produced by well-controlled crystalline growth process in clean-room (Class 1-10), by slowly pulling a crystal from an ultrapure bath of silicon.
- Slices are cut from a cylinder, followed by atomic polishing phase.
- Typical thickness of wafer ≈ 500 μm.
- The other dimensions ≈ 4 inches.
- Two inter penetrating face-centred cubic lattice network.
- Side of one cubic face is 5.43 A°.
- Highest density planes <1 1 1> form an angle of 54.74° to <1 0 0> plane.

# Wet etching

- Subjecting parts of an object to chemical reactions and subsequent dissolution.
- The other parts are protected by a mask.
- Technique is used in fifteenth century to decorate armours. Wax is used as a mask. Wax on parts, identified for etching is cut out. Dipping into reactive bath for a period of time ensures chemical etching / hollowed out pattern in places, not covered by wax. Wax is removed by heating later.
- Rate of etching follows Arrhenius expression, and is strong function of temperature, unlike solubilisation of resist.

# Anisotropic etching

- <1 1 1> planes of silicon are highly packed with atoms.
- Velocity of etching is slow along planes <1 1 1> and fast along <1 0 0> or <1 1 0>.
- Typical etching speed with KOH along <1 1 1> plane is 13 μm/hour.
- The etching speed is 60 times faster in other orientations.

# Anisotropic etching …contd.

- Etching happens preferentially along a crystallographic plane.
- The crystal will make the cut-out forms appear spontaneously along the planes where the etching is slowest.
- This form of etching makes possible cavities with facets, which can be useful.
- This type of etching is not possible in amorphous solid, e.g., glass.

# Isotropic etching

- Carried out equally in three spatial direction.
- Example: Etching of glass by HF.

# Dry etching of silicon

- Attack of a substrate by an ionic species, contained in gaseous or plasma phase.
- Isotropy or anisotropy controlled by the etching process, and <u>not</u> by crystalline structure.
- Types of dry etching:
    - Physical etching (sputtering, Ion-beam etching, Ion milling)
    - Chemical etching
    - Physico-chemical etching (RIE)
    - Physico-chemical etching with inhibitors (DRIE)

# Physical etching

- Ions are accelerated in an electric field (10 eV to 5000 eV generated between two electrodes).
- Ions bombard the surface of a target.
- Low pressure (~ mTorr ) helps in making the ion ballistic.
- Use of $CF_4$ gas is common in ionization.
- The method is inherently anisotropic because of the pathway taken by the reflected ions.
- Selectivity (about the portions of the substrate to be etched) is poor.
- Etching rate 0.6 µm/hour to 18 µm/hour for most materials.

# Chemical etching

- Chemical species (neutral) migrate towards the target by diffusion.
- Chemical reaction occurs on the surface of the target.

$2XeF_2$ (sublimated) + Si $\rightarrow$ 2 Xe + $SiF_4$ (volatile)

- Both the products desorb and diffuse back.
- Chemical species not accelerated by application of voltage.

# Chemical etching … contd.

- Volatile products are not reflected.
- Movement is diffusive, and not ballistic.
- Mostly isotropic etching.
- Etching rate ≈ μm/min.
- Etched surface is rough.
- Pressure is maintained at 0.1 Torr to 1 Torr.

# Physicochemical dry etching
# (Reactive ion etching: RIE)

- Combined effect of physical and chemical etching.
- Commonly used in microfabrication.
- Target is placed on the cathode.
- Cold plasma breaks down $CF_4$ to $CF_3^+$ + $F^-$ and makes the ion ballistic at low pressure.
- Anisotropic; Etch rate 10 times that of physical etching.
- Good in etching $SiO_2$ (wet etching of $SiO_2$ takes time) from the surface of Si wafer.

# Physico-chemical etching with inhibitor (Deep reactive ion etching: DRIE)

- Physicochemical etching with non-volatile reaction products.
- The gas is $CCl_4$.
- Non-volatile component (inhibitor) is deposited on the neighbouring vertical surface.
- A polymerized protective film is formed on the vertical surfaces.
- The film protects the vertical surface from the reflected products, arising from ballistic ions → Very deep structure ~ 500 µm with aspect ratio of 30:1 can be obtained.
- Making a through hole of high aspect ratio is difficult from wet etching due to anisotropy.

# Deposition on silicon and glass

- Deposition is required to form electrode, catalyst, thermal or electrical insulation, adsorbent, optical elements on the wall of the channel.
- Physical vapor deposition

  Certain species from the holding gas is adsorbed on the surface of the target. Two types are thermal evaporation and sputtering.
- Chemical vapor deposition

  Certain species from the holding gas reacts with the target forming compounds that are chemically bonded to the target.

# Thermal evaporation

- Material to be deposited faces the target in a container.
- High temperature ensures sublimation of materials.
- Low pressure ($10^{-8}$ Torr) ensures avoidance of unwanted parasite deposition of molecules present in the chamber.
- Deposit rate ~ A°/s
- Simple to implement. Good for metal deposit.

# Sputtering

- Target is on anode, and the material to be deposited is on cathode.
- Use of cold plasma gives the option of depositing variety of materials.
- Ionic kinetic energy of 0.3 keV to 2 keV ensures penetration by one or two molecular layers of the substrate → good adhesion.

# Chemical vapor deposition

- Homogeneous reaction takes place in the gas phase, and the product gets adsorbed on the target surface.

$SiH_4 \rightarrow Si + 2H_2$ nucleation and growth of Si crystal

desorption of $2H_2$

- Heterogeneous reaction at the surface of the target.

  $Si + O_2 \rightarrow SiO_2$

  2 nm layer of $SiO_2$ forms when silicon wafer is exposed to ambient air.

  The thickness of $SiO_2$ will be 1 μm when exposed to air at 650°C.

- Gas phase diffusion ensures low defect density in crystal.

# Chemical vapor deposition ….contd.

- Three types of CVD: LPCVD (low pressure ~ 1 Torr), APCVD(atmospheric pressure), PECVD (plasma enhanced, low pressure).
- Plasma enhances chemical reactions at the target surface using ionic bombardment. Also, energy of incident particles helps penetrating into the target surface, resulting in constant film thickness and conformal deposition.
- Lower pressure ensures that the mean free path of molecules is high for easy diffusion of species.
- Deposition rate ~ µm / hour.
- Versatility in material deposited (includes deposition of insulators)

# Electrolytic deposition

- Metal deposition on the substrate held as anode.
- Solution is metallic salt (e.g., $CuSO_4$).
- Metal ion ($Cu^{2+}$) migrates towards anode.

  $Cu^{2+} + 2\ e^{-} \rightarrow Cu$

- Metal ion captures electron at the electrode and form molecule that adsorbs onto the electrode.

# Sealing in silicon and glass

- Grooves, carved ~~curved~~ onto the silicon wafer must be covered with another glass wafer in water-tight manner to complete the making of device.
- Anodic bonding refers to glass-silicon sealing by the use of intense electric field (1 kV) and elevated temperature (400°C).
- The electric field induces migration of $Na^+$ ions in the glass to the interface, and interpenetration of atoms at the glass-silicon interface.
- Thermal expansion coefficient of glass lid should be same as silicon to avoid crack. Pyrex Corning 7740 may be a suggested one.

# Sealing in silicon and glass

- Cleanliness of surface is important.
- Anodic bonding between silicon to silicon is possible through intermediate glass layer. Thin glass layer (0.5 µm to 4µm) is deposited on silicon by physical vapor deposition or spin-on technique.
- Seal from anodic bonding is water-tight up to 100 atmosphere.
- Fusion bonding refers to bonding Si-glass wafers without application of any voltage, instead using higher temperature (600°C to 1100°C).

# Fabrication of plastic microfluidic devices

Moulding, Casting, and Microinjection

- Mould is the negative master, made of silicon, electrodeposited metal or reticulated polymer.
- Common method of making a mould is photolithography. For processing at high temperature and pressure, electrodeposited or traditional micro-machined metallic moulds are used.

# Moulding

- Catalyst + polymer is poured on a mould and heated.
- Later, the structure is peeled off the mould and contains the pattern of the mould in negative form.
- Common polymer is PDMS. Polydimethylsiloxane
- Temperature of about 70°C is required.

# Casting / hot embossing

- Mould / Stamp is pressed into a heated deformable material.
- After cooling and separation, the structure represents negative of mould.
- Common polymeric material is PMMA. Other plastics are polyethylene, PVC, PEEK.
- Temperature ~ 170°C
- Pressure ~ 10 – 100 bar.

# Microinjection

- Meant for serial production on industrial scale.
- The liquid plastic material is injected into a mould under a vacuum at a temperature  > Glass Transition Temperature.
- Next, the system is cooled down below the glass transition temperature.
- Mould is taken off, and a structure corresponding to the negative of the mould is obtained.

# Advantages of plastic microfluidic devices

- Precision is surprisingly high (sub-micrometric precision).
- PDMS is transparent.
- Elastomeric quantity helps water-tightness by holding on tightly to the lid / interconnect.
- Weak surface energy helps in peeling from the mould.
- PDMS is permeable to gas. Trapped air can escape during liquid fill-up operation.
- Untreated PDMS is hydrophobic. It becomes hydrophilic after oxidation of methyl group at the surface by oxygen plasma.

# Disadvantages of plastic microfluidic device

- Aging : It's not going to last for years as PDMS has shelf-life
- Deformability for high aspect ratio.
- PDMS cannot tolerate high temperature → evaporative deposition of metallic electrodes is not possible. However, hybrid structure made of PDMS layer placed on silicon wafer with evaporated electrode is possible.

# Direct micromachining of plastics

- μ-stereolithography: photosensitive resists are polymerized layer after layer by applying a spot of UV laser.
- Laser ablation: Plastic is removed locally by sublimation using intense laser beam.

# Bonding of two wafers
# to put a lid on carved microchannel

<u>Direct bonding</u>

- Bonding between substrates of same material – no problem of thermal stress from mismatch of expansion coefficient.

  Si – Si, Glass – Glass, Polymer – Polymer

<u>Adhesive bonding</u>

- Use of glass layer – sprayed, screen-printed, or sputtered on silicon. Annealing the stack at sealing temperature makes the glass layer to melt and flow. Upon cooling , a strong bond develops.
- Epoxies, UV curable epoxies and photoresists as glue has advantage of low process temperature and can be applied to all kinds of substrate material.

# Bonding of two wafers …..contd.

## Eutectic bonding

- A thin gold film, sputtered on silicon wafer.
- Gold-silicon eutectic bonding is achieved at a relatively low temperature of 363°C.

## Protocol for Si-Si direct bonding

- Hydration of silicon wafer by immersing in $H_2O_2$ – $H_2SO_4$ mixture or boiling $HNO_3$, or diluted $H_2SO_4$.
- Bonding at elevated temperatures (300°C to 1000°C).
- Annealing (800°C to 1100°C) to improve bond quality.

# Bonding of two wafers ….contd.

Protocol for glass-glass direct bonding

- Cleaning of glass wafer with $H_2O - NH_3 - H_2O_2$ solution.
- Removal of moisture at 130°C, followed by thermal bonding at 600°C for 6 – 8 hours.

Metal-metal direct bonding

- Use of high pressure (276 bars) and temperature (920°C) for 4 hours.

Polymer-polymer direct bonding

- For polymers with low surface energy (e.g., PDMS) use of surface treatment with oxygen plasma.

# Fluidic interconnects (Press-fit and glued)

Press-fit

- Utilizes elastic forces of coupling parts to seal fluidic access.
- Sealing force is relatively small → suitable for low pressure applications.

Glued

- Adhesives offer good sealing by filling the gap between the external tube and the device opening.
- Surface roughened for better adhesion.
- Polymer glue is used between glass capillary and silicon wafer.

# Fluidic interconnect ….contd.

- Glass-sealing on metal tube is possible on Kovar tube (alloy made of 29% Ni, 17% Cu, and rest Fe). After fitting Kovar tube in fluidic access, glass beads are placed around them. A carbon fixture around the joint helps annealing the assembly at 1020°C.

<u>Plastic Coupler</u>

- The coupler is a smaller annular material at the tip of the external capillary that is inserted directly into etched opening.
- Thermal annealing allows the plastic to reflow around the opening. Once cooled, hermetic seal results.
- Couplers can offer elastic force to some extent.

# Integrated 'O' ring

- Annular area will be etched first by one of the techniques (DRIE say).
- Deposit an oxide ($SiO_2$) or a nitride ($Si_2N_4$) layer.
- Silicone rubber will be squeezed into the cavities.
- Fluidic access will be opened from the back side by DRIE.
- Oxide / nitride layer is etched in buffered HF acid and $SF_6$ plasma.

# Biocompatibility - material response

- Generated by the device inside host tissue.
- Caused by diffusion of body-fluid from host tissue into the device.
- Device material swells, develops micro-cracks on the surface → altered mechanical properties, and in some cases leaching.

# Biocompatibility - cellular response

- Generated by the host tissue.
- Inflammation at the device-tissue interface, reddening and pain. If the inflammation lasts long and damages local cells → device is cytotoxic.
- Otherwise, chemical signals released by the damaged tissue attract white blood cells.
- A fibrous encapsulation made of foreign body giant cells and macrophages is formed around the device.
- If the encapsulation does not affect the functioning of the device → BIOCOMPATIBLE.
- Tests: *In vitro* (in laboratory glasswares) and *in vivo* (in live animal or human).