Design, Process Development and Fabrication of SU-8 based CMOS Compatible Capacitive MEMS Platform for Bio-sensing Applications

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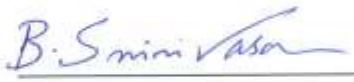

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Dedicated to

My Parents

Abstract

Biosensors are integrated devices which could provide information about the composition of Biological samples (semi) quantitatively. The key element of Biosensors are their transducers which are usually Micro or Nano scale structures with Biological recognition elements as part of them.

Realization of a practical and usable Biosensor involves a wide range of interdisciplinary activities. The emerging interdisciplinary fields of Nanotechnology and Nanofabrication has helped the heterogeneous integration of Biology with Engineering systems which was not possible earlier.

Concurrently, the continuous shrinking of Semiconductor Transistors for past 50 years as predicted by the famous Moore's Law might see its Physical limits soon. This has prompted the Semiconductor industries to develop exotic applications (which are also called More than Moore Technologies) using the conventional CMOS Microelectronics to grow their business. Bio-chips or Lab-on-a-Chip are one such application where Biosensors are integrated with Microfluidics and Microelectronics using suitable Nanofabrication techniques. Bio-chips are highly accurate, faster, reliable and cheaper in diagnosing medical samples. These devices have wide range of applications in futuristic health care, Bio-warfare, environmental screening and medicine research & development.

This dissertation presents a simple and novel technique for Bio-functionalizing SU-8, a popular MEMS polymer material. Also this work elaborates on the concepts of Surface Stress based Biosensor, CMOS compatible Process Development and Fabrication of a SU-8 based Micro-beam which can transduce the Adsorbent target Bio-molecule concentration into a variation in Capacitance. The variations in Capacitance could be converted in to an Electronic Signal by suitable Circuits for further processing and analysis. This device can be used as it is or can be suitably integrated over the Gate of a MOSFET to form a Flexure-FET or Suspended-Gate FET Biosensor device.

Contents

Chapter 1

Introduction

1.1 Definition of Biosensor

International Union of Pure and Applied Chemistry (IUPAC) defines Biosensor as follows:

A Biosensor is a self-contained integrated device which is capable of providing Specific quantitative or semi-quantitative analytical information using a biological recognition element (biochemical receptor) which is in direct spatial contact with a transducer element. A biosensor should be clearly distinguished from a bioanalytical system, which requires additional processing steps, such as reagent addition. Furthermore, a biosensor should be distinguished from a bioprobe which is either disposable after one measurement, i.e. single use, or unable to continuously monitor the analyte concentration.

1.2 Overview of Biosensor System

A Biosensor system consists of two main parts, the Transducer and the Interface Electronics. The Biological samples which are to be analyzed could be from various sources and various forms. Usually these samples has to be pre-processed to separate certain Biological elements (e.g. Blood plasma and serum) and made into fluid form before introducing them to the Transducers.

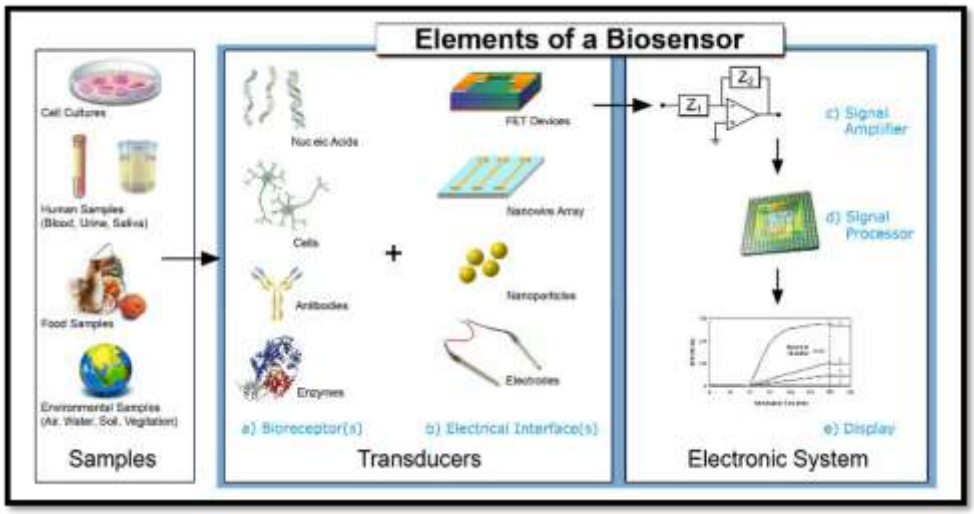


Figure 1.1: Elements of a Biosensor System

As shown in Figure 1.1 [Ref. Wikipedia.org], the Transducer part of a Biosensor contains some form of Micro/Nanostructures which can be directly interfaced with the Electronic circuits. The Biological recognition elements (also called the Bio-receptors) are bonded over the surface of these structure to form a complete transducer.

Here onwards the Transducer element would be called as 'Biosensor' for simplicity in this whole report unless otherwise mentioned explicitly. Also the focus of this work is in the Design, Process Development and Fabrication of the Biosensor (i.e., the Transducing element).

1.3 Micro and Nano scales for Biosensors

A Biosensor is usually a Micro or Nano scale device.

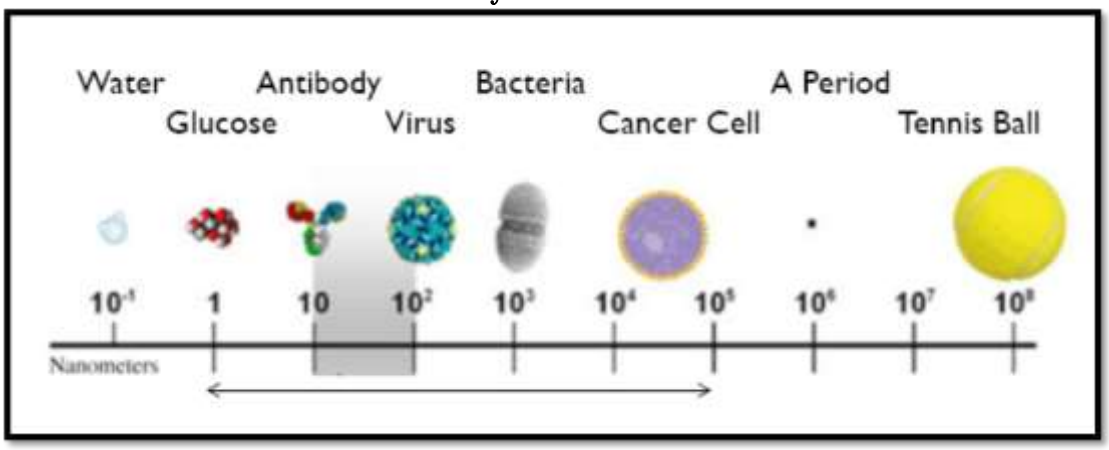


Figure 1.2: Dimensions of various Bio-molecules and Micro-organisms [40]

Biosensors are used in detecting the Biological entities like DNA, Proteins, Antibodies, Cells and also the micro-organism like Virus & Bacteria. Figure 1.2 represents the typical dimensions of these entities over a Nanometer scale. These dimensions are less than a micro-meter (µm). Reducing the sensor dimension to the size of the target elements could increase the sensitivity to the level of detecting a single molecule per sensing element.

One important property of Nano-scale materials is the increased surface to volume ratio. That is the amount of atoms of a material exposed over the surface is larger than its bulk. As the Surface atoms are more, the probability of adsorbing the target bio-molecule on one of them is very high. This in turn results in higher sensitivity and speed of detection in a Biosensor.

Apart from the above mentioned points, the typical concentration of Biomolecules in an analyte is less than a µM ranging as less as a atto M.

$$(1\ \mathrm{M} = 6 \times 10^{23}\ \mathrm{Molecules/liter} = (1 \times 10^{15}) \ /\ (100\mu\mathrm{m})^3\ )$$

At these concentrations, the job of a Biosensor is as equal to searching a grain of salt in Several large sized swimming pools. Hence a large surface area to volume of a Nano Biosensor greatly improves its sensitivity and speed in detecting the Biomolecules at lower concentrations. Also the other advantages are, reduced amount of samples, chemicals, reagents, higher integration density, lower transportation delay and the possibility of using in vitro (outside living beings, e.g. Cell culture) and in vivo (inside living beings, e.g. Implantable devices).

1.4 Classifications of Biosensors

Biosensors can be classified based on various traits like their application, Transduction type And the type of Bio-receptor. Apart from this, Biosensors can also be classified as Labeled and Label-free types. In Labeled type Biosensors, the analyte is attached with a chemical group or a nanoparticle for an amplified response of a sensor. The attached molecule or the particle usually has one of the properties; Chemiluminescence, Fluorescence, Radioactivity or Magnetism. These properties reduce the unambiguous signals and improve the sensitivity. Labeling the analyte is not cost effective and it alters the real properties of the molecules to some extent.

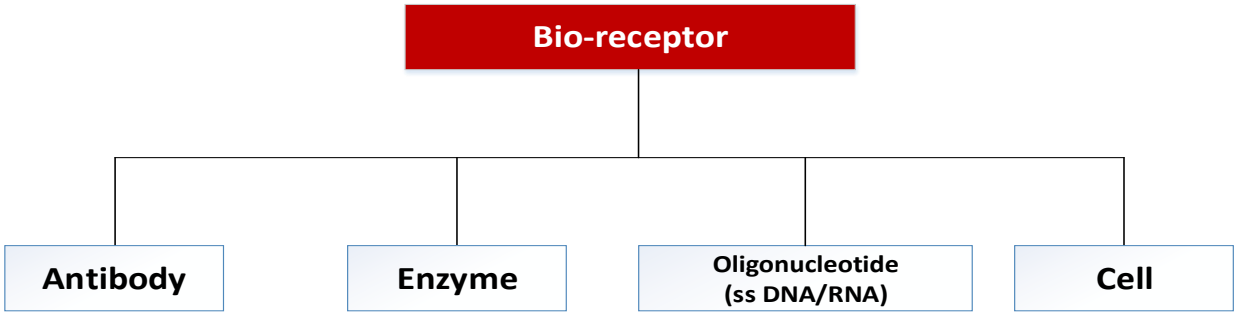


Figure 1.3: Classification of Biosensors based on Bio-receptor

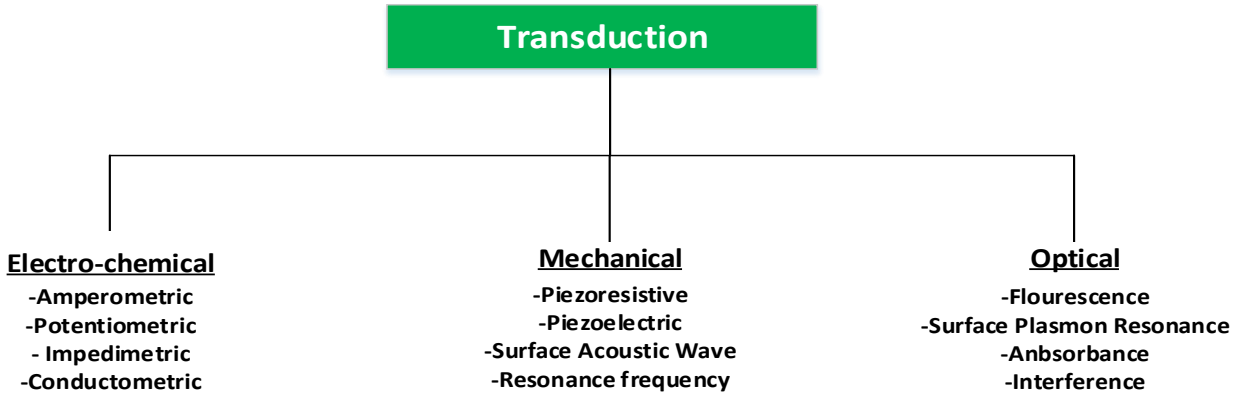


Figure 1.4: Classification of Biosensors based on Transduction

In Label-free Biosensors, the interaction of the target molecules can be directly transduced into an Electrical signal. In this work we focus on a type of Mechanical Biosensor which depends on the stress developed over the sensing surface by the adsorbed target biomolecules. These are called Surface-stress based Biosensors and are Label-free type.

The other category of Mechanical Biosensors depends on the change in the mechanical resonance frequency due to the mass of the adsorbed target biomolecules. They are very useful in measuring the exact quantities adsorbed and also in measuring the molecular mass in research labs.

1.5 Bio-receptors

The selective sensing of a particular Bio-molecule in an analyte which contains various other elements is due to the Bio-receptors embedded in the Biosensors. The Bio-receptors are nothing but some type of Biomolecule or any other Biological element which has the capacity to specifically bind to a target molecule. The most common and important Bio-receptors are Antibodies, Antigens, Enzymes and Single Strand DNA/RNA. However there are other types of Bio-receptors as well.

Antibodies and Antigens

Antibodies are large Y shaped proteins which are used by immune system to identify and neutralize foreign bodies like Virus and Bacteria. They have binding sites which helps in very specifically binding to Antigens using an interaction similar to lock and key. Both Antibodies and Antigens can be used as Bio-receptors depending on the type of target biomolecule.

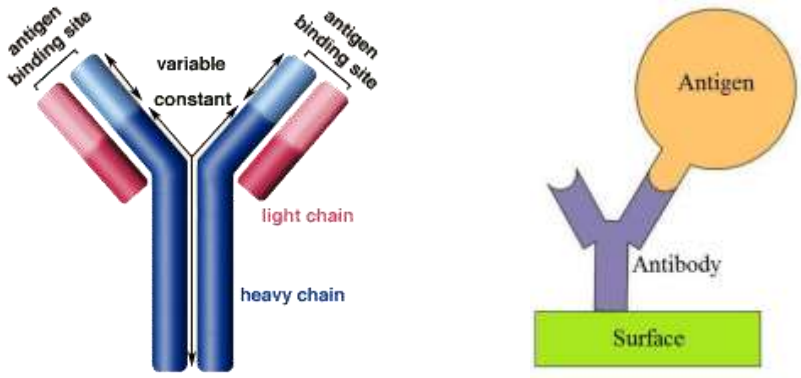


Figure 1.5: Antibody Structure & Antibody-Antigen binding

Enzymes

Enzymes are large protein molecules which acts as catalysts in chemical reactions. They are used as Bio-receptors due to their specific binding nature and catalytic properties.

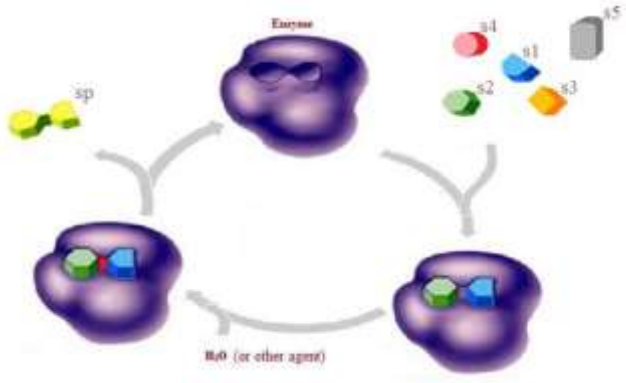


Figure 1.6: Enzyme reaction

Single Strand DNA/RNA

Single Strand DNA (DeoxyriboNucleic Acid) or RNA (RiboNucleic Acid) can selectively bind with another Single Strand of DNA or RNA by a process called Hybridization. DNA and RNA form the basic building blocks of Genetics. They are made up of 4 chemical bases, Adenine (A), Guanine (G), Cytosine (C) and Thiamine (T). The binding chemical pairs are A-T and G-C.

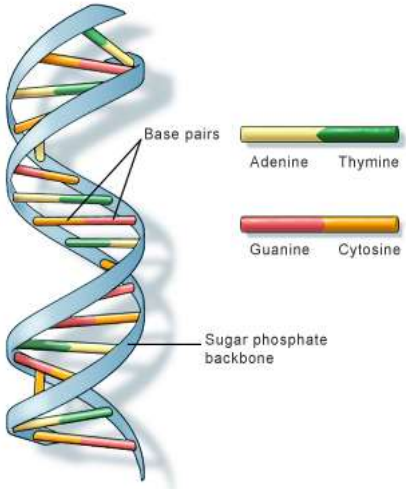


Figure 1.7: DNA molecular structure

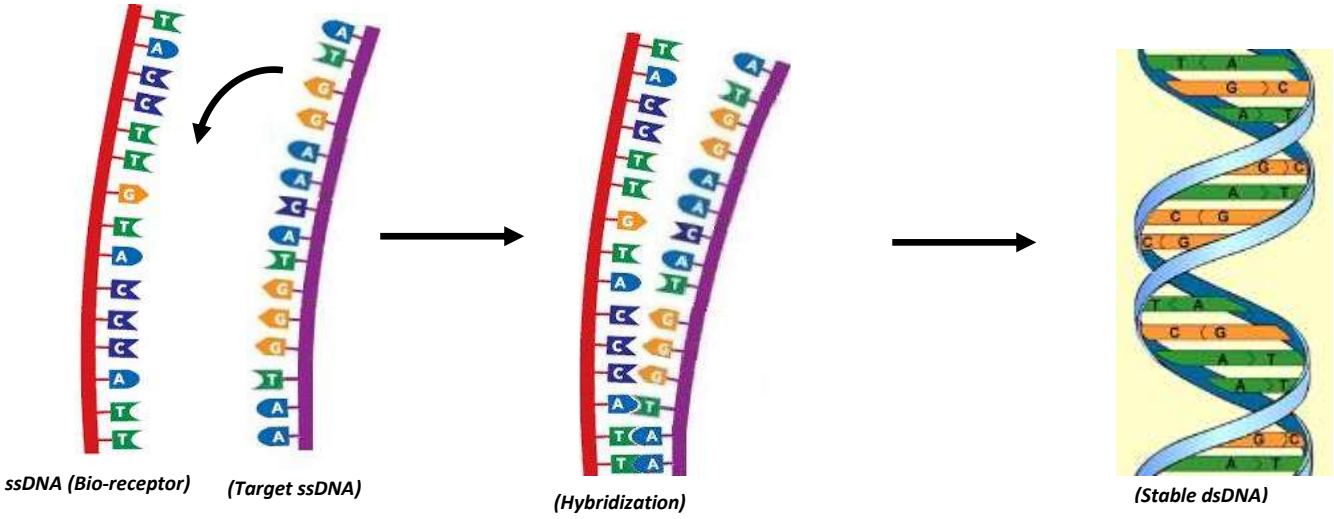


Figure 1.8: DNA hybridization process

Bio-receptors are integrated over the sensing surface by Chemical or Physical methods. These methods are commonly termed as Bio-functionalization or Immobilization process.

1.6 Applications of Biosensors

Biosensors has wide range of applications from Nano-Biotechnology research, Homeland protection, Extra-terrestrial life exploration in Space, Early disease detection, Point-of-Care diagnosis, Environmental monitoring etc.

Chapter 2

Motivation and Project Objectives

2.1 Motivation

In the year 2012, a publication on a Supersensitive Biosensor [1] appeared in Proceedings of the National Academy of Sciences of the USA. The motivation for this thesis work is derived from this paper. The publication detail is as follows.

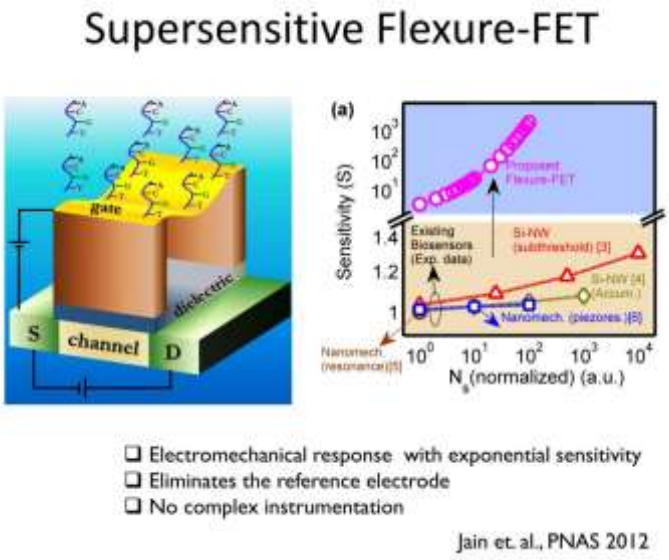


Figure 2.1: Flexure-FET Biosensor Device & its Sensitivity characteristics[1]

Jain, P. R. Nair, and M. A. Alam, "**Flexure-FET biosensor to break the fundamental sensitivity limits of nanobiosensors using nonlinear electromechanical coupling**," Proceedings of the National Academy of Sciences of the USA, vol. 109, no. 24, pp. 9304-8, Jun. 2012.

This publication by a group of researchers in Purdue University proved mathematically that the proposed Flexure-FET Biosensor has Ultra-sensitivity in comparison to Piezo-resistive Nano-cantilevers and Silicon Nanowire Biosensors.

2.1.1 Basics of Flexure-FET Biosensor

Flexure-FET Biosensor is a Surface-stress based Label-free type sensor. This device consists of a MOSFET with the Gate terminal suspended over a micro-beam which could deflect from it neutral position due to the surface stress induced by the target biomolecules.

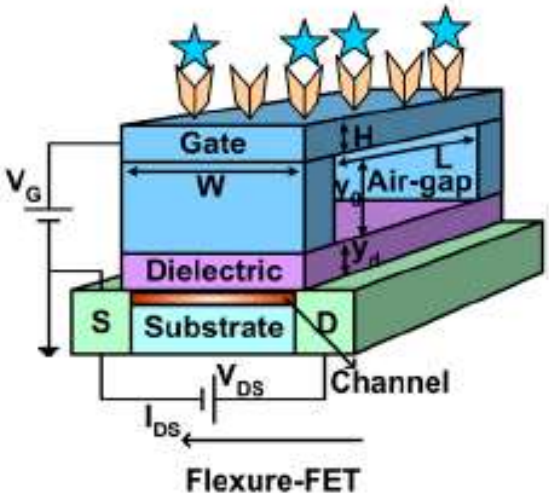


Figure 2.2: Artist's view of a Flexure-FET based Biosensor [1]

The figure shows the structure of a Flexure-FET device. The sensitivity of this Biosensor is exponentially related to the concentration (Ns) of the adsorbed target biomolecules. It is proven that this high sensitivity is due to the coupling of two highly non-linear electromechanical responses. They are, (i) Spring softening: Non-linear reduction in stiffness with applied Gate bias (Vg) and (ii) Sub-threshold electrical conduction of MOSFET which is exponentially related to surface potential.

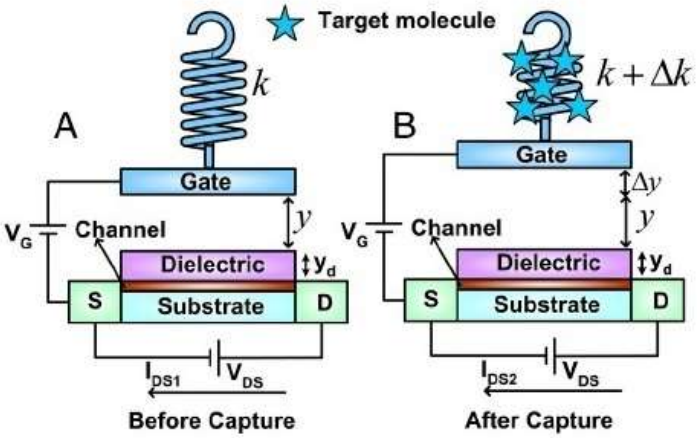


Figure 2.3: Position of Gate before and after Biomolecule adsorption [1]

As shown in the Figure the air gap between the suspended gate and the dielectric varies due to the surface-stress deflection. This in turn can be sensed as an exponential variation in the FET's Drain Current (I_D) in Sub-threshold region of operation.

$$S_{Flexure} \sim e^{(\gamma_1 \sqrt{N_s} - \gamma_2 N_s)} \quad [1]$$

Whereas the Piezo-resistive Nano-Cantilevers and Silicon Nanowire based sensors has either Linear or Logarithmic variation in the sensing parameter.

$$S_{classical} \sim \gamma_3 N_S \text{ or } \gamma_4 \ln(N_S), \quad [1]$$

We could see from the Figure 2.3 that the Micro-beam and the MOSFET's Substrate forms a MOS Capacitor with an additional Capacitor in series whose dielectric is air. The goal of this work is to Design and Fabricate this Micro-beam based Capacitance structure excluding the FET part. Fabricating only the Capacitive MEMS part would help in optimizing the processes and in preliminary validation of the sensor.

Also, the Capacitive form of coupling a mechanical response to an Electronic system is the most preferred method in integrating CMOS and MEMS as they do not involve any special materials (e.g. Piezo elements). Hence, the efforts to realize a Flexure-FET Biosensor would prove worthy for various other applications as well.

2.2 Project Objectives

The project objectives has wide range of activities from literature survey to fabrication of the sensor. The following are the key objectives,

- Literature survey on, (i) Surface stress based Biosensors
  (ii) Bio-functionalization.
- CMOS compatible Bio-functionalization process development, experimentation & characterization.
- CMOS compatible Fabrication of the MEMS part of Flexure-FET Biosensor.

Further chapters of this report addresses these project objectives. The MEMS part of Flexure-FET Biosensor is a device excluding the Insulated Gate Field Effect Transistor part. So it is a Capacitor like structure with one of its plate with the ability to deflect due to Surface-stress during Molecular interaction. This structure will be called as "Capacitive Micro-beam Structure".

Chapter 3

Fundamentals of Surface Stress based Biosensors & Literature review

3.1 Fundamentals of Surface Stress

Josiah Willard Gibbs defined Surface stress as the amount of reversible work per unit area needed to elastically stretch a pre-existing surface [2].

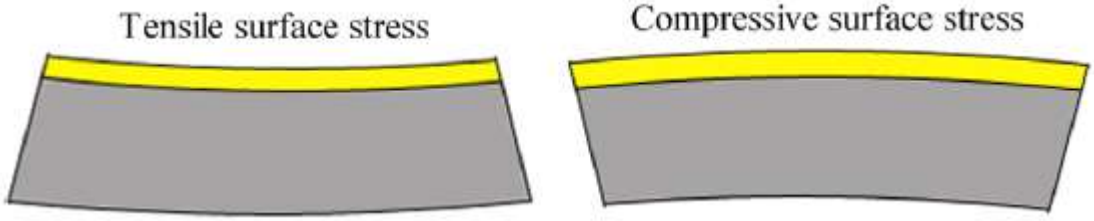


Figure 3.1: Directions of deflections due to different type of Surface stress [2]

The source of surface stress could be due to multiple reasons. It could be due to change in density of surface atoms of a thin film. In Microstructures made of multiple layers of materials, the bond strength between the atoms of different layers create surface stress. If the bond strength of a layer is stronger than that between the sub-surface atoms, a tensile surface stress develops, resulting in a concave curvature of the structure. On contrary, if the atoms of a layer repel each other or has less bond strength than that between the sub-surface atoms, a compressive surface stress develops, resulting in a convex curvature of the structure [18].

The difference in Coefficient of Thermal Expansion (CTE) of various layer of materials also can create tensile or compressive surface stress (generally unexpected and called residual stress), which is a common problem in MEMS design. In the case of Biosensors, the species (e.g. ssDNA, Antibodies etc.) adsorbed over the sensing surface, could either get attracted (usually due to van der Walls force) or repelled (usually Electro-static force) which in turn creates a surface stress [18-19]. The surface stress developed is directly proportional to the concentration of the adsorbed biomolecules. Hence a measurement of this stress by suitable transduction could help in quantifying and sensing.

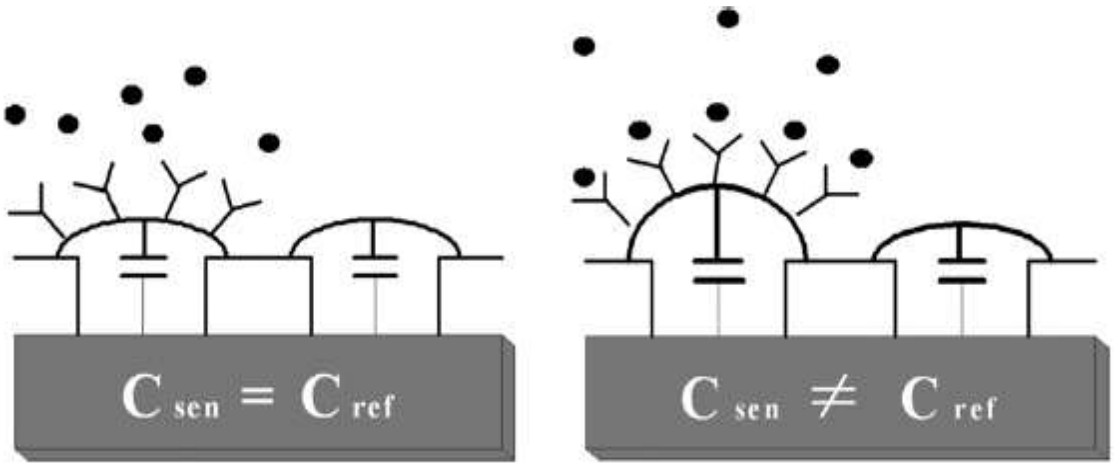


Figure 3.2: Illustration of Surface stress due to Antibody-Antigen interaction over a Capacitive Micro-beam or Membrane like structure [3]

3.2 Microstructures for Surface Stress Sensing & their Mechanics

Cantilevers, Beams (or Bridges) and Membranes are the structures used in various sensors depending on the applications, transduction method and sensitivity. The most basic and popular form of a MEMS structure in sensing is the Cantilever. A Cantilever is a beam hanging with support only at one end of it.

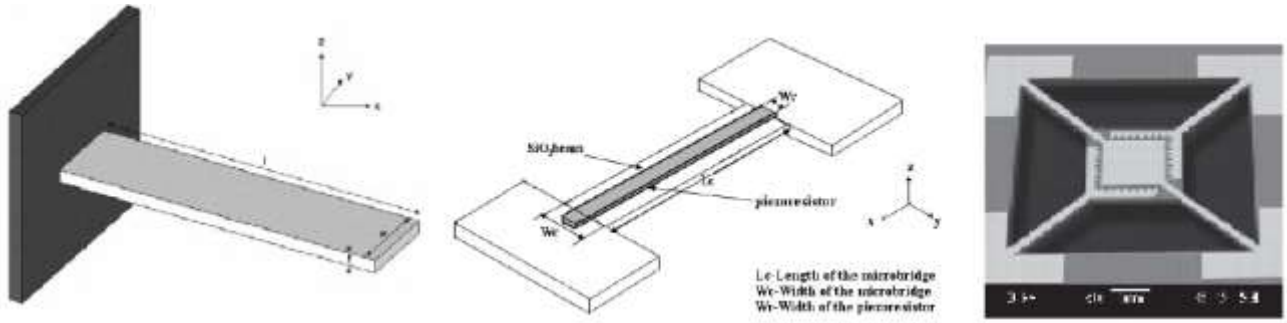


Figure 3.3: Cantilever, Beam (or Bridge) and Membrane structures [4]

In case of Surface Stress based sensors, the Cantilevers bend due to the difference between the surface stresses in either sides of it. It is essential in any Surface stress sensors to have this difference of stresses and hence only one side of the surface is modified or functionalized to achieve this difference. The change in surface stress in a Cantilever based sensor after adsorption of molecules is given by Stoney's equation [5].

$$\Delta g = \frac{E\Delta h}{4(1-\nu)}\left(\frac{t}{L}\right)^2, \quad [5]$$

Where, ν is the Poisson's ratio of the material, E is Young's (elastic) modulus of the cantilever material, Δg is the difference in surface stress, Δh is the Cantilever deflection and t & L are the thickness & the length of the cantilever, respectively. The surface stress also can be thought of as a change in surface energy density or a change in surface tension. But this simple form of Stoney's equation assumes that the material is homogeneous, isotropic and linearly elastic. Hence more advanced models are needed for structures made up of multiple materials and layers. There are various models developed for replacing Stoney's relation for crystalline materials to polycrystalline materials [6,17].

However, a more accurate analysis could be done for case to case basis using Numerical simulations based on Finite Element Analysis (FEA). Some of the FEA software tools are ANSYS, COMSOL Multiphysics and Coventorware. Practical MEMS structures are designed using these FEA tools. Wendong Zhang et al.[3] has optimized a Micro-membrane surface stress sensor made up of PDMS (Polydimethylsiloxane) and Gold layers for the best performance using ANSYS tool. Yanqing Lu et al. [4] has simulated a SiO2 and Si bi-layer based piezo resistive Micro-bridges (fix-fix beam) using Coventorware. Youzheng Zhou et al. [7] has designed and analyzed the Micro-cantilever for practical usage with the help of FEA tools. Hence, Multiphysics based simulations are mandatory and play significant role in designing accurate MEMS bases Sensors and Systems.

3.3 Material Selection for Surface-Stress based Biosensor

We can infer from Stoney's equation that a material with lower Young's modulus and Poisson ration can significantly improve the deflection for a given amount of change in stress. This higher deflection leads to higher sensitivity. Also for Biosensing applications, the materials used needs to be inert to Bio and Chemical fluids for improved shelf life of the sensor. Although Silicon CMOS materials dominate the conventional MEMS domain, certain special polymer and soft materials are popular in BioMEMS and Lab-on-a-Chip areas. SU-8, PDMS and Paralyne are the popular soft materials in BioChip fabrication. These materials have good Biocompatibility, i.e. inert to Biochemical environment.

Of these materials SU-8 is a popular epoxy based Negative tone photoresist invented by IBM, which is very useful in fabricating Biosensor devices. SU-8 can be directly patterned and structured using a UV Lithography process at low processing temperature (less than 100°C). These properties of SU-8 make it CMOS compatible as well. We can fabricate SU-8 based micro-

structures over the BEOL (Back End Of Line) layer of a commercially manufactured CMOS Integrated Circuit, without disturbing or altering the properties of underlying Electronics. The mechanical properties of SU-8 are given in Table 3.1 [8].

Table 3.1: Mechanical Properties of Popular MEMS Materials

COMPARISON OF MECHANICAL PROPERTIES OF VARIOUS MATERIALS

Properties	Polysilicon	Silica	Si_3N_4	SU8
E (GPa)	160	73	315	4.4
ν	0.22	0.17	0.27	0.22
ρ (kg/m^3)	2230	2200	3184	1190
k (N/m)	0.02	7.85×10^{-3}	0.03	4.32×10^{-4}

Where, E is Young's modulus, ν is Poisson ratio, ρ is material density and κ is the spring constant. From this it is evident that a SU-8 based cantilever can be more sensitive than its Silicon CMOS counterparts. The typical range of surface stress change due to Biomolecular interaction on a Cantilever surface is in the range of 1mN/m to 1N/m [9].

There are many works on SU-8 based Surface-stress Bio and Chemical Sensors reported in literature [10-16]. SU-8 is Optically transparent material and hence an optical read out method is suggested in [12]. SU-8 can also be made electrically conductive by suspending Conductive Nanoparticles [10] [20-2] or by adding Conductive polymer [23,27]. This conductive nano-composite can be easily photo-patterned to fabricate piezo-resistors and other conductive microstructures. However smaller structures will be difficult to realize due to high viscosity and density of SU-8 composites. SU-8 surface can also be modified to achieve Hydrophilicity for Microfluidic transport [24].

SU-8 can be easily surface micromachined or be released from a substrate using some simple sacrificial materials and wet processes [24]. But in case of Silicon and other CMOS materials, expensive and time consuming process like Dry etching or corrosive wet etching methods are necessary. SU-8 surfaces can also be Bio-functionalized using simple processes which will be addressed in upcoming chapters. Hence SU-8 can be used to fabricate Sensors, support structures and in packing Biosensor devices.

Chapter 4

Fundamentals of Bio-functionalization & Literature review

4.1 Introduction to Bio-functionalization

Bio-functionalization is a process in which a material surface is modified so as to have a Biological function or reaction. It is done by immobilizing Bio-receptors over the surface by one of the following processes, (i) Adsorption, (ii) Physical entrapment and (ii) Covalent binding.

In Adsorption, the functionalized layer is simply laid over the sensor surface by van der Waals forces and hydrogen bonds. Adsorption is a poor technique of Bio-functionalization. In Physical entrapment, the receptor molecules are polymerized along with a polymer. Hence the receptors are entrapped in a matrix of polymer material. Covalent binding is the most preferred and well known technique for Bio-functionalization. In this method, the Bio-receptor is covalently attached to the sensor surface. Since the covalent bond is the strongest one, the Bio-functionalized surface would be more stable without denaturing.

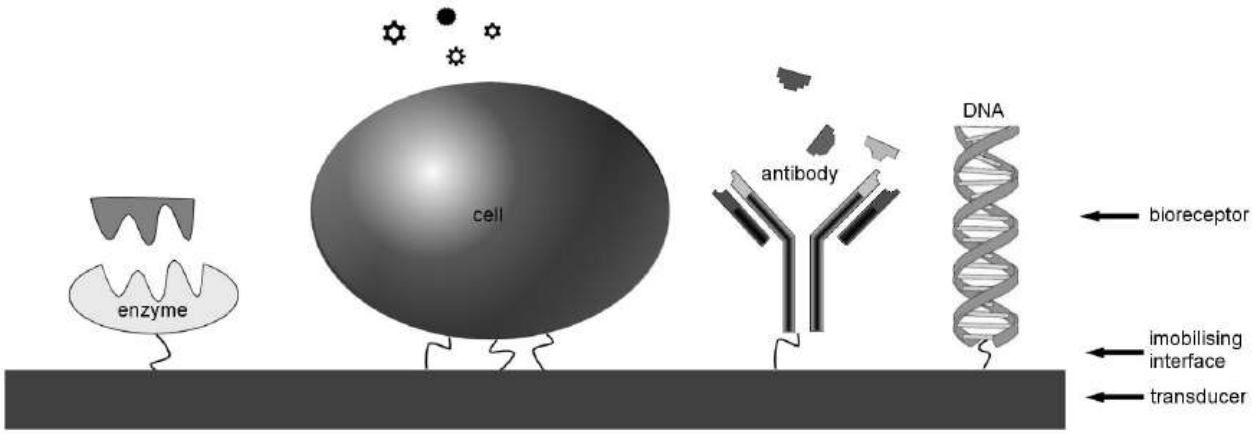


Figure 4.1: Type of Bio-receptors which could be immobilized on a surface [25]

Only covalently linked Molecular layer can induce a strong sur stress in the Micro-structures and hence it is essential to adapt covalent binding for Surface-stress based Biosensors.

4.2 Desired properties of the Bio-functionalization Processes and Materials

Although Bio-functionalization processes are developed by Bio and Chemistry fraternities, it has to satisfy the Engineering requirements in order to successfully manufacture a useful device. The following are the properties desired by a Microelectronic Engineer with regards to Bio-functionalization.

The materials, chemicals and processes needs to be CMOS compatible. A CMOS compatible material does not induce ions or other contaminant atoms in to Electronic devices during manufacturing. Similarly a CMOS compatible process does not harm or change the properties of the Semiconductor devices. A low temperature and low pressure process often meet this requirement. Apart from this the material needs to be Biocompatible and have longer shelf life. As we seen in the previous chapter, SU-8 polymer has all these properties and hence an ideal candidate for Biosensor fabrication. A layer of SU-8 can passivate any surface and protect it from Bio and Chemical environments. This naturally makes the whole Bio-functionalization process CMOS compatible.

4.3 SU-8 Bio-functionalization principles and techniques

At the very basics, Bio-functionalization is a chemical binding process. The material surface which is to be bio-functionalized, either naturally has some functional groups or made to have a desired functional groups to start this binding process. For example many Oxide materials has Hydroxyl (-OH) groups at the surface. This Hydroxyl groups can be used as an anchoring point to covalently bind the bio-receptors or Linker molecules which at the other end binds with the desired bio-receptor. Surface functional groups also make the surface Hydrophilic which is desirable for Microfluidic transport. Surface functional groups are obtained on SU-8 by either chemical treatment or by Oxygen plasma activation. Oxygen plasma forms CO and COO groups on the SU-8 surface and hence highly Hydrophilic. But Oxygen plasma could make SU-8 surface rough.

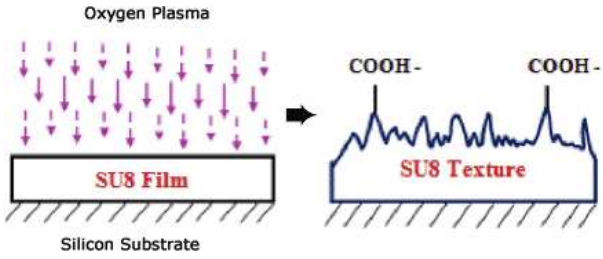


Figure 4.2: SU-8 Surface before and after Oxygen Plasma treatment [Ref. Microchem]

There are many methods of Bio-functionalizing SU-8 in literature [26-29]. As an example we would see one of the methods by Cuong Cao et al [29].

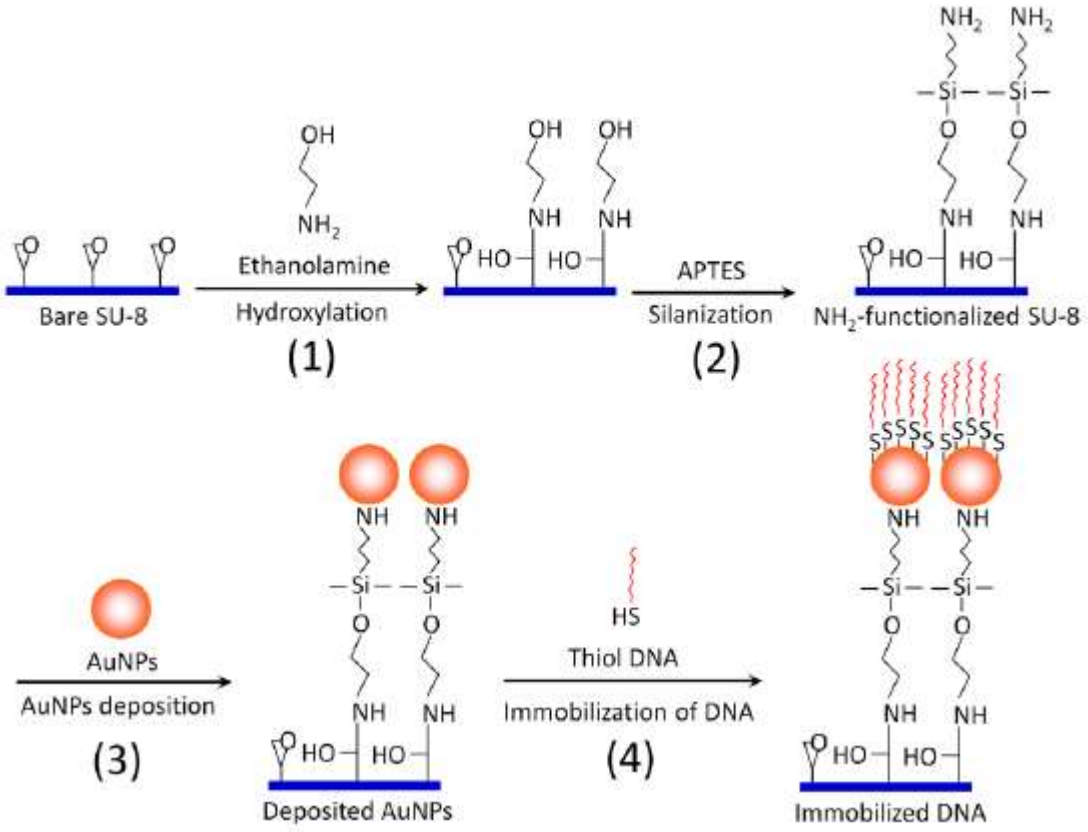


Figure 4.3: Process flow for immobilizing Thiolated ss-DNAs over SU-8 surface using APTES and AuNPs as linkers [29]

Figure 4.3 illustrates a process by which DNA strands are immobilized over SU-8 surface. A base SU-8 surface has epoxy groups which are treated with Ethanolamine resulting in a surface with (-OH) groups. Next APTES ((3-Aminopropyl)triethoxysilane), a type of Silane molecules are attached above the Hydroxyl (-OH) groups. The usage of Silane molecules help in obtaining a uniform monolayer of molecules after Bio-functionalization. This is due to the interaction between Si-O-Si linkages between the Silane molecules. The other end of APTES has Amine group (NH_2) which can further be used to attach any desired receptor particles or molecules. As already mentioned, some materials like SiO_2 and ZnO_2 has Hydroxyl groups at their surface naturally and hence chemical or Oxygen palsma treatments are not necessary for fucionalization.

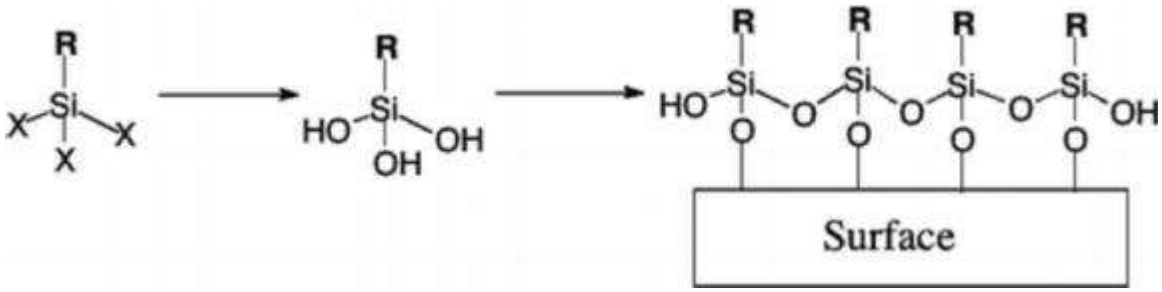


Figure 4.4: Si-O-Si reaction of Silane molecules on a Surface to form a Monolayer

SU-8 can also be functionalized by grafting other polymers over them during fabrication. There are various chemistries to achieve the same goal but choosing a facile and CMOS compatible process would be very helpful in realizing Electronic Nanobiosensors.

4.4 Issues in currently available methods for SU-8 Bio-functionalization

As we seen earlier SU-8 can be made a functional material by suspending Nano particles in them. Also can be used to fabricate Microfluidic channels and components. So, in a Lab-on-a-Chip system Sensor, Transducing element (e.g. Piezo resistor), Microfluidics, support structures and package all can be made using SU-8 itself. Microfabrication processes involving wet chemistry and evaporation are generally not selective in their reactions. The selectivity is brought in by using a protective layer of patterned photoresist or other materials like SiO_2 or Si_3N_4. Similarly the Bio-functionalization of SU-8 needs to be made selective by some techniques where Bio-receptors selectively bind to certain regions of a SU-8 surface. This would be very helpful in Wafer scale bio-functionalization in Semiconductor foundries. A new and innovative method is proposed by us and the same is elaborated in Chapter 5.

Chapter 5

CMOS Compatible Selective Bio-functionalization & Characterization of SU-8 Surface

5.1 Introduction

In this work we demonstrate a process for Selectively Bio-functionalizing SU-8 surface which is often desired for large scale manufacturing of Bio-chips in traditional Semiconductor foundries. As a proof of concept Biotin Hydrazide (a small Vitamin molecule with a residual or extra Amine group) is immobilized selectively over SU-8 surface. Fourier Transform Infra-Red (FTIR) Spectroscopy of the surface is carried out at various stages of the Bio-functionalization process.

5.2 Fundamentals of SU-8 Processing

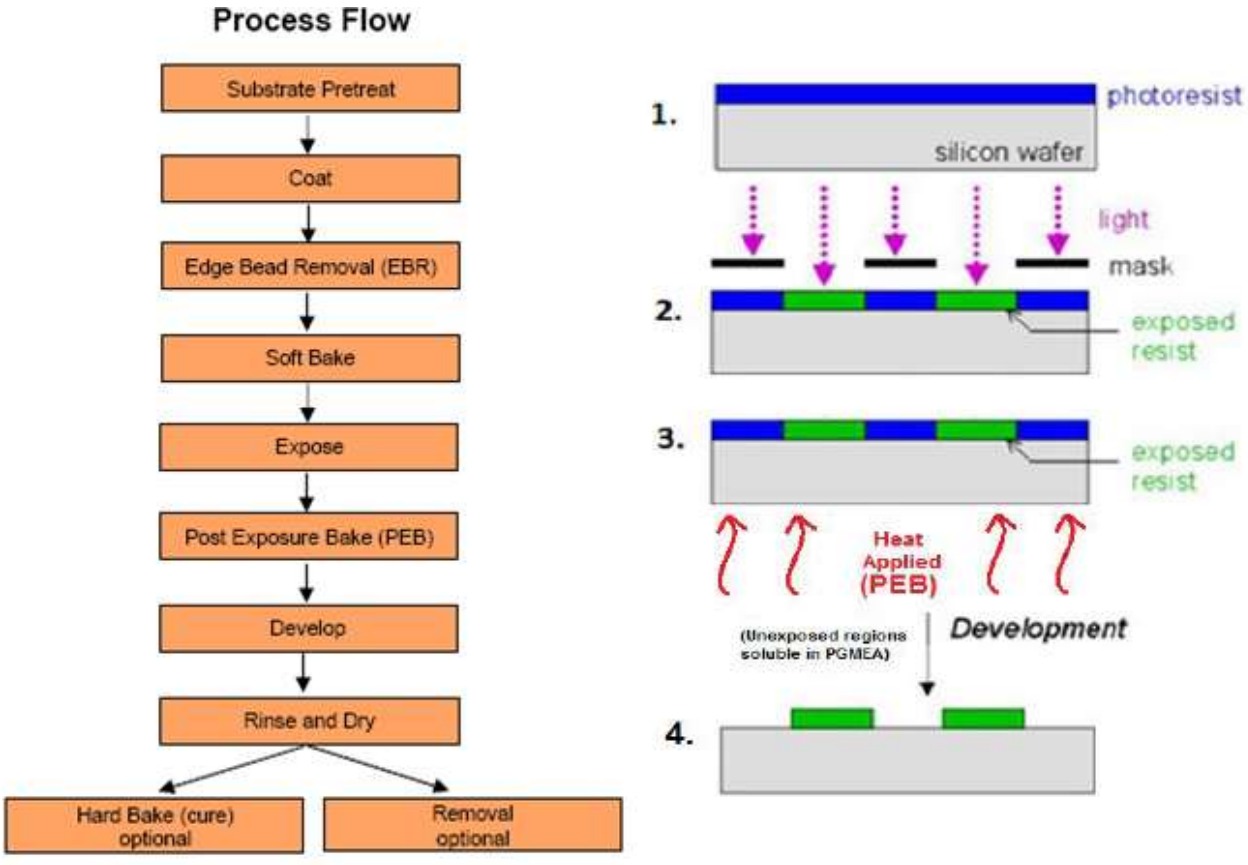


Figure 5.1: SU-8 Microfabrication Process Flow [Ref. Microchem]

Figure 5.1 shows the various process steps in fabrication of SU-8 structures. It involves 2 baking steps, UV photolithography exposure and chemical treatment. SU-8 photoresist is composed of the following 2 components.

- An epoxy, called Epon SU-8 (Shell Chemicals)
- Solvent, either gamma-Butyrolactone (SU-8 3000) or Cyclopentanone (SU-8 2000)
- A photo acid generator (Triarylium-Sulfonium salts)

The SU-8 monomer structure is showed in Figure 5.2. It contains 8 epoxy groups and hence the name SU-8.

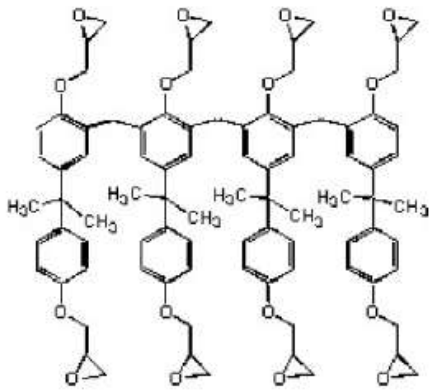


Figure 5.2: SU-8 Monomer Molecular structure

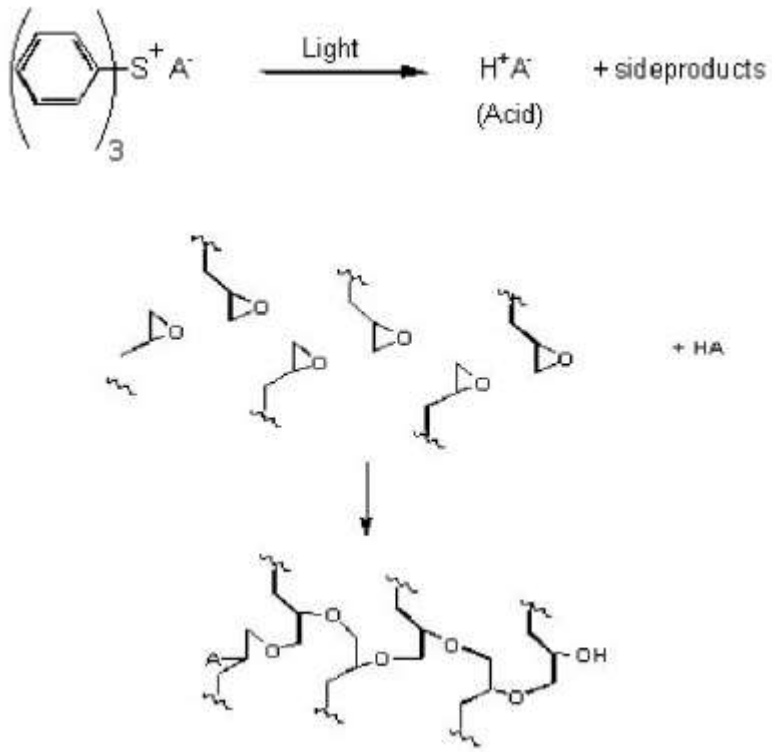


Figure 5.3: Molecular changes during UV exposure and PEB

The molecular changes which occurs during UV exposure and Post Exposure Bake is shown in Figure 5.3. The polymerization reaction is a cationic chain growth process and forms a densely cross-linked film, because the average functionality each SU-8 monomer is approximately 8. During PEB the exposed areas of SU-8 film partially crosslinks and with further heating (Hard bake), a permanent chemically and mechanically stable structure is obtained. The unexposed areas can be dissolved in SU-8 developer called PGMEA (Propylene glycol methyl ether acetate).

5.3 A Facile Process for Obtaining a Hydrophilic SU-8 Surface with Hydroxyl groups

In 2003, Chun-Lung Wu et al. [30] came out with a simple and novel method for obtaining Hydrophilic Surface. As we seen in Chapter 4 it is normally obtained by chemical treatment or Oxygen plasma treatment. But in [30] it is achieved by simply mixing an additional Hydrophilic Copolymer called Glycidol.

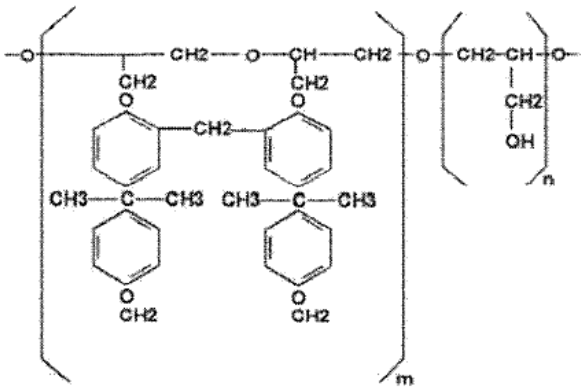


Figure 5.4: Glycidol Copolymer Structure [30]

Figure 5.4 is the molecular structure of Glycidol. It has an Hydroxyl group which makes the film made up of SU-8 and Glycidol mix (here onwards referred as SU-8+Glycidol), Hydrophilic. SU-8+Glycidol can be patterned using conventional SU-8 Photolithography process and hence involves lower temperatures only. In this work, we extend the techniques and bio-functionalize the SU-8 surface. As a proof of concept, immobilization of Biotin Hydrazide molecules is demonstrated. For achieving this APTES molecules were covalently attached to the –OH groups on the SU-8+Glycidol surface. Then the Biotin Hydrazide molecules were immobilized over this layer by using Glutaraldehyde molecules as crosslinkers. The immobilized Biotin can be used to detect Avidin or Streptavidin molecules.

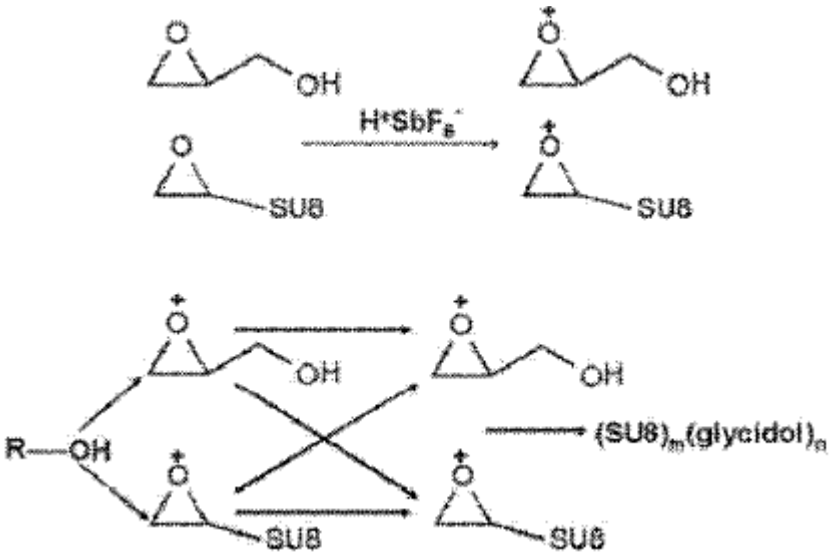


Figure 5.5: SU-8 and Glycidol Cross-linking reaction [30]

5.4 Experimental Procedure for Selective Bio-functionalization of SU-8 Surface

5.4.1 Inventory of Materials, Chemicals and Molecules needed:

The process was carried out on a Silicon wafer. The following were the chemicals used in this process. Microchem SU-8 2002, SU-8 developer (PGMEA), Sigma Aldrich Glycidol 96% (G5809), Biotin Hydrazide (B7639), Phospate Buffer Saline (PBS) tablets (P4417), Technic France high purity Dimethyl Sulfoxide (DMSO), Alfa APTES((3-Aminopropyl)triethoxysilane) 98% (Alfa A10668), Glutaraldehyde 25% aq. Solution (A17876), Toluene (high purity without moisture), Tween 20 and Isopropyl Alchohol (IPA).

5.4.2 Procedure

Initially a 4 inch Piranha cleaned Silicon wafer was diced into small pieces of about 2 cm^2 for using as substrate. These substrates were baked at 200°C on hot plate for 1 hour for completely removing moisture.

Step 1: Each sample was spin coated with SU-8 2002 and crosslinked using UV lithography. The procedure is as follows:

Spin coating rate: Step1- 500 rpm, 10 sec with acceleration of 300 rpm/sec and Step2- 3000 rpm, 30 sec with acceleration of 300 rpm/sec.

Soft bake: 65°C for 3 minutes and 95°C for 6 minutes.

Exposure: Flood exposure with UV (365nm) for 80 sec (80mJ/ cm^2) using Suss MicroTec MA6/BA6 aligner system.

Post Exposure Bake (PEB): 65°C for 3 minutes and 95°C for 6 minutes. The samples were cooled down to room temperature gradually in a time of about 1 hour to avoid internal stresses in the film.

Then the SU-8 coated samples were rinsed in IPA for dissolving uncrosslinked residues and dried using purified compressed air (CDA). This first layer of SU-8 was used as a base for SU-8+Glycidol layer which would be functionalized.

Step 2: Next a polymer mix of SU-8 2002 and Glycidol was made with a ratio of 1:0.3 (by wt. %) using micro-balance. This mixture was thoroughly probe sonicated in an ice bath for 30 minutes to avoid lumps and ensure uniformity. This mix was used to form patterned layer of SU-8+Glycidol over the pure SU-8 layer. The following steps were followed in fabricating this layer.

Spin coating rate: Step1- 1000 rpm, 40 sec with acceleration of 300 rpm/sec. This parameters helps in getting uniform film of SU-8+Glycidol polymer.

Soft bake: 65°C for 3 minutes and 75°C for 6 minutes. The flash point of Glycidol is 81°C and hence the highest temperature used was 75°C.

Exposure: Exposed using mask with square patterns ($1cm^2$) with UV (365nm) for 200 sec using Suss MicroTec MA6/BA6 aligner system. This dosage was chosen as the film thickness is larger than pure SU-8 2002.

Post Exposure Bake (PEB): 65°C for 3 minutes and gradually increased to 95°C in 15 minutes and holded at the same temperature for 6 minutes. The samples were cooled down to room temperature gradually in a time of about 1 hour to avoid internal stresses in the film. In PEB, 95°C was used as it is necessary for the SU-8 monomers to crosslink. However, gradual temperature raise is important, so that the SU-8 and Glycidol's epoxy structures open up and corsslinks to form a stable polymer matrix below the flash point of Glycidol.

Later the samples were developed using SU-8 developer (PGMEA), vigorously washed in IPA and dried using CDA. Contact angle measurement was done using goniometer and a lower angle (Approx. 65°) was observed on this layer than a pure SU-8 2002 (Approx. 78°) layer due to the presence of Hydroxyl groups on the sufrace [30].

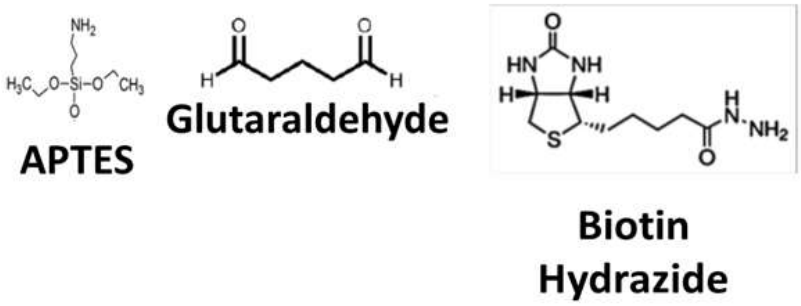


Figure 5.6: Structure of APTES, Glutaraldehyde and Biotin Hydrazide Molecules

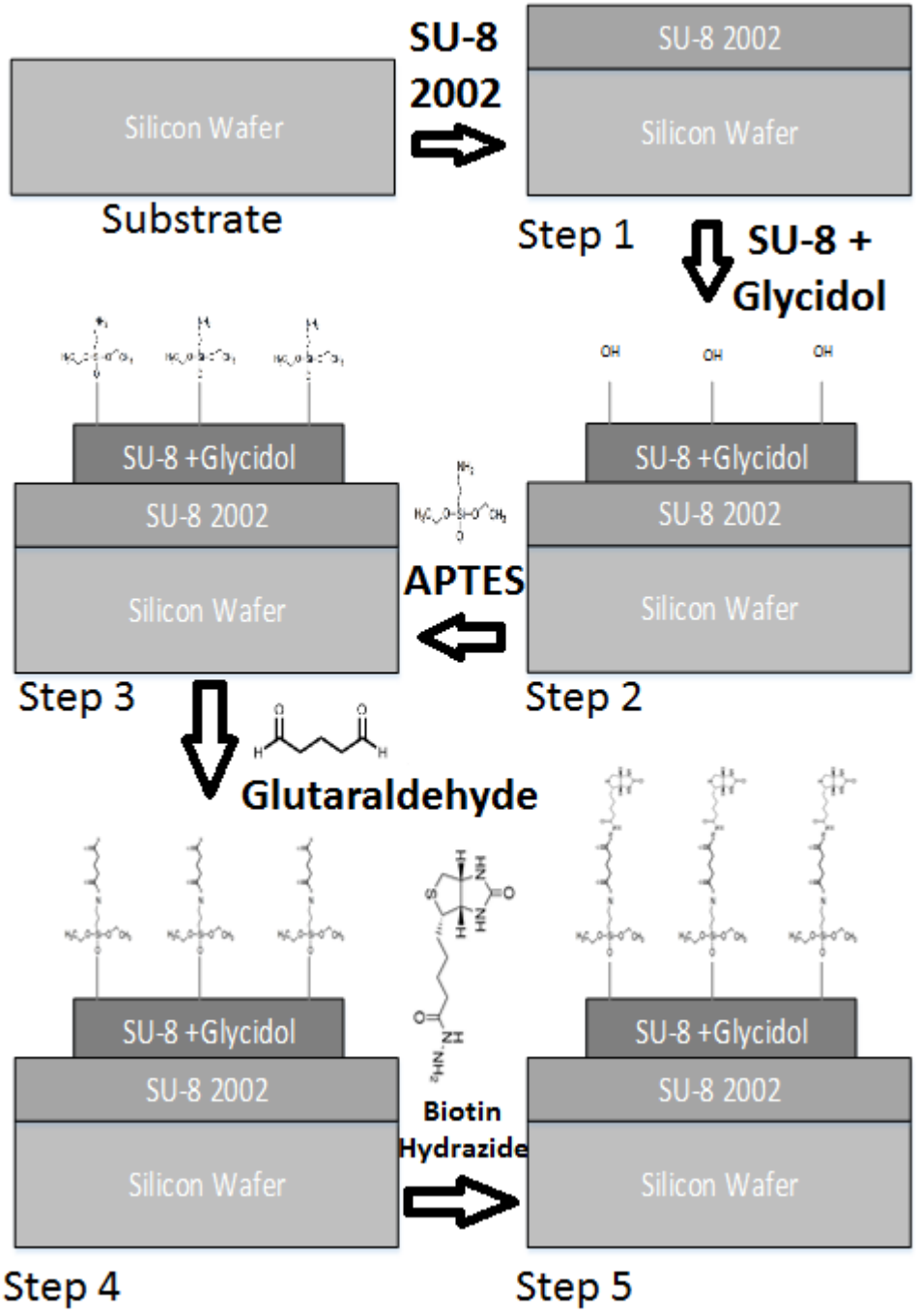


Figure 5.7: Process sequence for Selective Bio-functionalization of SU-8 surface

Step 3: APTES solution was prepared using Toluene as solvent with a concentration of 2.0% (v/v). Toluene was chosen as it supports in forming covalent bonds between APTES and –OH groups on the SU-8+Glycidol surface [31]. The samples coated with SU-8+Glycidol layer and one sample with only pure SU-8 layer were incubated in this solution for 2 hours at room temperature. Then the samples were thoroughly washed in pure Toluene to detach the unbonded APTES and dried with CDA.

Step 4: Next the samples were smeared with Glutaraldehyde solution and left for 2 hours at room temperature. Later the samples were washed using copious amout of DI water and dried using CDA.

Step 5: Further step is to immobililze Biotin Hydrazide over the surface. Biotin Hydrazide powder is dissolved in DMSO(1mL)+PBS(9mL) buffer (pH ~ 7.4) solution with a concentration of 0.5mg/mL. The samples were incubated in this Biotin Hydrazide solution for 12 hours at room temperature. Then the samples were washed thoroughly in PBS+Tween 20 solution to remove excess and non-specifically adsorbed Biotin Hydrazide molecules. The procedure for preparing the PBS+Tween 20 solution is as follows, 1 PBS tablet was dissolved in 200mL DI water to obtain a solution of pH 7.4 which is used for Biotin Hydrazide solution. Tween 20 (0.5% v/v) was mixed in this PBS solution to use for washing purpose.

5.5 Results and Discussions

A qualitative Fourier Transform Infra Red (FTIR) spectroscopy analysis to study the process was carried out using Bruker TENSOR37 system. FTIR Transmittance spectra characteristics were studied on SU-8+Glycidol film, Glutaraldehyde treated surface and Biotin Hydrazide immobilized surface.

A typical SU-8 film's FTIR transmittance spectra has epoxy-group and solvent signatures between 700 to 1800 cm^{-1} [32]. The Figure 5.8 is the spectra of the SU-8+Glycidol film which shows the presence of Hydroxyl groups by decreased transmittance around 3500 cm^{-1}. These Hydroxyl groups are part of Glycidol and acts as binding sites for the APTES molecules in the crosslinked layer.

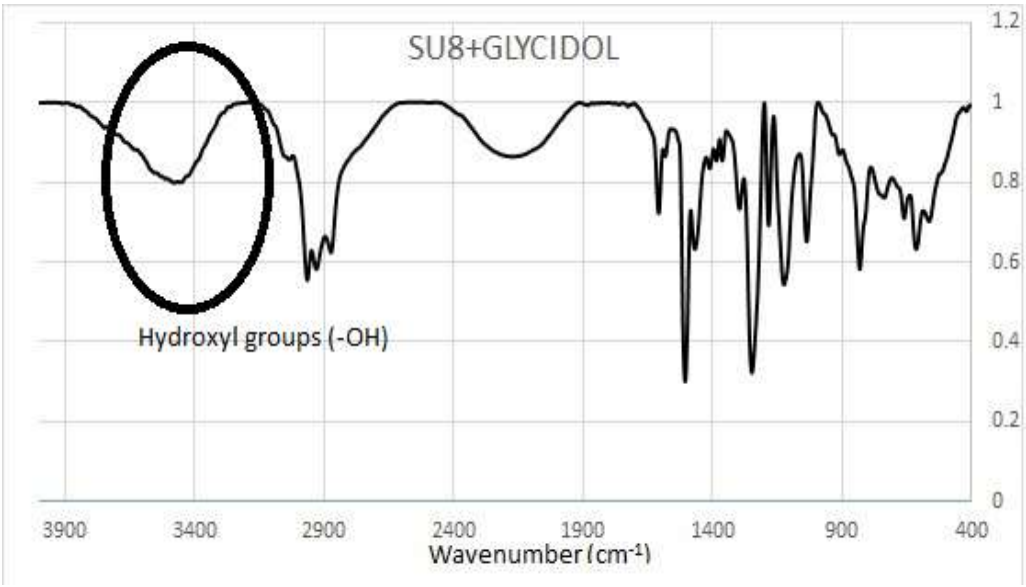


Figure 5.8: FTIR Transmittance spectra of SU-8+Glycidol film

Figure 5.9 indicates the presence of Aldehyde groups (2830–2695 cm^{-1}) after treating the SU-8+Glycidol surface with APTES and Glutaraldehyde solutions (GA curve). One of the Aldehyde groups in the Glutaraldehyde reacts with the Amine group (-NH_2) in APTES to form a bond. The other Aldehyde group of Glutaraldehyde is available for immobilizing molecules with -NH_2 at the surface.

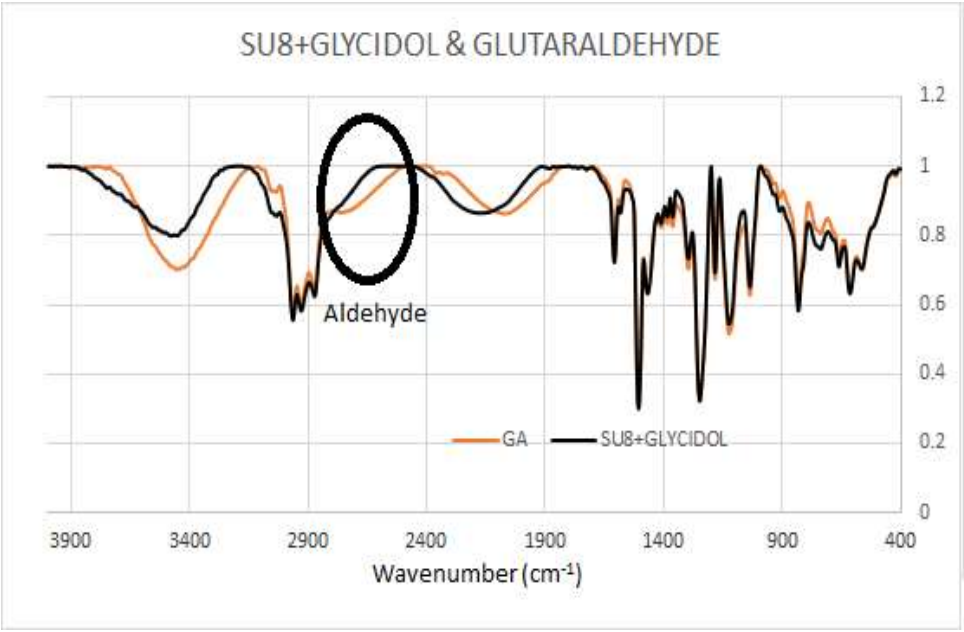


Figure 5.9: FTIR Transmittance spectrums of SU-8+Glycidol film and APTES+Glycidol (GA) treated films

The trasmittance at Aldehyde region of the spectrum has increased after immobilizing Biotin Hydrazide molecules. This can be observed in Figure 5.10.

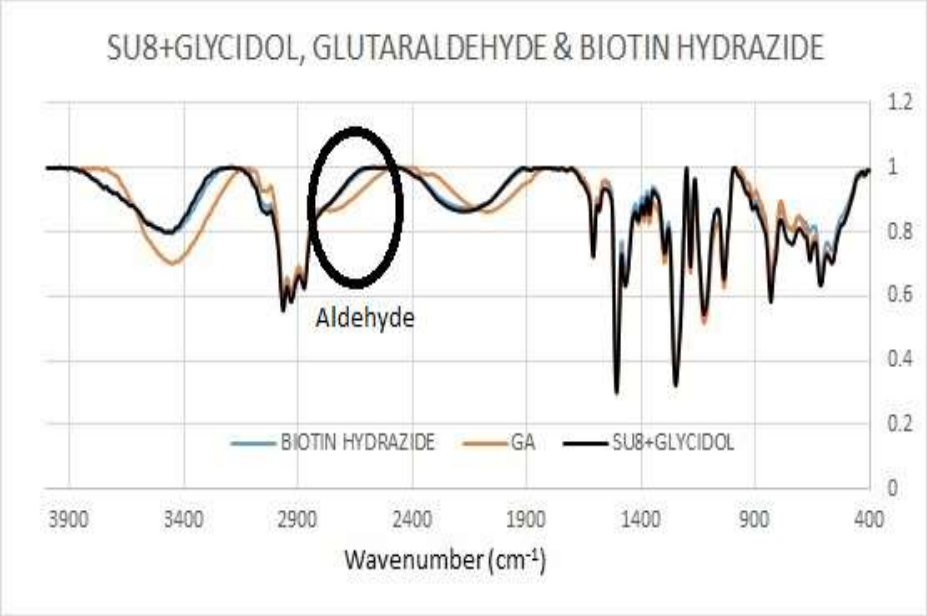


Figure 5.10: FTIR Transmittance spectrums of SU-8+Glycidol, GA and Biotin Hydrazide immobilized films

Figure 5.11 is the magnification of Aldehyde region of the FTIR spectrum. It can be observed that the Aldehyde region transmittance has increased to the level of normal SU-8+Glycidol film after Biotin Hydrazide immobilization.

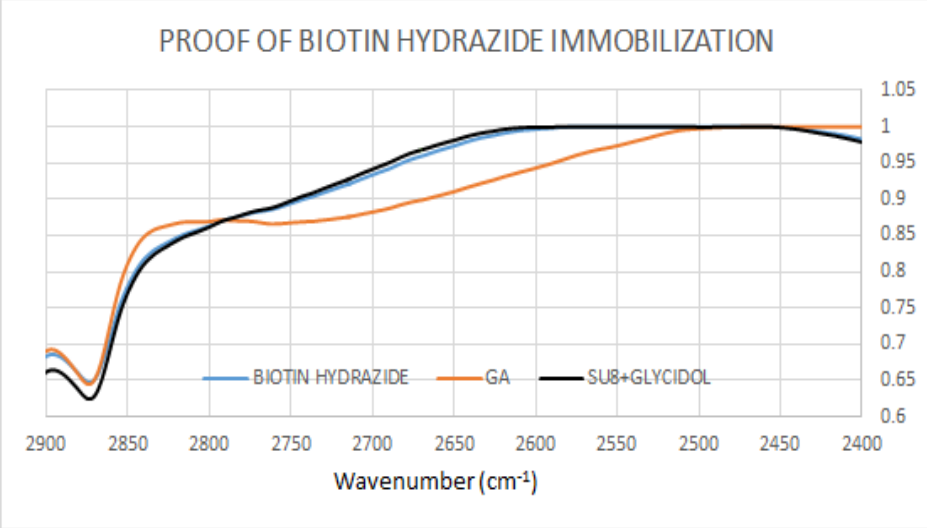


Figure 5.11: Magnified view of Aldehyde region of FTIR spectrum of SU-8+Glycidol, GA and Biotin Hydrazide

This is due to the well known fact that Aldehyde fuctional group in the Glutaraldehyde linker reacts with the Amine in the Hydrazide part of Biotin Hydrazide (in pH 6.5-7.5) to form Hydrazone bond [33]. This results in the reduction of Aldehyde signature in the FTIR spectrum. Hence we could achieve a stable Biotin functionalized SU-8 surface could be used to further immobilize other bio-molecules or sense Avidin and Streptavidin molecules.

It was also observed that the Aldehyde region transmittance has reduced only in SU-8+Glycidol regions of the sample and not in the background layer made up of pure SU-8. This is due to the lack of binding spots (-OH groups) for APTES molecules over the pure SU-8 surface. Hence selectivity in bio-functionlization has been achieved.

5.6 Facts on CMOS Compatibility

It is well known that SU-8 is chemically and biologically inert polymer after cross-linking in its pure form. A layer of SU-8 film can be used as passivation over the BEOL of ICs for protecting the underlying Electronic circuits. This layer restricts the chemicals and molecules used in the Bio-functionalizing process from diffusing into the Circuit areas. Also the processing temperature of SU-8 is less than 100°C, which does not affect the Semiconductor devices. Figure 5.12 shows an illustration of a Micro-beam based biosensing platform over a CMOS IC.

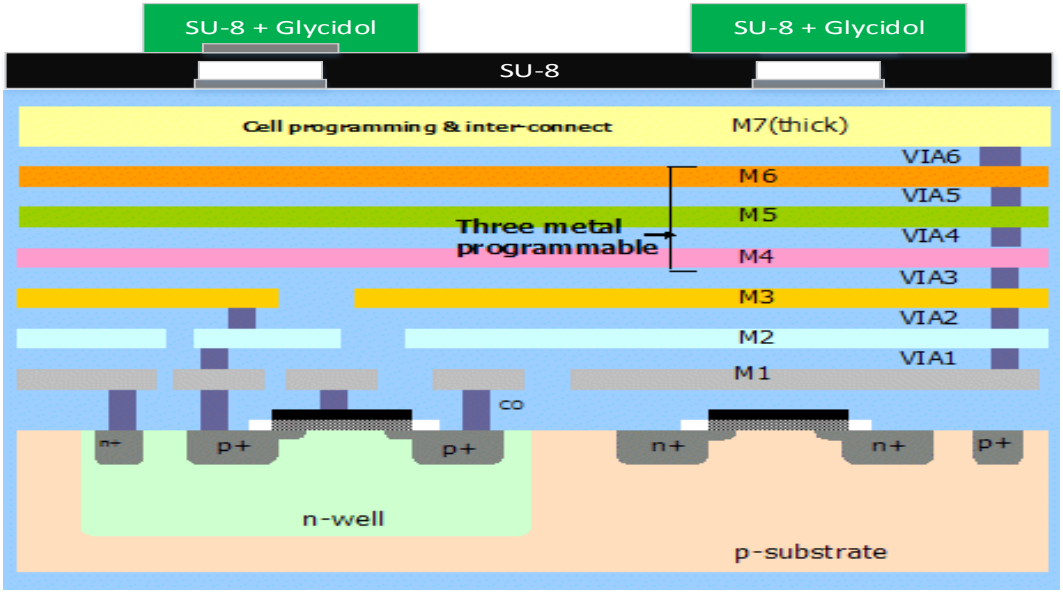


Figure 5.12 Cross section view of SU-8+Glycidol Micro-beam fabricated over the BEOL of CMOS IC by Surface Micromachining

The Black SU-8 layer acts as passivation as well as anchoring support for the SU-8+Glycidol Micro-beam. This structure can be obtained by CMOS Post processing method. Hence, the method proposed for Bio-functionalization could be CMOS compatible.

Chapter 6

Fabrication & Characterization of SU-8 based Capacitive Micro-beam Structure

6.1 Design Considerations

The structure is basically a Capacitor with one of its plate capable of deflecting due to Surface-Stress due to molecular interaction. This kind of structure mounted over a MOSFET's Gate Insulator leads to a Flexure-FET Biosensor device.

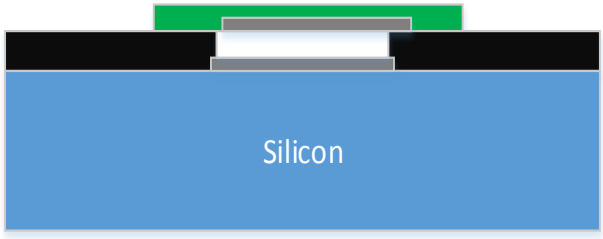


Figure 6.1: Cross-section view of Capacitive Micro-beam structure

The conductive surface for SU-8 based Capactivie Micro-beam structure is chosen to be Titanium thin-film in this work. The reason for this is the enhanced adhesion between SU-8 and Titanium [36]. This could significantly demolish the mechanical advantages of choosing SU-8 as structural material due to very high Young's modulus of Titanium. However, there are proven works [37] with membrane type sensors made up of PDMS film and Gold electrodes with good results. So we believe that using a multilayer structure of SU-8 and Titanium would not significantly spoil the performance overall. Apart from that unlike PDMS, SU-8 is directly photo-patternable.

Table 6.1: Mechanical properties of SU-8 and Titanium

Material	E [G Pa]	N
SU-8	2 - 4.4	0.22
Titanium	102 – 169.9	0.33

Also, Titanium does not Oxidize at room temperature, which is most desired for enhanced shelf life. So even a thin layer (like 20 nm) of Titanium is sufficient to provide good Electrical contact. On the other hand obtaining a very thin free standing Titanium beam could be difficult. In this case a supportive SU-8 layer could provide could flexibility and stability to the Titanium thin-film and also facilitates in Bio-functionalization.

The dimensions of the Micro-beam to be fabricated is chosen based on the FEA analysis results in [38].

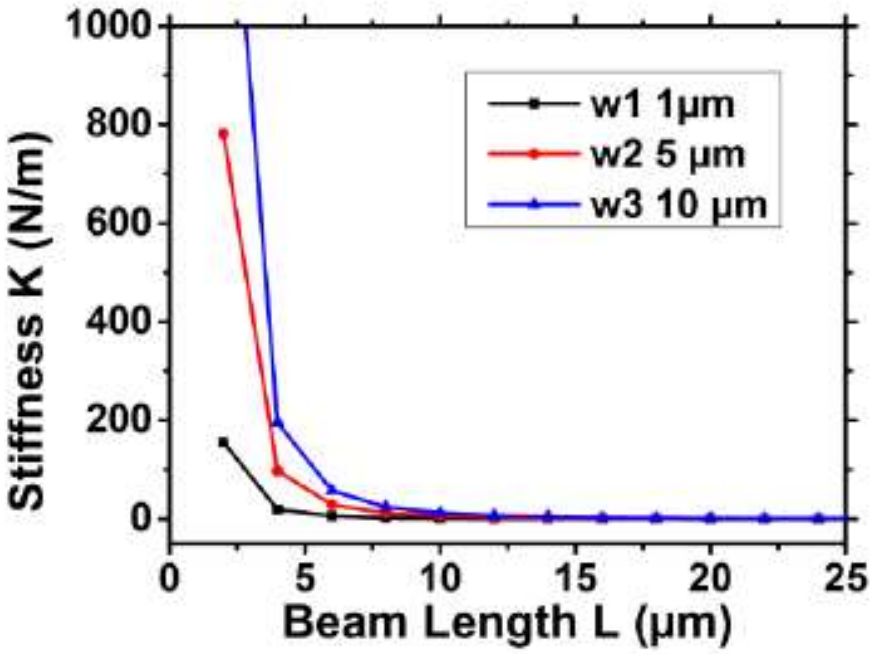


Figure 6.2: Variation of SU-8 beam's (E=2 GPa) Stiffness w.r.t Length for various thickness [38]

From figure 6.2, we could see that the stiffness of the beam reduces to a very low constant value for more than 6 μm length in all 3 cases of widths. It is also suggested that for Felxure-FET a Length to Width ratio of minimum 6 (L = 6 μm and W= 1 μm) will enhance the sensitivity. Due to certain situational and laboratory constraints a device with minimum dimensions would be fabricated and characterized in near future. But currently a suitably up-scaled device is fabricated due to limitations in photo-plot mask printing.

6.2 Lithography Mask Patterns and Dimensions

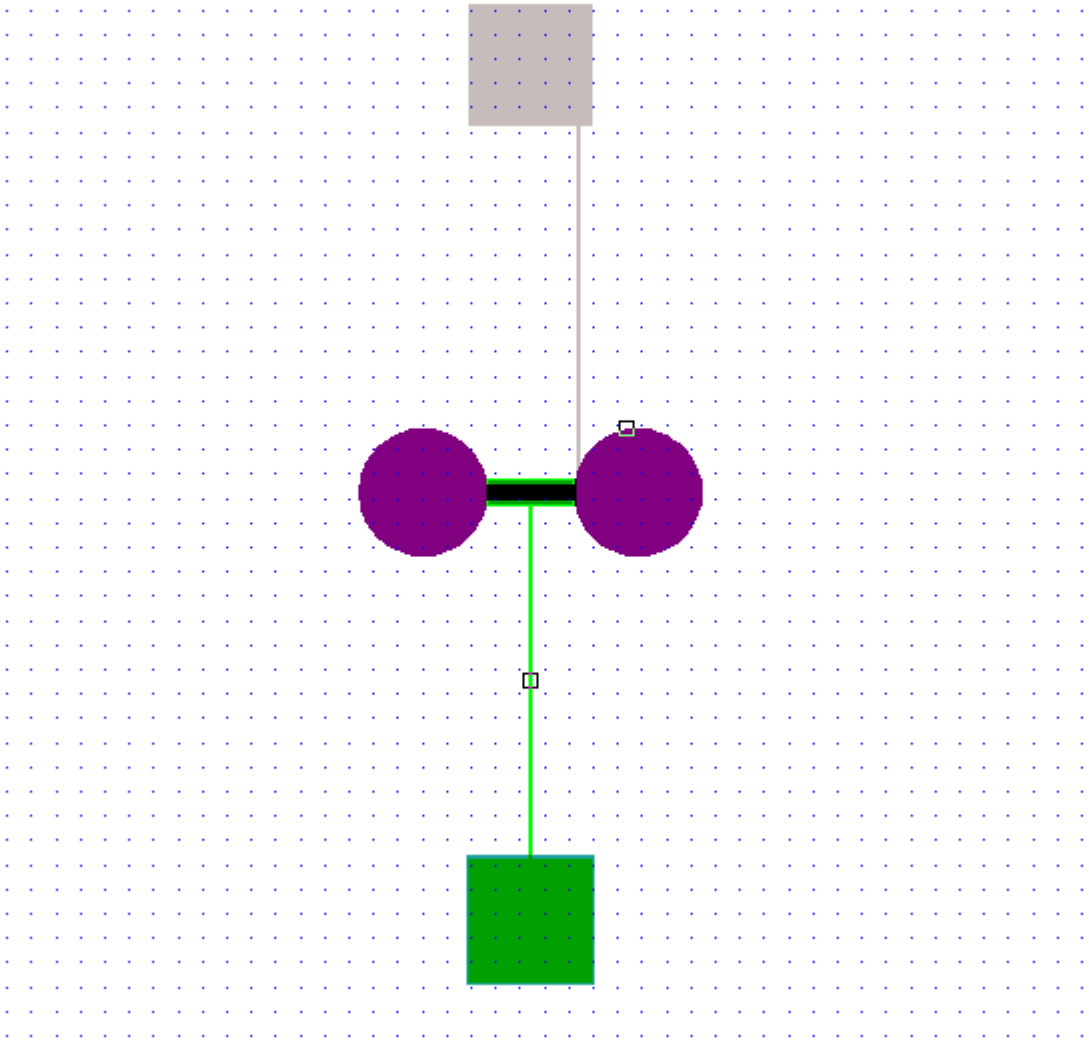


Figure 6.3: Composite view of the Capacitive Micro-beam structure

The Figure 6.3 is the compostive view of all layers in the Micro structure The two squares Green and Gray are in the Botton and Top Electrical contact layers respectively. The sides of the square are 1mm and can be used for Electrical probing. The Voilet coloured Circles and the beam forms the SU-8 layer which hangs when released. The big circles are for anchoring and also it can be used as reservoirs if properly secured by side walls in packaging. The diameter of these Circles are also 1mm.

Figure 6.4: Bottom Electrical Contact and Free-space Cavity

In Figure 6.4, the Light Green layer corresponds to Botton Electrical Contact and the dark area is meant for free-space cavity. The beam would be hanging over this cavity. The wire like protrusion which connects the bottom plate to the probing pad has a width of 20 μm. Also the cavity is inset to the bottom contact layer in all the four directions by 20 μm. This is necessary in order to get overlap between the layers even if there are alignment errors in mask prining or photolithography process. The mask is printed using a commercial High resolution photography printer on a tranparent plastic sheet, where there are inherent limitations in printing shapes and gaps less than 10 μm.

Dimensions of Bottom Electrical contact: Length = 720 μm and Width = 220 μm.

Dimensions of Free-space Cavity: Length = 700 μm and Width = 200 μm

Figure 6.5: Free-space Cavity and Micro-beam

In Figure 6.5, the Bluish layer is Free-space Cavity. The sacrifical material i.e. Copper is deposited and patterned to remain in this area which on etching becomes a cavity. The Violet layer is the inverted pattern of the Micro-beam. Hence the Micro-beam area is tranparent in the

mask inorder to cross-link the SU-8 there. So the beam is inset for 25 μm from both the length edges of the cavity to provide gap for Copper etchants and also to compensate alignment errors etc. The effective length of the hanging part of the beam is equal to the length of the Cavity itself and the width is 150 μm.

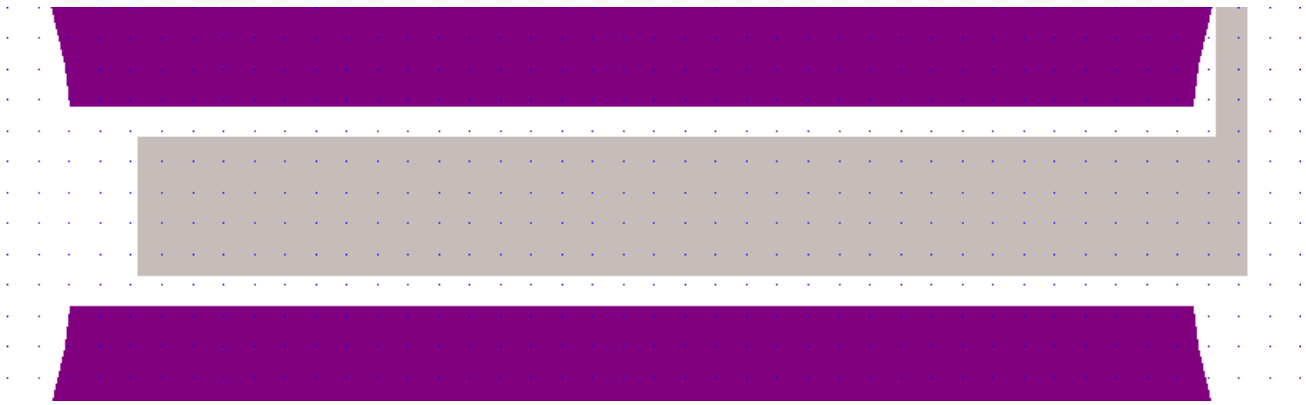


Figure 6.6: Micro-beam and Top Electrical Contact

In Figure 6.6, White area corresponds to the Micro-beam and the Gray pattern corresponds to the Top Electrical Contact. The rectangular contact is offset from the edge of the beam in the left side by 45 μm. This avoids anchoring of the contact in one of the ends, which might reduce the stiffness of the composite Micro-beam.

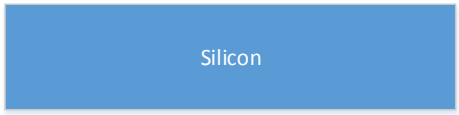
Dimensions of Top Electrical Contacts are: Length = 720 μm and Width = 90 μm

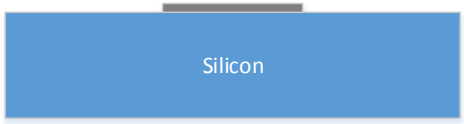
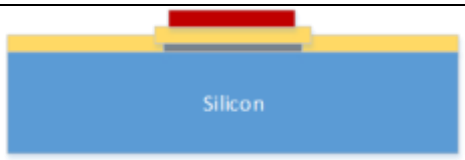
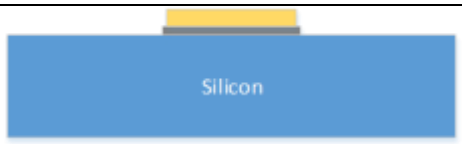
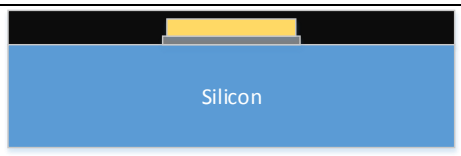
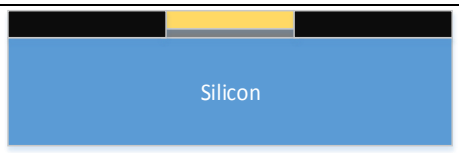
The wire of 20 μm width from the right most end connects to probing pads.

6.3 Fabrication Process

The following table illustrates the Surface Micromachining process for fabricating the SU-8 based Capacitive Micro-beam structure.

Table 6.2: A Comprehensive picturisation of Capacitive Micro-beam Fabrication Process sequence

Step No.	Cross Section View	Mask Pattern (Not to scale)	Microscopic Photograph	Process
1				Piranha Cleaning, HF dip and Dehydration bake at 200°C

2				100 nm Titanium Sputtering & UV Photolithography for **Bottom Electrical Contact**
3				Titanium etching
4				Copper Sputtering & UV lithography for **Sacrificial layer**
5				Cu etching
6(A) or 6(B)				SU-8 passivation & beam anchoring layer fabrication
				
7				100 nm Titanium Sputtering & UV lithography for **Top Electrical Contact**
8				Titanium etching
9				SU-8+Glycidol polymer beam fabrication (Should be the last deposition process)

10				Sacrificial Cu etching & releasing the Micro-beam in CPD

In this process Copper is used as Sacrificial material for creating void under the micro-beam. Ther are few other methods [38] where Uncrosslinked SU-8 and Lift-off-Resist (LOR) are used as sacrificial material [39]. Also by UV dosage control free standing SU-8 structure were formed [38-39]. But a Copper deposited using a calibrated Sputtering system would give us good control in nm accuracy over the void gap. Also Copper is CMOS compatible metal and can be etched easily with standard Microelectronic etchants. Shipley's Microposit S1813 Positive Photo Resist is used for patterning and etching metals. SU-8 is the main structural material. Pure SU-8 is used as passivation and anchoring layer.

Either Step 6(A) or 6(B) can be followed. Step 6(A) is intended to get a Top electrical plate which is sandwiched between 2 layers of SU-8, where the top one is Bio-functionalized. Step 6(B) avoids the sandwich by having only the top SU-8 layer. Depending on requirement and application either one can be chosen. In case of Flexure-FET Step 6(B) is desirable to reduce the pull-in voltage [1] of the Micro-beam. Step 9 is meant for fabricating the SU-8 beam (if Step 6(B)) or the top SU-8 layer (if Step 6(A)) which can be Bio-functionalized. In this work both pure SU-8 and SU-8+Glycidol is attempted in Step 9. In both scenarios a stable hanging Micro-beam is obtained after Critical Phase Drying (CPD) of the samples.

Microchem SU-8 2002 is used for fabrication. It is used in developing SU-8+Glycidol mix as well. Standard processing parameters are followed as per the datasheet of SU-8 2000. However, the Soft baking is done by gradually increasing the temperature from 65°C to 95°C gradually in a duration of 6 minutes at immediately taken out of hot plate after reaching 95°C. PEB is done at the same rate but sample is maintained at 95°C for about 10 minutes and gradually cooled down to 55°C in 30 minutes time duration. This PEB step is necessary to avoid cracking and shrinking of SU-8 film during cross-linking.

SU-8+Glycidol mix has different viscosity and hence different process parameters are to be followed. It is found that SU-8+Glycidol spin coats uniformly over Silicon or SiO_2 surface. But spin coating at higher speeds over a layer SU-8 results in a patchy layer. Hence it is found experimentally that a spin rate of 1000 rpm/sec with an acceleration of 300 rpm/sec^2 for 40 seconds

results in a smooth uniform film over pure SU-8 surface. Specific details for fabricating SU-8+Glycidol is given in Step 2 of 5.4.2 in Chapter 5.

The etchants used for patterning metals are as follows,

Titanium etchant: DI Water: Hydrogen Peroxide (30%) : HF (49%) = 20:1:1;
etch rate approx. 20 nm/sec

Copper etchant: DI Water: Hydrogen Peroxide (30%) : HCl (37%) = 10:1:2;
etch rate approx. 30 nm/sec

The above mentioned process flow uses all CMOS compabile materials and chemicals. Also all the steps are very conventional Microfabrication methods like UV Photolithography, etching and CPD. Hence, Surface Micromachining of the SU-8 structure can be done over the BEOL layers of a CMOS IC Chip.

6.4 Stiction and Critial Phase Drying (CPD) Process

One most common and critical problem in MEMS fabrication is Stiction. Stiction is the permanent deformation of free standing structures, which occurs during drying of the wafer after etching the sacrificial layers. As a final step usually samples are washed thoroughly in DeIonised Water (DI Water) and dryed using Compressed air or N_2 gas. The capillary force at the interface of DI water and the surfaces, pull the structures along the fluid flow which results in Stiction. There are other reasons like Cassimir force which is due to van der Waals attraction between two closely spaced layers (less than 1μm). But this force can be overcome if the material and structure has inherent strength.

To avoid stiction during fabrication, CPD is followed. After etching the sacrificial Copper, the beams are released in Tousimis Autosamdri 815B Supercritical dryer. The samples were transferred from etchant to DI water and finally to high purity Isopropanol environment in the dryer chamber. The sample is exposed to Isopropanol and Liquid CO_2 in CPD system which are necessary to create a Super critical phase environment.

6.5 Metrology and Observations of Fabricated Structure

An elementary and qualitative characterization of the fabricated structures were carried out. The thickess and depths are measure using a Laser based surface profiler. The hanging of the Micro-beams are confirmed by tapping it under a Optical Microscope using the tip of a Micro-probe.

The deflections of the beams were observed in the Microscope's Computer display and the same is recorded as a video.

Several Micro-beams were fabricated by using 1 μm and 200 nm thick Copper sacrificial layers. Also they were fabricated by following Step 6(A) and 6(B). It was found that in case of Step 6(B) based structures, the top Titanium contact is stick to the SU-8 beam due to good adhesion between them. The same was confirmed by checking for any shor-circuit between top and bottom contact using a Multimeter.

Here we will see some of the measurements of Cases,

(i) 200nm sacrificial Copper, Step 6(B) & SU-8 2005 and

(ii) 1 μm sacrifical Copper, Step 6(A) & SU-8 2002 +Glycidol

Case (i) 200nm sacrificial Copper & Step 6(B)

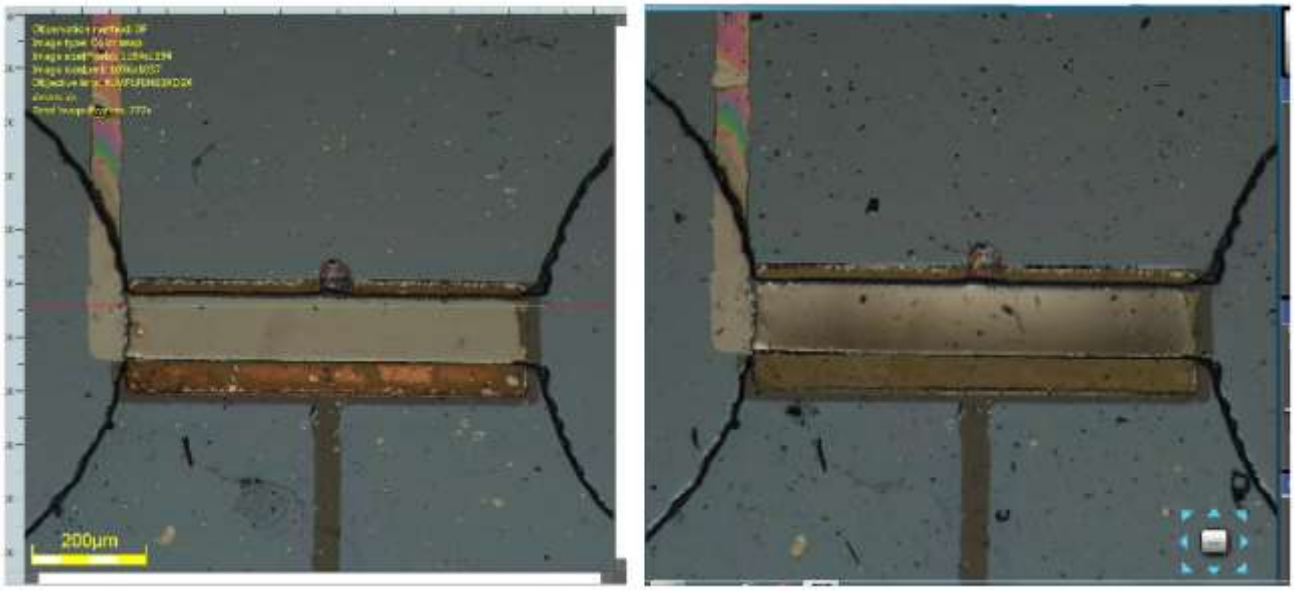


Figure 6.7: Fabricated Capactivie Micro-beam structure as per Case (i), Before & After Copper etching and CPD

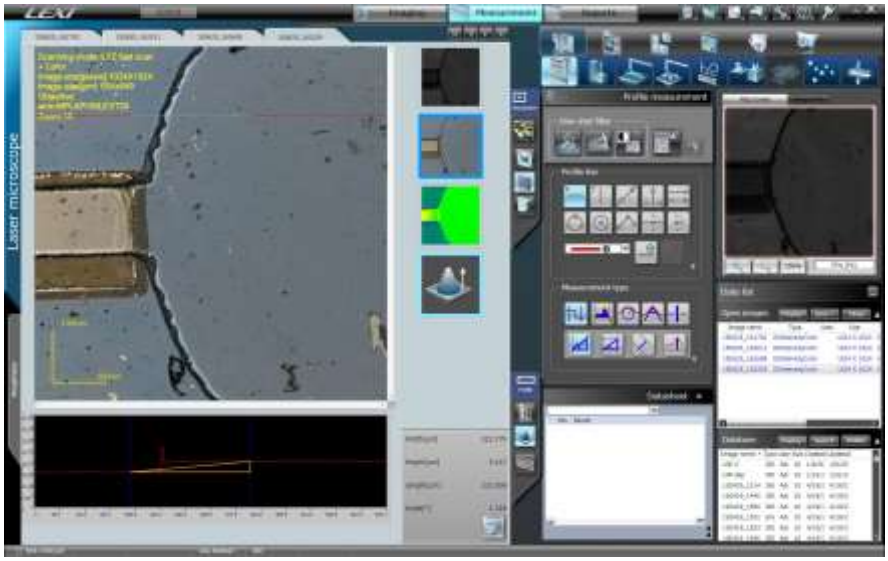


Figure 6.8: Thickness of SU-8 Beam layer = 5.147 µm (by measuring the anchoring part area)

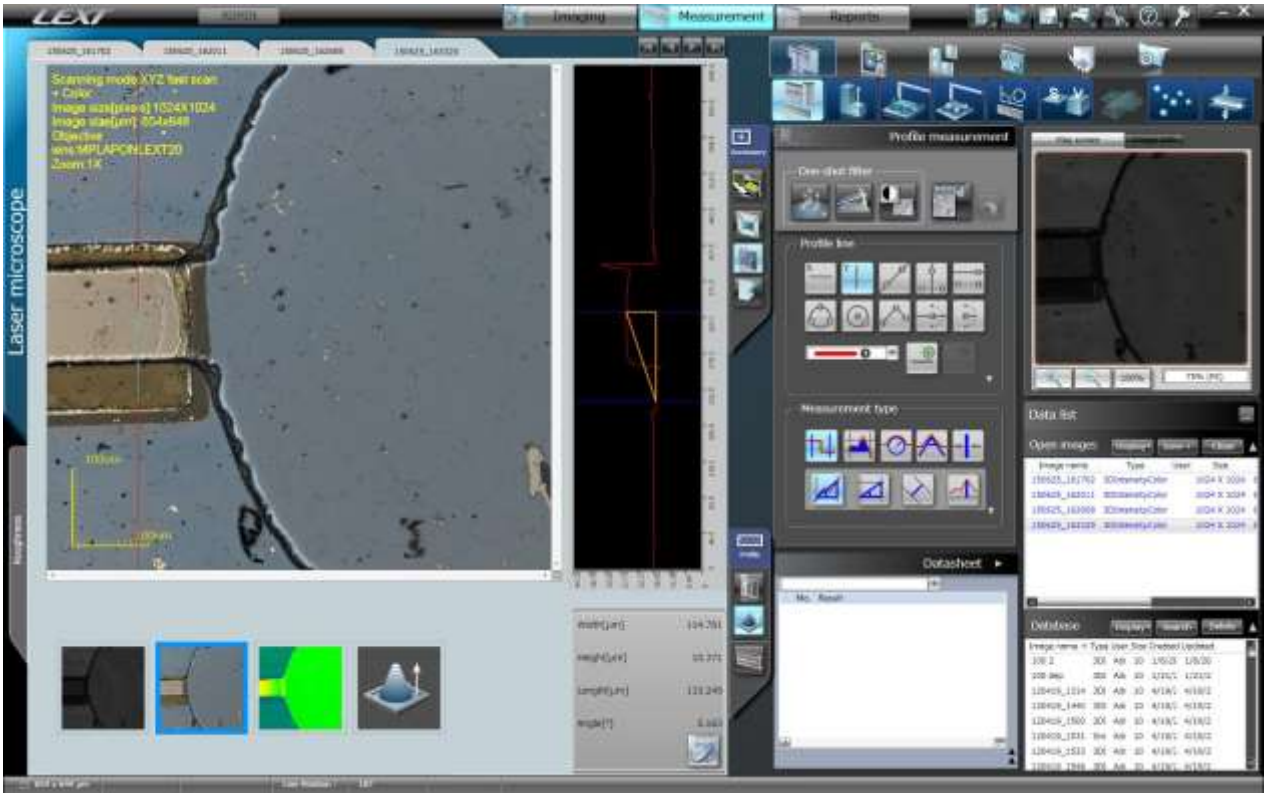


Figure 6.9: Distance between the top of Beam to the bottom Electrical contact = 10.371 µm at right most end.

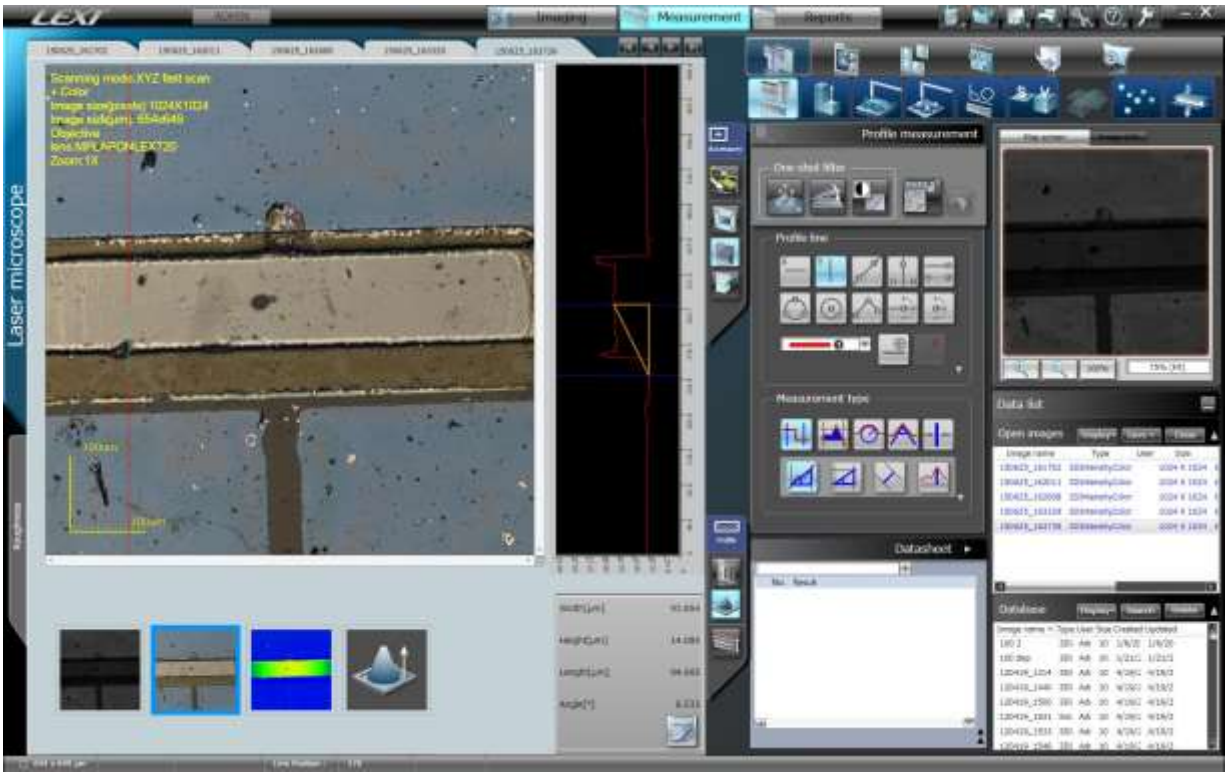


Figure 6.10: Distance between the top of Beam to the bottom Electrical contact = 14.085 µm at middle of the beam.

Case (ii) 1 µm sacrifical Copper & Step 6(A)

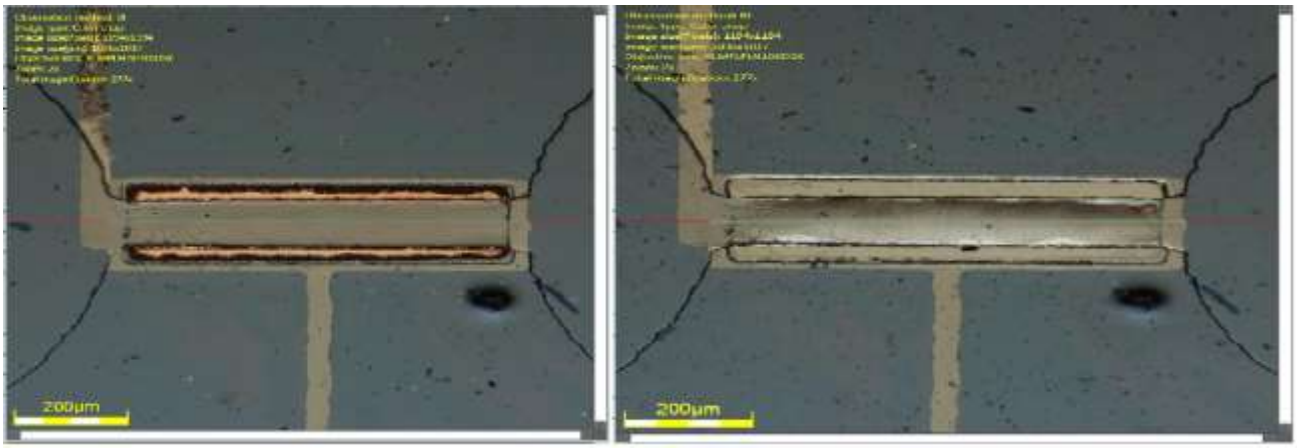


Figure 6.11: Fabricated Capactivie Micro-beam structure as per Case (ii), Before & After Copper etching and CPD

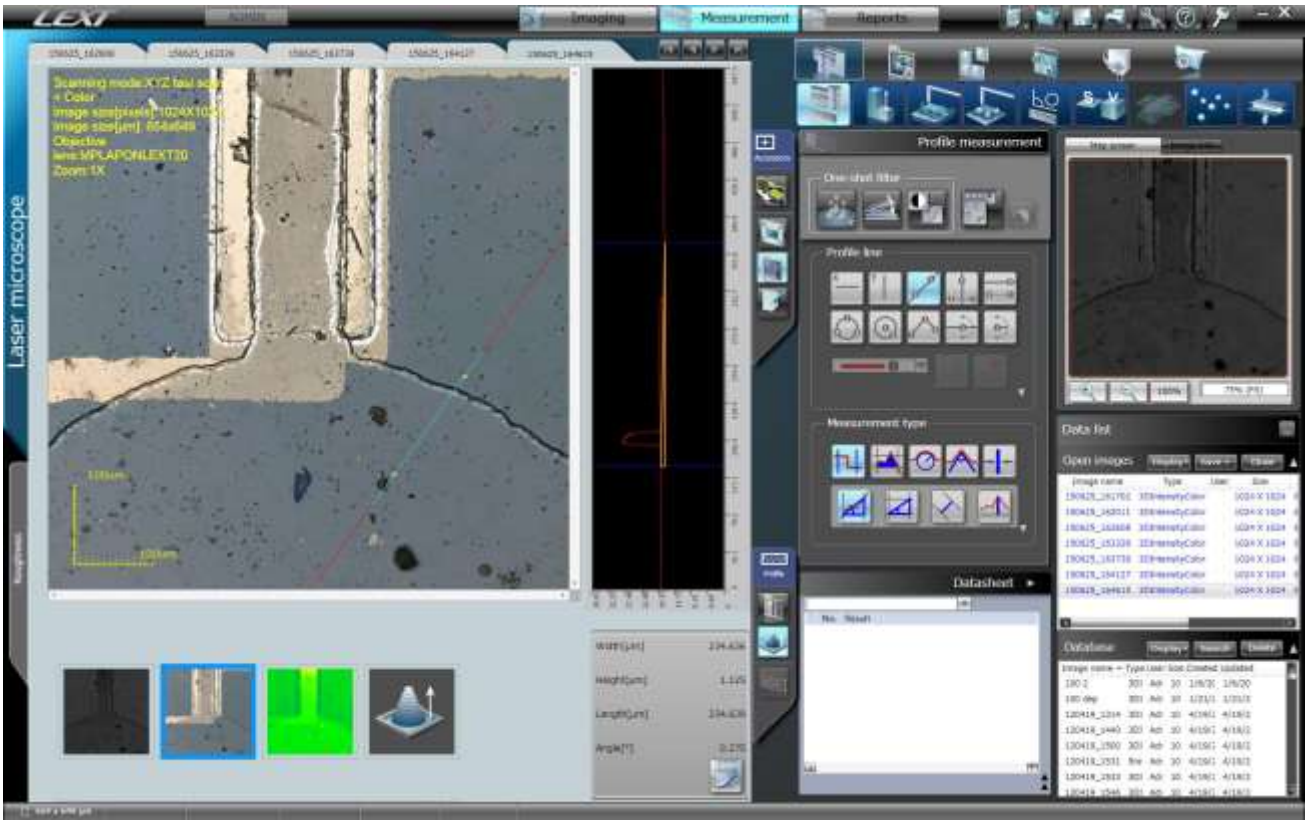


Figure 6.12: Thickness of SU-8+Glycidol top layer of the Micro-beam = 1.125 µm

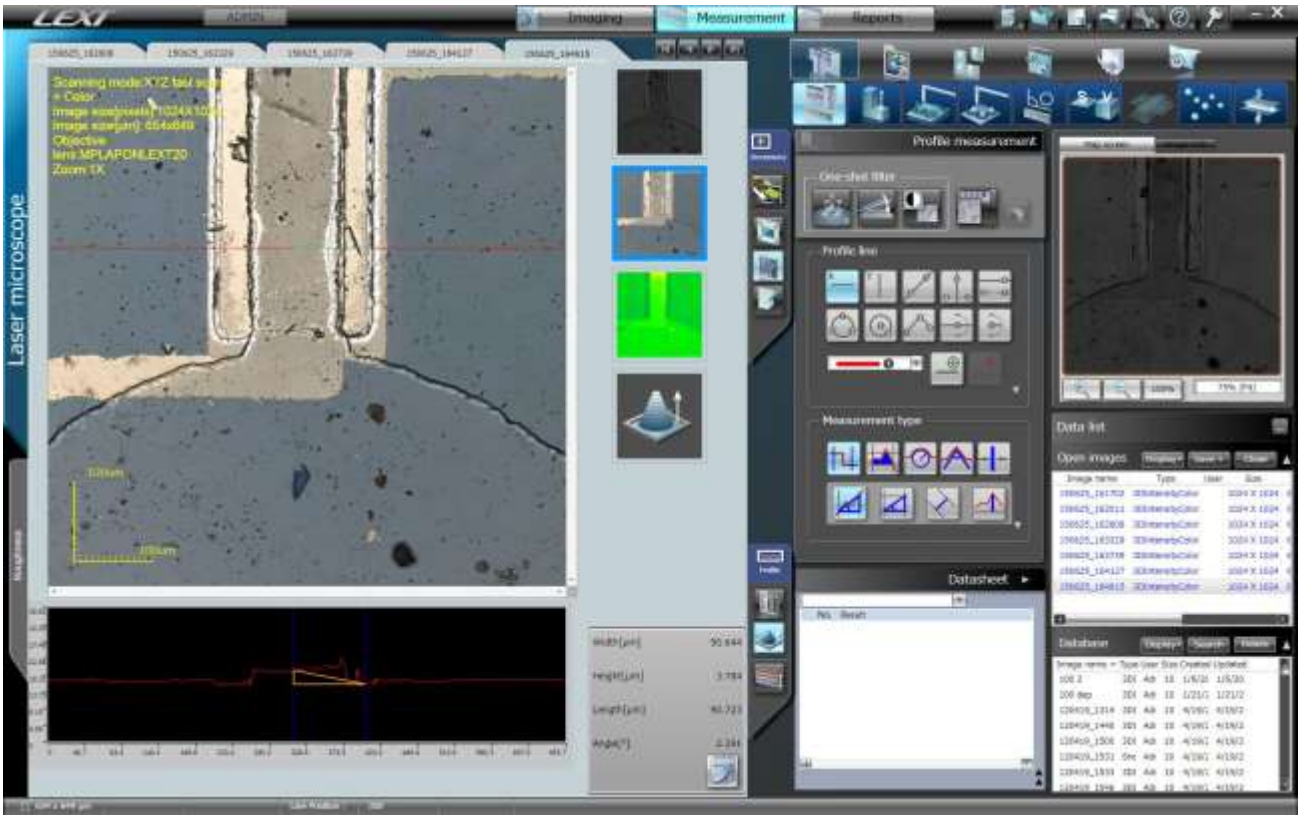


Figure 6.13: Distance betweem top of the beam to the Bottom Electrical contact = 3.784 µm

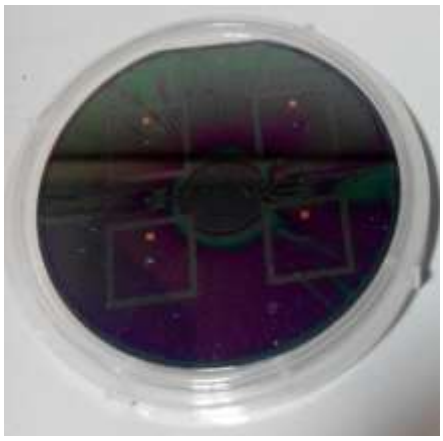


Figure 6.14: 2 inch Silicon wafer with 4 numbers of devices

We could observe that the beams are curved like an arch with maximum air-gap at the middle of it. For example in Case (i), the thickness of the SU-8 beam layer is 5.147 µm and distance between the top surface of the beam to bottom electrical contact the middle of the beam is 14.085 µm. So the air-gap at the middle region of the beam is approximately 8.982 µm. Even though the sacrifical layer is 200 nm, the excess air gap is due to the curvature of the beam. This curvature may be attributed to the stress developed due to the differences between Coefficients of Thermal Expansion (CTE) of SU-8 and Titanium. Similar trait is found in all the fabricated beams.

Case (ii) uses SU-8 2002+Glycidol polymer for top layer of the micro-beam. They are found to be mechanically stable after CPD process. For verifying the Hydrophilic nature of this SU-8 2002+Glycidol surface during the fabrication flow, separate films of the same polymer with large patterns underwent the same processes concurrently. The DI Water contact angle over these surfaces were checked before Copper etchant treatment, after Copper etchant treatment and after CPD process. VCA Optima Contact angle Goniometer was used for this check.

Figure 6.15: Pure SU-8 2002 UV Cross-linked film

Figure 6.16: SU-8 2002 + Glycidol film

Figure 6.17: SU-8 2002 + Glycidol film after Cu etchant treatment

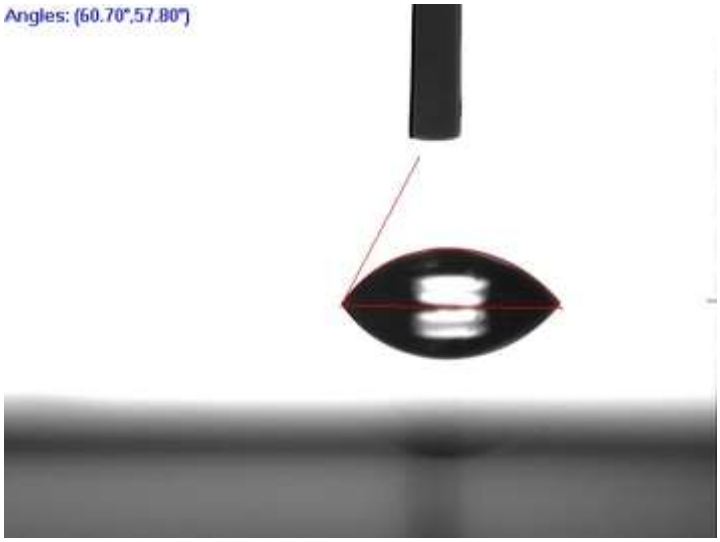


Figure 6.18: SU-8 2002 + Glycidol film after CPD process

From the above results we could conclude that SU-8+Glycidol films' contact angle further reduced which is a positive sign of presence of Surface functional groups. Hence, this layer is fit for Bio-functionalization after CPD process also.

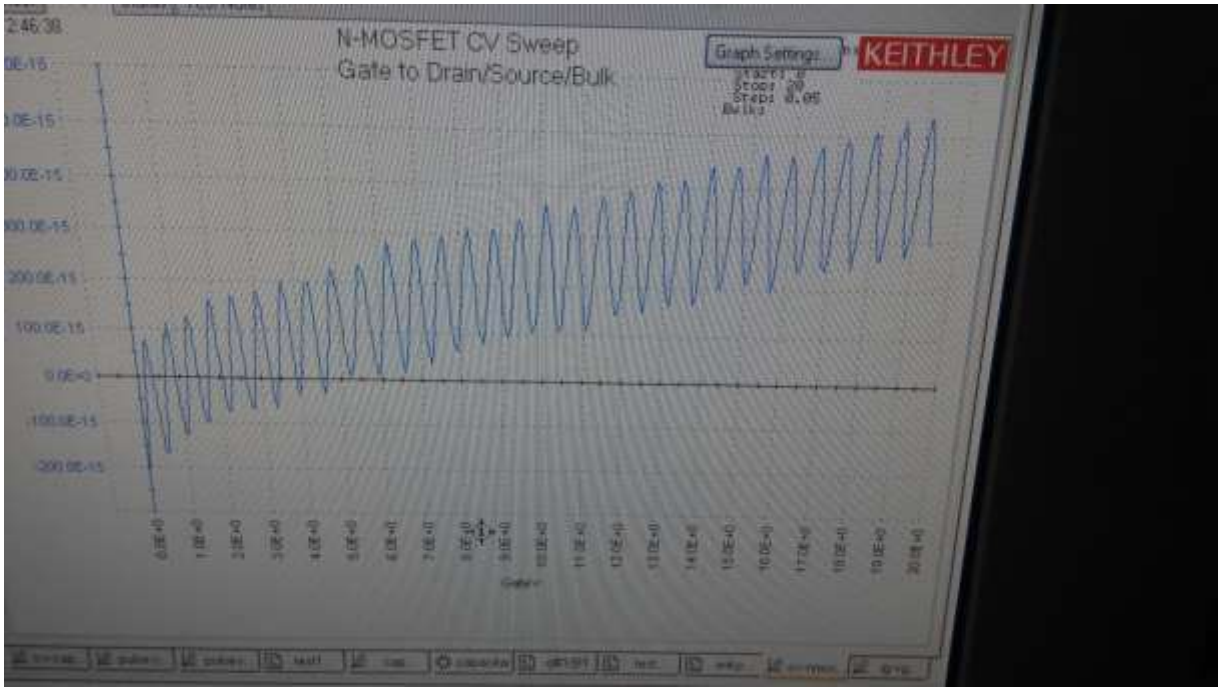


Figure 6.19: I-V Characteristics of the structure for a ramping step voltage waveform from 0V to 20V

A simple and qualitative Electrical check of the structure mentioned in Case (i) is done using Keithly 4200 Semiconductor Characterization System (SCS). The Voltage waveform is a ramping up step from 0 V to 20 V. We could see the corresponding Current waveform in Figure (), where the current flows during the Voltage transients. Further elaborate electrical characterization is needed using LCR meters and SCS for calibrating this sensor.

6.6 Equipments used in Fabrication and Characterization

The following are the important equipments used in fabricating this device. They are part of Nano-X Laboratory of Indian Institute of Technology, Hyderabad.

Figure 6.20: Suss MicroTech MA6/BA6 Mask Aligner and UV Exposer for Lithography

Figure 6.21: AJA International's DC & RF Sputtering tool for Thin-film depositon

Figure 6.22: Tousimis Autosamdri 815B Super Criticla Dryer for CPD process

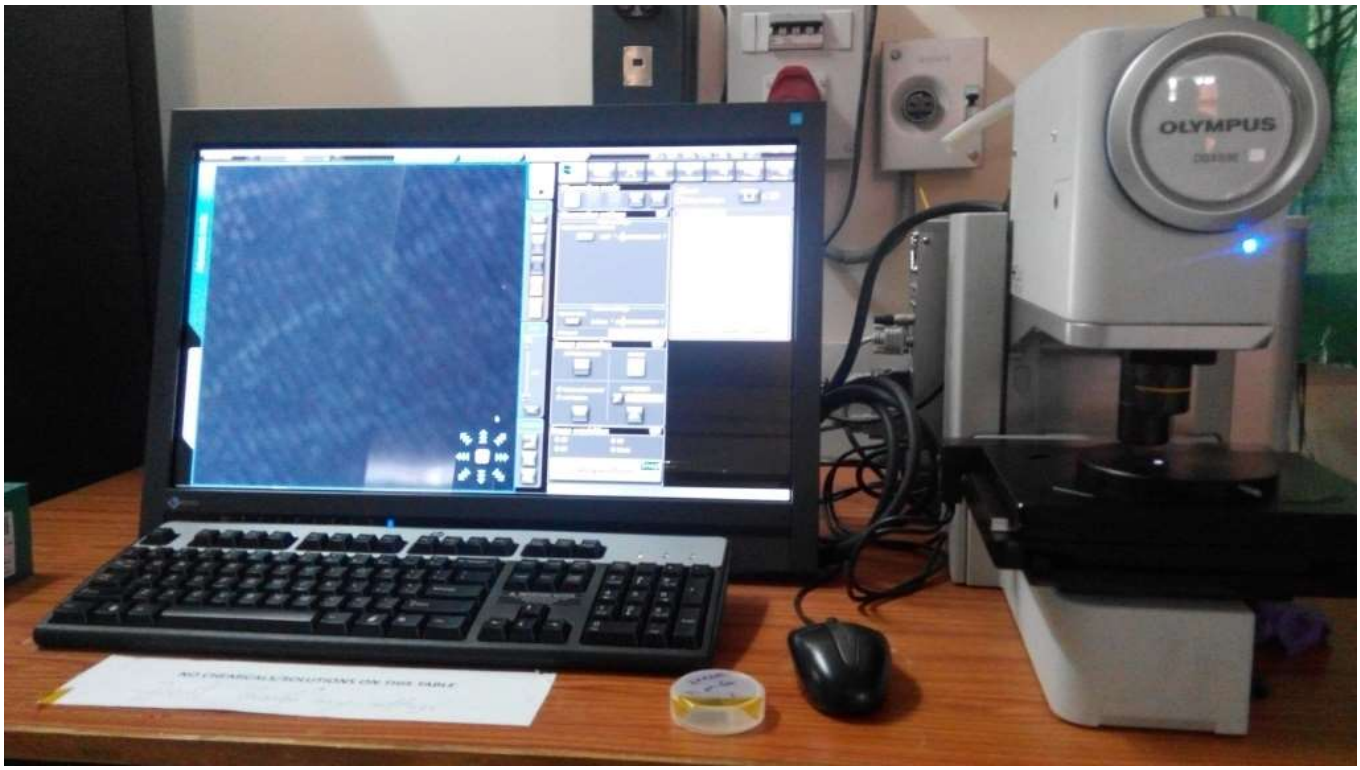


Figure 6.23: Olympus DSX500 Optical Microscope

Figure 6.24: Cascade Microtech Probe Station

Figure 6.25: Keithly 4200 Semiconductor Characterization System connected to Probe Station

Chapter 7

Conclusion and Future Scope of Work

7.1 Conclusion

In this work we have taken a good leap towards realizing an Ultra-sensitive Flexure-FET Biosensor. The Selective Bio-functionalization process for SU-8 has tremendous potential to be adopted by industry due to its simplicity and Wafer scale functionalization ability. A publication on this work was accepted and presented in a reputed International Conference. The details are as follows,

Srinivasan B, Durgesh Chaurasiya, Siva Rama Krishna Vanjari and Shiv Govind Singh, **"A Simple Process for Selective Bio-functionalization of SU-8 surface for Lab-on-a-Chip applications",** TechConnect World Innovation Conference & Expo – Nanotech 2015, June 14-17, 2015. Washington, DC., USA.

Many works in SU-8 based surface stress Biosensors, uses a reverse structure (upside down) fabrication, release and flip-chip bonding over another surface [12, 14-15]. This is not convenient during batch processing in foundries. Whereas the proposed method is the direct surface micromachining of the desired structure. SU-8+Glycidol polymer based Micro-beam is also fabricated without degrading its surface property. This proves the potential of the proposed technique for usage in Bio-Chip technologies. Also, SU-8 based MEMS fabrication is cheaper and involves very low temperatures. Capacitive coupling is also the most preferred in MEMS to CMOS coupling due to its simplicity in materials and fabrication. Enough reasons are given regarding the CMOS compatibility of both Bio-functionalization and Micro-beam fabrication process. This device can be used as Capacitive sensor or be integrated to form a Flexure-FET Biosensor. Hence, the works presented in this dissemination has high potential for practical usage.

7.2 Future Scope of Work

There are tremendous work to be done in realizing a properly calibrated and usable Surface-stress based Biosensor or Flexure-FET Biosensor. The future works are categorized as follows.

Bio-functionalization

- Verifying Selective Bio-functionalization using Fluorescent tagged Bio-molecules.
- SU-8+Glycidol layer Bio-functionalization using other Bio-molecules like Antigens and ssDNA.
- Developing a CPD compatible Bio-functionalization protocol or a method to avoid CPD process.
- APTES can be deposited in vapor phase deposition in a Vacuum Oven for obtaining good Monolayer.

MEMS Design, Optimization and Fabrication

- Electrical conductivity enhancement of SU-8 without compromising its mechanical properties.
- Thorough Device optimization using 3D MEMS process simulators like Coventorware.
- Numerical & experimental optimization of the thickness of Titanium & SU-8 to reduce the stress and curvature.

Flexure-FET Fabrication

- Smaller dimension device fabrication using Chromium mask.
- MOSFET fabrication and integration with MEMS structure.

Characterization

- Characterization and calibration of Capacitive Biosensor & Flexure-FET Biosensor using Bio-analytes.

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