# Louisiana State University LSU Digital Commons

### LSU Master's Theses

Graduate School

2011

# Back-end processing of scanning mirrors with scratch drive actuators

Pradeep Pai Louisiana State University and Agricultural and Mechanical College

Follow this and additional works at: https://digitalcommons.lsu.edu/gradschool\_theses Part of the <u>Electrical and Computer Engineering Commons</u>

#### **Recommended** Citation

Pai, Pradeep, "Back-end processing of scanning mirrors with scratch drive actuators" (2011). *LSU Master's Theses*. 1652. https://digitalcommons.lsu.edu/gradschool\_theses/1652

This Thesis is brought to you for free and open access by the Graduate School at LSU Digital Commons. It has been accepted for inclusion in LSU Master's Theses by an authorized graduate school editor of LSU Digital Commons. For more information, please contact gradetd@lsu.edu.

# BACK-END PROCESSING OF SCANNING MIRRORS WITH SCRATCH DRIVE ACTUATORS

A Thesis Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Electrical Engineering in The Department of Electrical and Computer Engineering

By Pradeep Pai B.E, Visveswaraiah Technological University, Belgaum, India, 2006 May 2011 Dedicated to my parents and sister

#### ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to Dr. Dooyoung Hah, my research advisor for his unending support and guidance over the course of my degree program. I am inspired by his ways of tackling a problem and his ability to manage various tasks in parallel. I learned a lot of new things from him and re-learned in a simpler way, things that I already knew. I am also thankful to him for being patient, understanding and caring.

It was an honor to have Dr. Pratul Ajmera and Dr. Martin Feldman in my final exam committee. I would like to thank them for their time and valuable suggestions. I would also like to thank Mr. Golden Hwaung for training me to handle equipment in the lab, and teaching me to solve problems practically. I acknowledge the support from National Institute of Health and the Research and Development Program of the Louisiana Board of Regents that partially funded this research work through research grants 1R21CA139093 and LEQSF(2006-09)-RD-A-05, respectively.

I sincerely thank Mr. Anirban Sarkar, Ms. Dan Zhang, Mr. Naga Korivi, Mr. Karthik Balasubramanian, Ms. Pallavi Rao and Mr. John Bordelon for their technical and non-technical suggestions that helped me to complete this thesis work.

Finally, I would like to thank my parents and my sister for their unconditional love and support, without which I would not have pursued my Masters' degree.

# TABLE OF CONTENTS

List of Tables	V
List of Figures	vi
Abstract	viii
Chapter 1. Introduction	1
1.1 Scratch Drive Actuator (SDA)	2
1.2 Scanning Mirrors Driven by SDA	4
1.3 Device Fabrication – Surface-Micromachining by MUMPs® Process	6
1.4 Device Fabrication – Post-MUMPs® Processing	6
1.5 Challenges in the Work	11
1.6 Literature Review	13
Chapter 2. Front Side Protection During Silicon Substrate Etching	17
2.1 Materials	18
2.2 Method	18
2.3 Results and Discussion	24
Chapter 3. Formation of Photoresist Links	
3.1 Materials.	
3.2 Method	
3.3 Results and Discussion	35
Chapter 4. Conclusion	44
Bibliography	45
Vita	52

# LIST OF TABLES

Table 1. Summary of KOH etching results	25
Table 2. Description of the characteristic Raman peaks for PDMS	30

# LIST OF FIGURES

Figure 1. Schematic of a parallel-plate type micro-mirror	1
Figure 2. Axial cross-section of the scanning probe	2
Figure 3. Illustration of SDA actuation	3
Figure 4. Schematic of scanning mirror driven by SDAs	4
Figure 5. Layout of the MUMPs® chip	5
Figure 6. Cross-section of MUMPs® chip showing top and bottom layers	7
Figure 7. Radial cross-section of scanning probe	8
Figure 8. Overview of post-MUMPs® processing steps	9
Figure 9. Cross-section of MUMPs® top layers showing variation in topography	13
Figure 10. Illustration of PDMS protection method used by Khoo et al	20
Figure 11. Illustration of PDMS seal ring method used by Brugger et al	20
Figure 12. Illustration of silicone rubber protection method used by Obreja	
Figure 13. Isometric view of the setup for making the PDMS seal	22
Figure 14. Isometric view of the setup after PDMS is cured	23
Figure 15. Cross-section view of the etch protection setup	24
Figure 16. Pictures of etched samples	
Figure 17. Pictures of failed and successful samples	29
Figure 18. Raman plot of etched and un-etched samples	
Figure 19. Isometric view of the leveling setup after assembly	
Figure 20. Cross-section view of the leveling process	
Figure 21. Photoresist patterns after exposure	37
Figure 22. Photoresist patterns after hard bake at 180°C	

Figure 23. Photoresist patterns after hard bake at 200°C	.39
Figure 24. Photoresist patterns after hard bake at 220°C	.40
Figure 25. Comparison of the photoresist dimension in the layout against the actual re dimension	esist 41
Figure 26. Top view of the disengaged SDA chain on one of the devices	.42
Figure 27. Plot of the percentage shrink in the photoresist length as a function of the origin photoresist dimensions	inal 43

## ABSTRACT

The use of micro-electro-mechanical system (MEMS) processes in fabrication of micrelectro-mechanical devices for optical applications has been widespread. The invention of digital light processing (DLP) technology by Texas Instruments Inc. popularized the use of micromirrors. One of the applications of the micro-mirrors is in the form of scanners for biomedical imaging. The small size of mirrors and actuators makes them a good candidate for *in vivo* measurements/imaging. Various techniques in bulk- and surface-micromachining, and various actuators have been used to fabricate scanning mirrors. The scanning mirrors used in this work make use of scratch drive actuators (SDA) for their scanning motion. Both surface- and bulkmicromachining technologies are used to fabricate the devices. Surface-micromachining (Multi User MEMS Processes or MUMPs®) is used to fabricate scanning mirrors, and bulkmicromachining is used to separate dies, to create sloped sidewalls for efficient packaging and to make grooves for optical fibers.

This work describes the techniques used in the post-MUMPs processing of the devices. The main features of the post-MUMPs processing are substrate etching and formation of insulating links. The presence of devices on the front side necessitates the use of a protection method in the substrate etching step. Polymers have drawn attention in recent years as protective materials due to their chemical stability as well as the ease of use. This thesis work utilizes poly dimethylsiloxane (PDMS) as a protection material, and examines the effect of PDMS process conditions on the quality of the protection. Protection for at least 10 hours was achieved in this work.

The scanning mirror devices of this thesis work require non-conductive links between the actuators and the mirror. Thick photoresists such as AZ 4620, AZ 9260, SU-8 etc, are a good

choice due to the ease in their patterning as well as their excellent insulating properties. This thesis documents the processes followed to achieve optimal link structures using thick photoresist (AZ 9260). The resist was optimally hard baked to make it chemically inert to common organic solvents.

## CHAPTER 1

# **INTRODUCTION**

Micro-mirrors are one of the most common devices in MEMS and find widespread applications as reflectors, scanners, projection devices, etc. The earliest micro-mirrors were realized out of silicon substrates. Due to progress in thin film technology, other materials like metal coated polysilicon and aluminum are also used. There are different operation mechanisms employed in actuation of mirrors, with the electrostatic being the most common one. Among the various types of electrostatic actuation mechanisms, the parallel-plate type  $^{[1 - 2]}$  and the comb-drive type  $^{[3]}$  are widely used; and the parallel-plate type has found commercial use. Figure 1 illustrates the operation mechanism of a simple parallel plate type micro-mirror. It can be understood from the figure that, at rest, the mirror plane is parallel to the substrate, thus requiring a substantial angle for the incident light.



Figure 1. Cross-sectional view of a parallel-plate type micro-mirror in its actuated position when the bottom right electrode is active. The bottom electrodes are embedded in a dielectric material to avoid shorting with the top electrode. Figure not drawn to scale.

The micro-mirror used in this work operates from a near vertical position. The vertical position is necessary since the mirror is used in a forward looking endoscope, <sup>[4]</sup> and the optical fiber is parallel to the substrate of the device. Figure 2 shows the setup. Arrays of scratch drive actuators are used in this work both for initial mirror positioning and for scanning operation.



Figure 2. 3-D view of the scanning probe after assembly. A 2 mm  $\times$  2 mm die with scanning mirror is fitted on top of an optical fiber with the groove restricting the lateral motion of the fiber. The optical fiber is glued to the lens. Bottom half of the lens is coated with a metal layer to be used as a mirror. Figure not drawn to scale.

#### **1.1 Scratch Drive Actuator (SDA)**

Electrostatic actuation has also been used to obtain linear in-plane motion and a large driving force. <sup>[5]</sup> One of these devices is scratch drive actuators (SDA) which utilizes the

principle of inch worm motion. The step-wise motion of these devices can be precisely controlled. Peyman et al <sup>[6]</sup> have provided a detailed account on the characteristics of SDAs. Figure 3 schematically illustrates the operation of SDAs.



Figure 3. Schematic diagrams illustrating the operation of SDAs. (a) shows the resting position of the SDA and (b) shows the actuated position. Thick arrows indicate the direction of motion. The bushing directs the movement of the SDA. Figure not drawn to scale.

The operation of the SDA is explained as follows. Following the release of the structures, the SDAs remain in their resting position as shown in figure 3 (a). Application of a voltage greater than a threshold voltage between the SDA and the bottom electrode causes the body of the SDA to be pulled down to the surface of the dielectric layer. Typically, bipolar pulses with amplitude of tens to hundreds of volts are used to drive SDAs. This produces a force in the SDA that pushes the bushing forward as illustrated in figure 3 (b). Removing the excitation voltage brings back the SDA body to its resting state keeping the bushing anchored at the new position. Thus, the SDA is now displaced from its original position. One such a step of motion is called a stroke. Each stroke is typically tens of nanometers in length. Two strokes are obtained by one

period of the pulse. The force produced by these strokes is large enough to lift other mechanical structures as demonstrated by Akiyama et al. <sup>[7]</sup> This force can be used to produce rotational motion if a structure is coupled to the SDAs through hinges. The magnitude of the driving force can further be increased by connecting multiple SDAs in tandem. The scanning mirrors referred to in this thesis are operated by this principle.

#### **1.2 Scanning Mirror Driven by SDA**

Figure 4 roughly depicts the operation of the scanning mirror. The figure has been drawn only for the purpose of illustration and the actual device design varies from this. It is to be noted that the motion of SDA is unidirectional, and hence two sets of SDAs facing opposite directions are needed in this work where bidirectional actuation is needed.



Figure 4. Scanning mirror operated by SDAs. Thick arrows indicate the directions of motion of the mirror and SDAs. The mirror can rotate from  $0^{\circ}$  at its resting position to  $90^{\circ}$  or beyond when actuated. Two sets of SDAs are used for forward and backward motion, respectively.

The SDAs, the connecting shafts and the mirror are made of polysilicon. Only one set of SDAs are actuated at a time as can be expected. The shaft controlling the mirror needs to be electrically isolated from the shafts connecting the SDAs to avoid unnecessary electrostatic actuation of the mirror. Also the shafts connecting the forward SDAs need to be isolated from the shafts connecting the backward SDAs. The precise and step-wise motion of the SDA provides good control over the deflection angle of the mirror. The devices are expected to have long life due to the excellent mechanical properties of polysilicon (Young's modulus ~ 160 GPa, Poisson's ratio ~ 0.22).

Figure 5 shows the layout of the actual chip and a device designed by Dr. Dooyoung Hah.



Figure 5. Layout of the actual chip with a close up of one of the devices. The chip measures 1 cm  $\times$  1 cm and each device occupies an area approximately 2 mm  $\times$  2 mm.

#### 1.3 Device Fabrication – Surface Micromachining by MUMPs® Process

Multi User MEMS Processes (MUMPs®) by MEMSCAP Inc. <sup>[8]</sup> is one of several commercial foundry services for MEMS. The MUMPs® process uses a standardized set of processes for surface-micromachining and utilizes various layers of thin films like phosphosilicate glass (PSG), polysilicon, polysilicon hard mask made of PSG (poly HM), silicon nitride, etc. The fabricated chips are sent to the customer for a final release step or post-processing. MEMSCAP Inc. offers three different flavors of MUMPs® fabrication namely SOIMUMPs<sup>TM</sup>, PolyMUMPs<sup>TM [9]</sup> and MetalMUMPs<sup>TM</sup>. It also offers customers the flexibility of altering the standard process flow or customizing thickness of individual layers at an additional cost.

The devices on the chip are made by patterning, etching and deposition of multiple layers of polysilicon with layers of PSG in between that serve as sacrificial layers. The topmost layer is 500 nm gold, which is used to improve the reflectivity of the mirrors, and as the bonding pads. The final chip measures 1 cm  $\times$  1 cm and contains nine SDA-driven scanning mirrors along with other test devices. Individual devices occupy an area of 2 mm  $\times$  2 mm approximately. Figure 6 shows the cross-section of the MUMPs® chip after fabrication at MEMSCAP Inc.

#### **1.4 Device Fabrication - Post-MUMPs Processing**

As shown in figure 6, the silicon substrate in the MUMPs® chip has multiple layers of polysilicon and PSG on both the front side and backside. The presence of multiple layers of polysilicon and PSG on the backside adds additional processing steps to the backend processing.

There are two major processes in the post-MUMPs® fabrication which include forming of mechanical links between SDAs and a mirror, and etching of the substrate to provide grooves



Figure 6. Cross-section of the MUMPs® chip after surface-micro-machining at MEMSCAP Inc. Dimples are provided to reduce the effect of structure collapsing due to liquid viscosity following the release process. PSG and poly HM are acronyms for phosphosilicate glass and polysilicon hard mask.

for optical fibers. Grooves are also necessary to form sloped edges on the dies for snug fitting inside the stainless steel tube as shown in figure 7.

Figure 8 provides a complete overview of the steps involved in post-MUMPs® processing. Tasks involved in each step are explained as follows. The multiple layers of polysilicon and PSG on the backside are stripped by successive dry and wet etching steps. A reactive ion etching (RIE) equipment (70 series from Plasma-Therm Inc.) is used to etch polysilicon (and poly HM). The etchant used is a mixture of tetramethyl fluoride (CF<sub>4</sub>, 100 sccm) and oxygen ( $O_2$ , 42 sccm) gases. Plasma was generated using RF power source (120 W), at a pressure of 250 milli-Torr. It was also found that PSG gets etched by CF<sub>4</sub> and O<sub>2</sub> plasma although at a slower rate. The two layers of polysilicon (Poly 2 and Poly 1), two layers of poly

HM and one layer of PSG (Oxide 2) on the backside are etched in  $CF_4$  and  $O_2$  plasma for 1.5 hours. The second layer of PSG (Oxide 1) is etched by immersing the chip in 49% hydrofluoric acid (HF) for 1 minute.



Figure 7. Radial cross-section of the scanning probe showing the need for sloped sidewalls on the die for fitting inside the stainless steel tube.

The front side of the chip is covered by photoresist to prevent HF from attacking the PSG on the front side. A further 10 minutes of plasma etching clears the last layer of polysilicon (Poly 0), exposing the silicon nitride layer underneath (figure 8 (b)).

The chip is then subject to a lithography process to define the etching windows for the grooves (figure 8 (c)). Since the alignment marks are on the front side, a double-side alignment is required to align the groove patterns on the backside. Following the patterning, the chip is again put in the RIE chamber to etch the silicon nitride layer in  $CF_4$  and  $O_2$  plasma (1.5 hours with the same process parameters mentioned earlier) to expose the etch windows (figure 8 (d)).



Photoresist works as an excellent mask to selectively etch silicon nitride. Following this step, the chip is immersed in acetone to strip the photoresist.

Figure 8. Overview of the steps involved in the post-MUMPs® processing. The thick arrows indicate the direction of etching. (a) shows the cross-section of the chip when it is received from MEMSCAP Inc. (b) shows the chip after stripping the multiple layers of polysilicon and phosphosilicate glass (PSG) on the back side. (c) shows the chip after a photoresist layer is coated on the backside for patterning the silicon nitride. (d) shows the chip after silicon nitride is patterned and the photoresist is stripped. (e) shows the formation of photoresist links on the front side of the chip. (f) shows the chip cross section after forming the grooves on the backside. (g) shows the chip after the structures are released and the chip is dried. Figures not to scale.

As mentioned earlier, the shaft controlling micro-mirror needs to be electrically isolated from the SDA shaft and yet coupled mechanically. An insulating material needs to be deposited and patterned to serve this purpose. Photoresist AZ 9260 was chosen for this purpose in this work. The photoresist is spin coated and patterned to form the mechanical links between the shafts (figure 8 (e)). Following this step, the backside of the chip is etched using silicon nitride as a mask to form grooves (figure 8 (f)).

The chip is then diced to separate the individual scanning mirrors that will go into the final assembly process. The dicing and assembly process will be managed by Ms Anandi Dutta and Mr Ragavendra Murthi. The assembly involves fixing an optical fiber underneath a die. A groove on the backside of the die secures the optical fiber in place and restricts its lateral motion. The scanning mirror needs to be released in 49% hydrofluoric acid (HF) after fitting the optical fiber (figure 8 (g)). A lens is attached to the fiber following the release, since HF severly attacks the lens. Since the scanning mirror and the optical fiber lie in the same axis, a special type of lens (Gradient-Index (GRIN)) is used for this work. The probe after complete assembly would resemble the schematic in figure 2. Note that the bottom half of one end of the GRIN lens is coated with metal. This is necessary to provide a reflecting surface to couple the optical fiber and the mirror.

Following the release of the structures in HF, the die needs to be rinsed in de-ionized (DI) water to get rid of the HF residues. This step can cause the free standing structures to collapse due the viscosity of water and to remain so even after the die is dried. To overcome this problem, the die is subject to critical point drying. A critical point dryer uses the property of fluids (carbon dioxide in this case) to co-exist in liquid and gaseous phase under certain conditions of temperature and pressure. When the fluid reaches this point, referred to as the critical point, the

chamber is rapidly evacuated to release all the gas. This leaves behind the sample dry in the chamber. To make this process effective, the die has to be introduced into the chamber when it is still wet. Since liquid  $CO_2$  does not mix with water, an intermediate solvent is needed. Alcohol is a good choice since it is miscible with both water and liquid  $CO_2$ . Isopropyl alcohol was used as the intermediate solvent in this work.

#### **1.5 Challenges in the Work**

The most critical stage in post processing is the substrate etching to make grooves for the optical fiber and the sloped sidewalls. Any method that will be used for this purpose has a good chance of attacking the devices on the front side since polysilicon has chemical properties similar to silicon. The etch depth targeted here covers almost the entire thickness of the substrate which is approximately 675 microns. Dry etching for such depths can be done only by deep reactive ion etching (DRIE). However, DRIE produces vertical sidewalls after etching; whereas, this work requires sloped sidewalls for reasons explained earlier. Wet etching is a simple solution and a good alternate to dry etching. There are several etchants available for wet etching of silicon <sup>[10]</sup> like mixture of nitric acid, hydrofluoric acid and acetic acid (HNA), potassium hydroxide (KOH) diluted in DI water, ethylenediamine pyrocatechol (EDP), tetramethyl ammonium hydroxide (TMAH) in DI water and hydrazine to name a few. HNA is an isotropic etchant which would cause severe undercuts when deep etching is needed. EDP and hydrazine are highly toxic and their use requires special apparatus. KOH and TMAH are anisotropic etchants and most frequently used. KOH was selected for this work because of its higher etching selectivity of 400 between (100) and (111) crystal orientations compared to TMAH of which selectivity is 50.

The front side of the chip contains polysilicon devices and photoresist links, both of which are rapidly etched in KOH. The etching rate of silicon in KOH is  $1.4 \mu m$  per minute at

85°C. <sup>[9]</sup> For a depth of 675  $\mu$ m, the chip would have to be immersed in the etch bath for at least 7 – 8 hours at that temperature. Any material used to protect the front side would have to endure throughout this etch duration. The pervasiveness of the etchant makes it essential for the masking material to provide a water tight seal. One of the main objectives of this thesis work has been to devise a technique to protect the front side of the chip during the KOH etching overcoming all the related problems. Various methods that were evaluated to protect the front side are discussed in the next section (1.6).

The presence of devices on the front side of the chip demands caution in the handling of the chip. Although the devices are not released before completing all the post-processing steps, extra care is necessary to prevent scratching of the surface to preserve the mirror surface. The slightest of scratches on the surface of mirrors could be detrimental to the functionality of the device.

A suitable insulating material is required to serve as mechanical link between the SDAs and mirror shafts as shown in figure 4. The dimensions of the required mechanical links are from a minimum of 30  $\mu$ m × 40  $\mu$ m to a maximum of 100  $\mu$ m × 40  $\mu$ m. Uneven topography (figure 9) formed by the shafts requires good sidewall coating for the insulating material. The topography made by these shafts is 4 microns and hence the insulating material would have to be at least 8 microns thick to form strong links. A thick photoresist is the most suitable material for this application. The thickness of the photoresist can be controlled by varying the spin coating speed. Also patterning the photoresist is a straightforward technique which results in well-defined patterns. AZ 9260 photoresist from MicroChemicals <sup>[11]</sup> is a positive tone photoresist that can achieve thickness around 30  $\mu$ m with a single spin cycle. <sup>[12]</sup> There are published works <sup>[13 - 14]</sup> that report on achieving 100  $\mu$ m thickness with multiple spin coats.



Figure 9. Cross section of the device showing the topographic variations due to the shafts. Figure not drawn to scale.

The problem in using a photoresist is that it is easily dissolved in common solvents like acetone and alcohol. These solvents are necessary to clean the chip between some processes. Also, the chip needs to be immersed in isopropyl alcohol when placed in the critical point dryer, which is an inevitable step. Chapter 3 deals with formation of the photoresist links and solving the shrinkage problem associated with the photoresist. It is to be noted that the photoresist patterning has to precede the KOH etching process. Reversing this order will make photolithography extremely difficult due to the fragility of the chip after KOH etching.

#### **1.6 Literature review**

#### KOH Etch Mask

Silicon anisotropic etching by KOH is a well-established technique and many different materials have been used as masking materials. The most frequently used ones are silicon nitride and silicon dioxide. Between these two materials, silicon nitride has better etch selectivity with silicon in KOH.<sup>[15]</sup> However, it is essential to use good quality nitride/oxide to ensure fail proof masking. Nitride/oxide deposited by low pressure chemical vapor deposition (LPCVD) has good quality, i.e. free of pin holes, long withstanding in KOH and conformal coating.<sup>[10]</sup> The LPCVD technique, however, has its limitation, i.e. the high deposition temperature (> 500°C) ambience which can be detrimental both to the metal layer on the scanning mirror and the photoresist links. Plasma enhanced CVD (PECVD) is a relatively low temperature (< 400°C) process, but it does not result in good step coverage and produces lots of pin holes.

Metals inert to KOH have also been used as masks. Works using chromium (Cr), <sup>[16]</sup> titanium tungsten alloy (TiW, as adhesion promoter for gold) and gold (Au), <sup>[17]</sup> nickel (Ni) <sup>[18]</sup> and platinum (Pt) <sup>[19]</sup> have been reported. Although these materials can serve as good masks, removal of these metals require use of strong acids which is not desirable as it might damage the devices. Wang et al <sup>[20]</sup> used a 50 nm layer of zirconium oxide (ZrO<sub>2</sub>) as etch mask. The material was dispensed in the form of sol-gel and required baking temperature of 600°C. This is again a high temperature process. Hydrogenated amorphous carbon was used as etch mask by Fissore et al.<sup>[21]</sup> A 50 – 80 nm of this compound was deposited on silicon using PECVD. Although the material succeeded in protecting underlying silicon, the method was tested only for 1 hour, and hence, prolonged experiment is necessary. There is also no mention of how the compound was removed. Similarly, silicon carbide (SiC)<sup>[22]</sup> deposited by PECVD at 300°C and followed by an annealing at 450°C was tried as a mask. The etch duration in the report was only 20 minutes. Another group <sup>[23]</sup> reported using RF-sputtered glass onto the substrate. This also required a high temperature treatment of 550°C for 3.5 hours to anneal the glass. Apart from thin film coating, researchers have tried other techniques for protection as well. Kung et al <sup>[24]</sup> used a mechanical

clamp made of Teflon block with Teflon O-rings to make water tight seal. There are wafer holders commercially available <sup>[25]</sup> based on this technique that can work with chips of any dimension as well as whole wafers. The downside of this method is that the force generated by the clamping screws could be large enough to break the wafer at later stages of etching. There is also a possibility of leakage caused by curvature resulting from the long etching durations. Ivanov et al <sup>[26]</sup> introduced a new technique based on the meniscus property of liquid. In this method, the wafer side that needs to be etched is brought into close proximity of the etchant till it barely makes contact by meniscus. The wafer is then held in that position by Teflon tape. A Teflon O-ring is attached on the front side of the wafer periphery to avoid any spillage of the etchant. The problem with this method is that it requires continuous monitoring of the liquid level and may require adjustments in the placement of the sample if there is loss of the etchant due to evaporation. Also, since the method uses only the surface of the etchant, it is not efficient in the use of the etchant especially when batch processing is needed.

Polymer materials have gained popularity as a protecting mask in recent years due to the ease of use. Bodas et al <sup>[27]</sup> tried etching with polymethyl methacrylate (PMMA) as a masking layer. Two different techniques of dispensing the material were compared based on their adhesion to silicon. The adhesion to substrate was poor in case of the spin coating. Sputtering resulted in better bonding with the substrate, but the material was delaminated after 5 hours of etching in KOH. Recrystallized parylene <sup>[28]</sup> and a polymer blend of a polystyrene-co-polyacrylonitrile (SAN) copolymer and a polymer-containing epoxy <sup>[29]</sup> were also tried as etch masks. Both of them had problems with undercutting. Although the material themselves were inert to KOH, the undercuts were due to weakening of the interface between the polymer and the substrate. Brewer Science Inc <sup>[30]</sup> manufactures the Protek® series of polymers that can provide

protection against KOH. However, there are reports  $[^{31-32}]$  of both Protek B2® and Protek B3® delaminating on the edges up to 2 mm which caused undercuts after 3 hours and 7 hours of etching, respectively. In order to overcome the delamination problem, it would require removal of the Protek coating and re-coating of a fresh layer. This step would have to be repeated until the desired etch depth is achieved. And as the wafer gets thinner, it may not be possible to recoat the material due to concern of wafer breakage.

One polymer that has drawn attention of people in different areas of research is PDMS. PDMS has excellent chemical stability <sup>[33]</sup> and good mechanical properties. <sup>[34]</sup> It also finds use in optical MEMS due to its transparency. A simple yet novel technique using PDMS and glass as protective materials is introduced in this thesis that provides protection of front side for at least 10 hours. A detailed account of the protection method and results are provided in chapter 2.

#### • Formation of Photoresist Links

Photoresist is the most commonly used substance in device fabrication. Although it is used mostly to make patterns, its applications vary widely. It is used as a mold for electroplating  $^{[13 - 14]}$  and soft lithography.  $^{[35]}$  Subjecting the photoresist to high temperature (above its glass transition temperature) causes the material to soften and flow. This property is used to make lens structure.  $^{[35 - 37]}$  High temperature treatment also makes the photoresist chemically stable and improves adhesion with the substrate. This process is referred to as hard bake. Photoresist inertness to common solvents can be achieved by heat treatment. Chapter 3 contains details of the processes followed to achieve chemical stability in the AZ 9260 photoresist.

## CHAPTER 2

# FRONT SIDE PROTECTION DURING SILICON SUBSTRATE ETCHING

The silicon substrate in the MUMPs® chip needs to be etched from the backside to create grooves for the optical fiber and provide sloped sidewalls. The etch depth in the case of sidewall slopes goes all the way up to the silicon nitride layer on the front side. This means the whole substrate needs to be etched. So the target depth is approximately 675-microns. The presence of polysilicon devices on the front side of the chip necessitates the use of some protection method during the etching process.

The etching bath is prepared by mixing KOH pellets, DI water and isopropyl alcohol (IPA) in a ratio of 70 g: 190 ml: 40 ml in a thick-walled 2-L glass beaker. IPA is added to the solution to get smooth etched surface and to improve the etching selectivity between (100) and (111) crystal orientations. The etch bath is heated on a hot plate. A magnetic stirrer is placed in the beaker and spun at 80 rpm to keep the temperature and concentration of the etchant uniform. A sheet of aluminum foil is used to cover the opening of the beaker to prevent evaporation.

Anisotropic etching of silicon in KOH is typically performed at 80°C. The etch rate at this temperature is 1.4  $\mu$ m/minute. <sup>[15]</sup> The protection method described here uses an industrial hot glue that has a melting temperature around 80°C. To avoid any adverse effects due to the melting of the hot glue to the protection setup, the etch bath is maintained at a temperature below 75°C. The etching rate of silicon varies with the temperature of the etching bath. Assuming that the etching rate at a temperature of around 75°C is 1  $\mu$ m/minute, etching duration of approximately 10 hours is necessary to etch 675 microns. This duration set the target for the protection tests.

#### **2.1 Materials**

PDMS is a silicone polymer that finds use both in its liquid state and when solidified. Solidification is achieved through crosslinking of the polymeric chains usually with the aid of a catalyst. When solidified, the material becomes like rubber and turns hydrophobic so that it can be used to waterproof devices. <sup>[38]</sup> PDMS is available as a commercial product from many manufacturers. In this work, Sylgard 184®, <sup>[39]</sup> 2 part elastomer from Dow Corning <sup>[40]</sup> is used, which consists of a base and a curing agent (catalyst). Curing is initiated by mixing the base and the catalyst in 10:1 ratio by weight. The mixture can be either cured at room temperature for 24 hours or at elevated temperatures <sup>[41]</sup> for a shorter time. PDMS can be molded to any shape before curing. It can also be spin coated to a desired thickness. Although it is difficult to pattern the PDMS after curing due to its inertness to most common laboratory chemicals, there are reports <sup>[42–43]</sup> that describe wet and dry techniques of etching the material to pattern it.

All etching tests were performed on 500  $\mu$ m thick single-side polished p-type silicon dies of 1.5 cm × 1.5 cm dimensions. All the samples were cleaned prior to use in a boiling solution of piranha (mixture of concentrated sulfuric acid and 30% hydrogen peroxide in the ratio of 2:1). 10 minutes of immersion is sufficient to devoid the samples of any organic impurities. The polished side of the die had a layer of LPCVD silicon nitride that was used as a mask during the substrate etching. A 1-micron-thick aluminum layer was deposited on the unpolished side by thermal evaporation and patterned by lift-off process to indicate the beginning of etching if there was an attack on the protected side. Aluminum was used since it rapidly dissolves in KOH.

#### 2.2 Method

The etch protection method used in this work relies on the inertness of both PDMS and glass to the KOH etching solution. Different methods have been tried in the past with PDMS as a

protective layer. <sup>[44 - 48]</sup> Khoo et al <sup>[44]</sup> covered the whole wafer with PDMS except the backside (see figure 10). The etching duration was, however, very short in their work, just enough to etch a 50 µm-thick membrane of silicon. Considering an approximate etch rate of 1 µm/minute, the etching would not exceed 1 hour. Brugger et al <sup>[45]</sup> placed a thick PDMS ring on the backside of the wafer (facing down) and used the adhesive nature of PDMS to confine a small quantity of KOH within the ring (see figure 11). Heat was provided by an infrared lamp. Obreja et al <sup>[46]</sup> and Korivi et al <sup>[47]</sup> covered the front side of the wafer by dipping in (see figure 12) and spin coating PDMS, respectively. Since the PDMS remains in direct contact with the wafer surface, there might be complications in removing PDMS after etching is completed. Obreja et al. <sup>[46]</sup> reported on using chemicals to remove PDMS from the wafer. There is, however, no mention of what kind of chemical was used. This is a nontrivial process since PDMS is inert to most chemicals and requires either strong acids <sup>[33]</sup> or specific chemicals <sup>[42]</sup> for removal. Dry etching with CF<sub>4</sub> and O<sub>2</sub> plasma <sup>[43]</sup> cannot be used since it also attacks silicon and polysilicon. Instead, a PDMS layer can be peeled by hands, <sup>[47]</sup> but it is not desirable to apply shear force on the wafer surface especially when the wafer becomes fragile after substrate etching.

All the methods described above coat a protective layer directly on the front side of the wafer. On the contrary, the method presented in this thesis attempts to isolate the protected side completely from PDMS by placing the die faced down on a microscope slide. The microscope slide provides an impregnable barrier against KOH. The emphasis now lies in protecting the edges of the die from where KOH can leak into and attack the protected side. PDMS is used to seal these edges. Similar method was reported with silicone rubber as a sealing agent for etching in TMAH, <sup>[51]</sup> however, with no process details. In addition to testing the etch protection technique, this work also attempts to study the effects of the curing condition of PDMS on its



Figure 10. Illustration of the PDMS protection method by Khoo et al, adapted from [43]. Figure shows the cross section of the setup. The molded PDMS is the actual protection layer. Cr-coated PDMS is used as a de-bonding agent between the spin coated PDMS layer and a molded PDMS layer. Figure not to scale.



Silicon wafer

Figure 11. Isometric view of the PDMS seal ring protection method used by Brugger et al adapted from [44]. Figure shows a PDMS seal ring on top of a silicon wafer. The space inside the ring is used to hold the KOH etching solution. Figure not to scale.



Figure 12. Cross-section view of the silicone rubber protection method used by Obreja et al, adapted from [45]. Figure shows a silicon wafer immersed in silicone rubber with one face exposed. The silicone rubber is also attached to a glass slide. Figure not to scale.

chemical stability in KOH and effectiveness as a sealing agent.

Glass (microscope slide, dimension:  $3^{\circ} \times 1^{\circ} \times 1 \text{ mm}$ ) was used to provide additional mechanical strength to avoid damage to the die while handling. A (111) silicon wafer would have served the purpose as well, but glass was chosen due to its transparency that allows visual inspection of the die during etching. An industrial hot melt glue <sup>[48]</sup> (hereafter referred to as hot glue) was used to secure the die to the microscope slide to prevent any slipping during the application of PDMS. The hot glue is available in the form of sticks that can be melted and dispensed using a heating gun. Melting temperature of the glue used in this experiment was around 80°C. However, the glue starts to soften beyond 65°C. Although the etching bath temperature (75°C) was higher than the softening temperature of glue, it did not affect the bonding between the glue and the chip. Using glue has another advantage of making a watertight seal between the die and the microscope slide. Removing glue from the die can be easily done by immersing the die in boiling acetone for 5 – 10 minutes. The glue layer delaminates completely

without leaving behind any residue. The presence of glue layer between the die and microscope slide does not affect the visibility since the hot glue is also transparent.

Application of PDMS and forming a seal is explained as follows. A mixture of the PDMS base and curing agent was prepared in 10:1.2 ratio. A slightly more quantity of the curing agent than the recommended one-eleventh resulted in better curing. The mixture was then placed in a vacuum jar for 30 minutes to degas it. Meanwhile, a mold was prepared on the die and the microscope slide to obtain 2 mm thick PDMS seal. Kapton® tape <sup>[49]</sup> was used to define the boundaries of the mold. A small lump of hot glue was applied on the etching side of the die to keep that region free of PDMS. This region defined the window for KOH etching solution to access the etching side of the wafer. PDMS was then poured into this mold. The assembly was then degassed again in a vacuum jar. Following the degassing, the setup was either placed in a convection oven or left at room temperature for curing. Various curing temperatures were tried. Figures 13 and 14 illustrate the PDMS sealing process.



Figure 13. Isometric view of the setup for making the PDMS seal. A patterned silicon die is placed face down on the microscope slide secured with hot glue. Kapton® tape serves as a mold. PDMS mixture is poured into the mold for curing. A hot glue lump in the center inhibits PDMS in the region, defining the window for etchant access.



Figure 14. Isometric view of the setup after PDMS is cured. The mold is removed by peeling with hand. Hot glue in the center is removed by piercing it with a sharp tipped flat screw driver and plucking it out when it is still soft immediately after curing.

Following the curing process, the hot glue lump in the center was immediately removed using a sharp tipped flat screw driver when it was still soft.

Extreme care has to be taken while removing the glue lump so as not to disturb the surrounding PDMS and cause its delamination. It is to be noted that the adhesion of PDMS to the underlying microscope slide and edges of silicon is by weak Van der Waal's force. Figure 15 shows the cross-sectional view of the setup.

The sample was then immersed in the etching bath and held in place with a Teflon sample holder. The etching was stopped if there was any leakage observed on the edges or if the PDMS itself was getting attacked by KOH.

The manufacturers recommend <sup>[41]</sup> the following curing conditions.

- ✓  $150^{\circ}$ C for 15 minutes,
- ✓  $100^{\circ}$ C for 1 hour,
- ✓  $65^{\circ}$ C for 4 hours,
- $\checkmark$  room temperature for 24 hours.

They also mention that although PDMS cures in 24 hours at room temperature, it takes at least 7



Figure 15. Cross-section view of the etch protection setup after PDMS is cured and mold tapes are removed. The dashed lines show the anisotropic etch regions forming the grooves. Figure not to scale.

days for the material to gain its complete mechanical properties. To test the effect of curing condition of PDMS on its chemical resistance to KOH, several samples were prepared by the method described above at different curing temperatures. Following curing conditions were tried.

- $\checkmark$  room temperature for 48 hours,
- $\checkmark$  room temperature for 7 days,
- ✓  $65^{\circ}$ C for 4 hours and
- ✓ 80°C for 2 hours.

Curing temperatures beyond 80°C were not tried since the glue would lose its consistency beyond this temperature. Different etching bath temperature was also tried from 65°C to 80°C to study its influence on the PDMS.

#### 2.3 Results and Discussion

Etching duration of 10 hours was targeted assuming an etch rate of 1  $\mu$ m/minute for the temperature range of 70°C to 75°C which is sufficient to etch through the complete thickness of

the silicon die. However, the etch rate at this temperature range was found to be just 0.6  $\mu$ m/minute. This shows that the etch rates are very sensitive to temperature changes. With this rate it would take approximately 17 hours to completely etch through the MUMPs® chip.

Table 1 summarizes the results of the etching experiments. There were 36 test samples prepared out of which 15 (batches A and B) were cured at 70°C for 4 hours. Among them 12 samples (batch A) were treated to room temperature after cure for 24 hours before beginning the etching. The other 3 (batch B) were immediately subject to etching. PDMS on all of these 3 samples got completely dissolved in KOH within 5 hours. The samples in the batch A were tested up to 10 hours with no signs of attack on the front side of the die or to the PDMS till the end. Only 3 samples out of the batch A had aluminum patterns on the front side and silicon nitride on the backside. Silicon nitride was patterned with CF<sub>4</sub> and O<sub>2</sub> plasma using AZ 9260 photoresist as the etch mask. Patterning was not necessary for the rest of the samples since there were already 3 samples that survived etching. The rest of the samples had their polished side attached to the microscope slide. This made it easier to detect any attack to the front side.

Batch	No. of samples	Curing temp. (°C)	Curing duration (hours)	24 hours wait period	Results
А	12	70	4	Yes	10 hours (S)
В	3	70	4	No	5 hours (D)
С	3	80	5	No	5 hours (D)
D	3	80	2	Yes	10 hours (S)
E	3	60	5	No	5 hours (D)
F	5	65	4	Yes	10 hours (S)
G	3	Room temp.	48	-	5 hours (D)
Н	3	Room temp.	168	-	10 hours (S)

Table 1. Summary of the KOH etching results. (S) – survived without any signs of attack, (D) – PDMS attacked severely or completely destroyed

Similar results were obtained with other samples. 6 samples (batches C and D) were cured at 80°C. Among them, 3 samples (batch C) were cured for 5 hours and immediately immersed in the etching bath. The other 3 (batch D) were cured for 2 hours and let to stay at room temperature for 24 hours before etching. Again, PDMS on the batch C samples got completely dissolved in KOH within 5 hours; whereas batch D survived for 10 hours. 3 samples (batch E) were cured at 60°C for 5 hours, and immersed in the etching bath immediately. The PDMS layers on these samples were completely attacked in 5 hours. 5 samples (batch F) were cured at 65°C for 4 hours, and left at room temperature for 24 hours. PDMS on these samples remained unaffected at the end of the etching duration of 10 hours. The last set of 6 samples was cured at room temperature for 48 hours (batch G) and 7 days (batch H). As stated by the manufacturer, PDMS cured at room temperature needs 7 days to gain its full mechanical strength even though it cures within 24 hours. The results obtained after etching corroborate this fact since the samples cured for 7 days endured etching for 10 hours but the others were completely destroyed. It was observed that the curing temperature did not have any influence on the chemical stability of PDMS in KOH. However, the duration of curing affected the quality of PDMS. It seemed that the quality of PDMS was better when it was left at room temperature for 24 hours after curing and before immersing in KOH.

Results from earlier work <sup>[33, 45, 47 and 51]</sup> also support the experimental observations made here. There was either a wait time of 24 hours before beginning the etching or the curing duration itself was 24 hours due to which the PDMS survived up to 10 hours <sup>[51]</sup> or more in the earlier published works. <sup>[33, 45, 47 and 51]</sup>

12 test samples (batches B, C, E and G) out of 36 failed because they were not rested for 24 hours before beginning the etching. PDMS on the remaining samples looked intact (except for

slight delamination at the inner edge on few samples) and almost like a fresh layer even after 10 hours of etching. It seemed like they could be used for much longer etching duration. Brugger et al <sup>[45]</sup> mentioned about the PDMS seal ring being reusable many times for etching. 4 of the successful samples were again introduced to etching bath to see how much longer the PDMS would endure. Not surprisingly, the protected side of these dies remained unharmed till the substrates were etched through. Figure 16 shows pictures of one of the samples before and after 10 hours of etching.

Detaching the die from the microscope slide after etching is a simple task. First, the PDMS layer is manually peeled off. Since PDMS overlaps the chip in only a small area on the edges, peeling PDMS applies very small amount of force on the chip and hence does not disturb the setup or damage the chip. The layer usually peels off cleanly as a whole. Since the die is attached to the microscope slide by hot glue, the glue needs to be melted first before pulling the die apart. Placing the sample on a hot plate at around 100°C turns the glue into liquid. The die is now slowly pushed to the edge of the microscope slide until it starts protruding out. At this point, the die can be held on the edge by tweezers, detached gently from the microscope slide and immersed in a beaker of boiling acetone. This makes any remaining glue on the die to be peeled off cleanly in acetone. Figure 17 shows some of the failed test samples. These samples were not with silicon nitride or aluminum layers. The attack to PDMS is clearly evident in figures 17 (a) and (b).

The etching duration of samples in figure 17 (a) and (b) was less than 5 hours, whereas the sample in 17 (c) underwent 10 hours of etching. As seen in the figure, there is no sign of degradation in the PDMS material for the sample in figure 17 (c). The effect of etching on the chemical structure of PDMS was also studied by comparing the Raman spectroscopy of an unetched an un-etched PDMS sample against one of the samples that survived for 10 hours in KOH. Figure 18 shows the Raman spectra of the un-etched and etched PDMS samples. The peaks in a Raman spectroscopy plot denote the chemical composition of a material. Any shifts in the peaks or presence/absence of the peaks would indicate a change in the chemical composition of the material.



Figure 16. Pictures of one of the samples from batch A (cured at 70°C for 4 hours). (a) and (b) show backside and front side of the die before etching respectively. (c) and (d) show backside and front side of the die after 10 hours of etching in KOH respectively. The etched pits in (c) are 350  $\mu$ m deep. PDMS is seen as a transparent layer surrounding the die in all the 4 pictures.





Table 2 lists the bonds corresponding to each of the characteristic peaks in PDMS <sup>[50]</sup> marked in figure 18.

From the figure 18, it can be observed that there are no changes in the characteristic peaks of PDMS for the etched sample except for a shift in the intensity. The intensity shift is due

to the roughening of the surface of PDMS after etching. This suggests that the PDMS remains unaffected by the 10 hours of KOH etching.



**Raman spectra of PDMS** 

Figure 18. Raman plot of an un-etched PDMS sample and 10 hours etched PDMS sample. The characteristic peaks are labeled (see table 2) and each one denotes vibrational peaks due to the molecular bonding present in the material.

Table 2. Description of the characteristic Raman peaks for PDMS. Adapted from [50].

Peak	Wavenumber (cm <sup>-1</sup> )	Description
1	488	Si-O-Si symmetric stretching
2	687	Si-CH <sub>3</sub> symmetric rocking
3	708	Si-C symmetric stretching
4	787	CH <sub>3</sub> asymmetric rocking + SiC asymmetric
	/8/	stretching
5	862	CH <sub>3</sub> symmetric rocking
6	1262	CH <sub>3</sub> symmetric bending
7	1412	CH <sub>3</sub> asymmetric bending
8	2907	CH <sub>3</sub> symmetric stretching
9	2965	CH <sub>3</sub> asymmetric stretching

## **CHAPTER 3**

# FORMATION OF PHOTORESIST LINKS

Photoresist links are necessary to provide mechanical coupling between the SDA shafts and the mirror shafts while maintaining electrical isolation. The photoresist chosen in this thesis work is AZ 9260 since it can be spin coated to form thick layers (> 5  $\mu$ m). Along with their functionality of serving as links, the photoresist should be able to survive common organic solvents like acetone and methanol or IPA. The following sections describe the steps followed to form photoresist links in detail.

#### **3.1 Materials**

AZ 9260 is a positive-tone novolac-based photoresist from MicroChemicals. It is widely used as electroplating molds due to the thickness that it can achieve. Due to its semi-transparent nature, patterning the resist is possible up to thickness of 100  $\mu$ m with a UV source of a typical contact aligner. The patterns exhibit a well-defined profile with near-vertical sidewalls even at 100-micron thickness. <sup>[13 - 14]</sup> The recommended <sup>[12]</sup> soft bake treatment by the manufacturer is 100°C on a hot plate for 1 minute for every 1  $\mu$ m of thickness. Baking conditions vary when a convection oven is used due to the different nature of heat transfer mechanism. Like some other thick resists, AZ 9260 requires a rehydration time of at least 10 minutes following the soft bake. Rehydration time is just a resting time at room temperature for the resist after the soft bake to regain some of its lost water content from the humidity in the air. The manufacturers recommend this to ensure good development.

Following the exposure, the resist can be developed with AZ 400K developer (MicroChemicals) diluted in DI water in a ratio of 1:4, either by immersion or spray. The development time again depends on the thickness and the exposure dose. Hexamethyldisilazane

(HMDS) from Transene Company<sup>[52]</sup> was used to promote adhesion between the photoresist and the substrate. HMDS is a low viscosity liquid that can be dispensed on the substrate by spin coating prior to coating the photoresist.

#### 3.2 Method

The target thickness of the photoresist layer was 8  $\mu$ m as described in chapter 1. However, the thickness used in this thesis work is 10  $\mu$ m. Based on the spin coating curve of AZ 9260 and several trial runs on the spincoater®, Specialty coating Systems Inc. (model P6700), <sup>[53]</sup> 2400 rpm for 30 seconds was found to be the right condition. Due to the high viscous nature of the resist, there was a problem of edge bead that needed to be solved. This problem is more pronounced since the micro-mirror chip has a very small margin (0.5 mm) around the edges to utilize the maximum possible area of the foundry-serviced die. The following steps describe the technique used to remove the edge bead.

Edge beads arise due to the viscosity of the resist that drags back and accumulates some of the resist at the edge of the wafer during spin coating. The following technique pushes the edge bead area off the edge of the micro mirror chip (hereafter referred to as MUMPs chip) by virtually extending the boundary of the chip. This is achieved by placing four  $3^{"} \times 1^{"}$  microscope slides in close proximity to the edges of the chip as illustrated in figure 19. It is important to ensure that the surface of the microscope slides and the chip are at the same level for the technique to work. A  $3^{"} \times 2^{"}$  microscope slide is used as a base for this setup. The  $3^{"} \times 1^{"}$ microscope slides are cut to fit within the  $3^{"} \times 2^{"}$  microscope slide. A mechanical spring (a small piece of thin hollow plastic tube) is used to compensate for the difference in thickness between the chip (~700 µm) and microscope slides (1 mm). The small microscope slides along with the chip (face up) are fixed to the base using hot glue (refer to chapter 2). The quantity of glue used should be just enough to suffice the need to avoid penetration of the glue through the edges to the front side of the chip. The whole assembly is done on a hot plate to prevent the glue from hardening. Another  $3^{\circ} \times 2^{\circ}$  microscope slide is placed on top of the whole assembled substrates and pressed gently to ensure that the surfaces are leveled. The hot plate is now turned off and the setup is let to cool off and stay fixed. The leveling large microscope slide is removed from the top and the setup is now ready for spin coating. Figures 19 and 20 illustrate the technique. The above described technique was developed by Dr. Martin Feldman and Dr. Dooyoung Hah and tested by Mr. Ragavendra Murthi.



Figure 19. Isometric view of the leveling setup after complete assembly. Figure not to scale.

Following the spin coating of the photoresist, the resist needs to be soft baked. Soft baking on a hot plate would melt the glue under the chip and make the chip pop up due to the force from the mechanical spring. This would prevent proper heat conduction to the photoresist on the chip. Therefore, the soft baking was done in a convection oven at 87°C for 25 minutes (baking condition obtained from trials). Even though the chip also pops up in the oven, this does not affect the temperature distribution since the heating mechanism is due to convection.



3"×1" microscope slides

Figure 20. Cross-sectional view of the leveling process. The large microscope slide on the top is for temporary use and will be separated from the assembly prior to spin coating. Figure not drawn to scale.

The dummy large microscope slide is again used to level the setup before exposure. Exposure was done using a Karl Suss MJB3 mask aligner. Exposure dose of 1000 mJ/cm<sup>2</sup> was sufficient to obtain good patterns with a minimum feature dimension of  $\sim$ 30 µm.

Following the exposure, the chip was detached from the assembly to be used in further processing. Glue residue on the backside of the chip was scraped off gently using a razor blade. The chip was then treated to  $O_2$  plasma (ashing <sup>[54]</sup>) in the RIE apparatus at EMDL for 15 minutes to clear any photoresist residues from the developed area. Excessive plasma treatment should be avoided to preserve the dimensions of the patterns. <sup>[54–55]</sup> The etch rate of AZ 9260 in  $O_2$  plasma (120 W, 240 mTorr) was found to be 75 nm/minute. To improve adhesion between the resist and the chip, and to make the resist inert to organic solvents, it is essential to hard bake it. <sup>[56]</sup> The hard baking process has some side effects that can be either beneficial or detrimental

in general sense. During hard bake, the resist turns soft and tends to deform slightly on the edges. This phenomenon has been used to reduce the line width and line edge roughness in the resist. <sup>[57]</sup> <sup>-58]</sup> Increasing the hard bake temperature beyond the softening point causes the resist to flow and assume a spherical shape due to surface tension. This effect is widely used to produce micro lens or micro lens molds. <sup>[35 - 37]</sup> A secondary effect of this treatment is increase in the line widths. <sup>[59]</sup> Heating beyond the reflow temperature range leads to complete evaporation of the solvent in the resist and initiates crosslinking of the polymers in the resist which causes the resist to shrink in volume. The chemical stability of the photoresist after hard bake is due to this crosslinking. This property is used in this work for strengthening of the photoresist against common organic solvents like acetone and alcohol. Similar work has been done earlier to chemically stabilize the photoresist lens structure. <sup>[36]</sup> The main objective in this thesis was to find the optimum thermal treatment necessary for chemical resistance and yet preserve the shape of the resist patterns. The effect of surface topography on the resist shape is also studied.

#### **3.3 Results and Discussion**

As stated earlier, the effect of hard baking on a hot plate is different from that in a convection oven. The hard baking process for this thesis work was carried out in a convection oven. The typical hard bake temperature recommended by the manufacturer for AZ 9260 is around 140°C. Different temperatures above 140°C were tried to find the optimum temperature for this specific application. The hard baking duration was 1 hour for all temperatures. Initial tests were performed on bare silicon wafers to find the minimum temperature at which the resist became inert to acetone and IPA. It was found that the patterns baked at 140°C and 160°C got completely dissolved in acetone and IPA within 5 – 10 minutes of immersion. The patterns baked at 180°C were immersed in acetone or IPA for 1 hour and they seemed to be unaffected to

the naked eye. Inspection under a microscope showed some discoloration in the resist. Results were the best for the samples baked at 200°C. The resist remained intact after immersion in acetone or IPA for more than 1 hour. This temperature can be considered as the minimum required for passivation.

The next step was to examine changes in the resist profile. There was no change in the resist profile for hard bake temperatures of 140°C and 160°C. The resist edges started bulging slightly for 180°C hard bake and the extent was greater at 200°C and 220°C. The resist features also shrunk by approximately 7% in length at 200°C. The following figures show the top views of resist patterns for different baking conditions.

Apart from the bulging and shrinking, there was also a change in the color of the resist after hard bake. The photoresist turned from semi-transparent after exposure to completely opaque and reddish brown for hard bake beyond 180°C.

The process was next tried on the MUMPs chip. Although there was no bulging of the resist, it shrunk considerably in both directions. Figure 25 compares the mask layout against the actual resist profile to show the extent of shrinkage. The reason for this shrinkage is the topography of the substrate due to devices (figure 9). As shown in figure 9, the thickness of the resist does not remain the same throughout the pattern. Figure 26 shows the severity of shrinkage in one of the devices, where the shafts are disengaged. It was found that the extent of shrinkage varied with dimension. Shrinkage reduced as the length of the pattern increased, except for the resist link at 25 microns. Figure 27 shows the shrinkage measurement data. Despite the shrinkage, the hard bake step cannot be avoided since it is essential for the chemical strengthening. However, the shrinkage can be compensated for by adding a bias in the mask patterns.



Figure 21. Photoresist patterns after exposure. No hard bake. The dimensions of the patterns are (a) 25  $\mu$ m  $\times$  27  $\mu$ m, (b) 95  $\mu$ m  $\times$  27  $\mu$ m and (c) 41  $\mu$ m  $\times$  27  $\mu$ m. The images were captured at 25X magnification in a bright field optical microscope.



Figure 22. Photoresist patterns of figure 21, after hard bake at 180°C for 1 hour in a convection oven. Although there was no change in the effective dimensions of the patterns, there was slight bulging at the edges as can be seen. The dimensions of the patterns are (a) 25  $\mu$ m × 27  $\mu$ m, (b) 95  $\mu$ m × 27  $\mu$ m and (c) 41  $\mu$ m × 27  $\mu$ m. The images were captured at 25X magnification in a bright field optical microscope.



Figure 23. Photoresist patterns of figure 21, hard baked at 200°C for 1 hour in a convection oven. The bulging at the edges is more prominent here and there is also shrinkage in the dimensions at the corners. The dimensions of the patterns are (a) 25  $\mu$ m × 27  $\mu$ m, (b) 95  $\mu$ m × 27  $\mu$ m and (c) 41  $\mu$ m × 27  $\mu$ m. The images were captured at 25X magnification in a bright field optical microscope.



Figure 24. Photoresist patterns originally measuring (a) 28  $\mu$ m × 35  $\mu$ m, (b) 105  $\mu$ m × 35  $\mu$ m, and (c) 48  $\mu$ m × 35  $\mu$ m, after hard bake at 220°C for 1 hour. Dimensions after hard bake are (a) 26  $\mu$ m × 32  $\mu$ m, (b) 105  $\mu$ m × 34  $\mu$ m, and (c) 51  $\mu$ m × 34  $\mu$ m. The images were captured at 25X magnification in a bright field optical microscope.



Figure 25. Comparison of the photoresist dimension in the layout (right) against the resist dimension after hard bake at 200°C. The dotted square in the left picture shows the actual photoresist dimension before hard bake.

The hard baked (200°C) photoresist was exposed to UV light at a dose of 3000 mJ/cm<sup>2</sup> to see if the UV radiation can weaken the links. This had no effect on the photoresist and it still continued to be inert to acetone and IPA.



Figure 26. Top view of the SDA chain on one of the devices. Shrinkage in the photoresist caused the SDA shafts to be disengaged from the mirror shaft.



Figure 27. Plot of the percentage shrinkage in the photoresist link length as a function of the original dimensions for hard bake temperature of 200°C for hour.

## **CHAPTER 4**

# CONCLUSION

The challenges present in the post-MUMPs processing of the scanning mirror have been successfully addressed in this thesis work. The techniques developed in this thesis work can also be used for other applications with some customization, if necessary.

The front side protection method described in chapter 2 proves to be a cost-effective and simple technique. The absence of any influence of the curing temperature on the quality of PDMS allows for flexibility in the thermal treatment of the material depending on applications. The experiments also show the necessity of a resting time (approximately 24 hours) to ensure complete chemical stability of the Sylgard 184® silicone elastomer, a new data that has not been reported before. Although the tests were performed for only 10 hours of etching duration, the condition of PDMS at the end of etching show that the method can provide protection for more than 10 hours. In addition, several samples survived through-wafer etching experiments.

Photoresist strengthening has already been tried in the past. However the experiments carried out in this work show that it is possible to retain the shape of the patterns and yet achieve chemical resistance by finding the optimum hard baking conditions. The extent of shrinkage of the photoresist was found to be consistent with repetitive experiments. Therefore, with the addition of a suitable bias in the mask design, shrinkage is not a serious problem.

# BIBLIOGRAPHY

[1] Tae-Sik Kim, Sang-Shin Lee, Youngjoo Yee, Jong-Uk Bu, Chil-Geun Park, and Man-Hyo Ha, "Large Tilt Angle Electrostatic Force Actuated Micro-Mirror," IEEE Photonics Technology Letters, November 2002, Vol. 14, No. 11, pp 1569 – 1571.

[2] Harald Schenk, Peter Dürr, Thomas Haase, Detlef Kunze, Udo Sobe, Hubert Lakner, and Heinz Kück, "Large Deflection Micromechanical Scanning Mirrors for Linear Scans and Pattern Generation," IEEE Journal Of Selected Topics In Quantum Electronics, September/October 2000, Vol. 6, No. 5, pp 715 – 722.

[3] O Tsuboi, Y Mizuno, N Koma, H Soneda, H Okuda, S Ueda, I Sawaki, F Yamagishi, "A rotational comb-driven micromirror with a large deflection angle and low drive voltage," The Fifteenth IEEE International Conference on Micro Electro Mechanical Systems, 2002, pp 532 – 535.

[4] Anandi Kalyan Dutta, "The development of compact optical system for forward-looking endoscope," http://etd.lsu.edu/docs/available/etd-11182010-193155/, Master's thesis, 2010

[5] T Akiyama, K Shono, "Controlled stepwise motion in polysilicon microstructures," Journal of Microelectromechanical Systems, Sep 1993, Volume: 2 Issue:3, pp 106 – 110.

[6] Peyman Honarmandi, Jean W. Zu and Kamran Behdinan, "Analytical study and design characteristics of scratch drive actuators," Sensors and Actuators A: Physical, May 2010, Volume 160, Issues 1-2, pp 116-124.

[7] T Akiyama, D Collard, H Fujita, "Scratch drive actuator with mechanical links for self-assembly of three-dimensional MEMS," Journal of Microelectromechanical Systems, Mar 1997, Volume: 6 Issue:1, pp 10 - 17.

[8] MEMSCAP Inc., 12 TW Alexander Drive, Building 100, Research Triangle Park, NC 27709, USA

[9] PolyMUMPs design rules, MEMSCAP Inc, http://www.memscap.com/mumps/documents/PolyMUMPs.DR.v11.pdf [10] Marc J. Madou, "Fundamentals of microfabrication. The science of miniaturization," CRC Press, 2002, Second edition.

[11] MicroChemicals GmbH, Schillerstrasse 18, D-89077 Ulm, Germany.

[12] Thick photoresist processing, MicroChemicals, http://www.microchemicals.eu/technical\_information/thick\_resist\_processing.pdf.

[13] V Conédéra, B Le Goff and N Fabre, "Potentialities of a new positive photoresist for the realization of thick moulds," Journal of Micromechanics and Microengineering, June 1999, Volume 9, Number 2, pp 173 – 175.

[14] Magali Brunet, Terence O'Donnell, Joe O'Brien, Paul McCloskey and Seán Cian Ó Mathuna, "Thick photoresist development for the fabrication of high aspect ratio magnetic coils," Journal of Micromechanics and Microengineering, July 2002, Volume 12, Number 4, pp 444 – 449.

[15] K R Williams, R S Muller, "Etch rates for micromachining processing," Journal of Microelectromechanical Systems, Dec 1996, Volume: 5 Issue:4, pp 256 – 269.

[16] Alex K. Raub, Dong Li, Andrew Frauenglass, and S. R. J. Brueck, "Fabrication of 22 nm half-pitch silicon lines by single-exposure self-aligned spatial-frequency doubling," Journal of Vacuum Science & Technology B, Nov/Dec 2007, Volume 25(6), pp 2224-2227.

[17] F. E. Rasmussen, M. Hesche1 and O. Hansen, "Batch Fabrication of Through-Wafer Vias In CMOS Wafers for 3-D Packaging Applications," Proceedings of the 53rd Electronic Components and Technology Conference (ECTC) (ISBN: 0-7803-7791-5), 2003, pp 634-639.

[18] Lisby, T.H.; Hansen, O.; Branebjerg, J.A.; "Fabrication and characterization of flexible silicon substrates with electroplated gold leads," Proceedings of IEEE Sensors, 2002, vol.1, pp 568 - 571.

[19] Jürgen Hildenbrand, Jan Korvink, Jürgen Wöllenstein, Carolin Peter, Andreas Kürzinger, Falk Naumann, Matthias Ebert, and Frank Lamprecht, "Micromachined Mid-Infrared Emitter for

Fast Transient Temperature Operation for Optical Gas Sensing Systems," Proceedings of IEEE Sensors, 2008, Vol. 10, No. 2, pp 297 – 300.

[20] ZheZhe Wang, GaoYang Zhao, JieYuan Ma, HaiJun Peng and MeiLian Li, "Fabrication of inorganic film gratings and study on their diffraction properties," SCIENCE IN CHINA SERIES E: TECHNOLOGICAL SCIENCES, Nov 2008, Volume 51, Number 11, pp 1995-2000.

[21] Alfeu Fissore, Marco AR Alves, Edmundo da Silva Braga and Lucila Cescato, "Selective area deposition of a-C:H films as masks for anisotropic etching of crystalline silicon in aqueous potassium hydroxide," Vacuum, Jan 1998, Vol 49, Issue 1, pp 49-50.

[22] Haixia Zhang, Hui Guo, Yu Wang, Guobing Zhang and Zhihong Li, "Study on a PECVD SiC-coated pressure sensor," Journal of Micromechanics and Microengineering, January 2007, Volume 17, Number 3, pp 426 – 431.

[23] J.W. Berenschot, J.G.E. Gardeniers, T.S.J. Lammerink and M. Elwenspoek, "New applications of r.f.-sputtered glass films as protection and bonding layers in silicon micromachining," Sensors and Actuators A: Physical, April 1994, Volume 41, Issues 1-3, pp 338-343.

[24] Joseph T. Kung, Andrew N. Karanicolas and Hae-Seung Lee, "A compact, inexpensive apparatus for one-sided etching in KOH and HF," Sensors and Actuators A, 1991, Volume 29, pp 209-215.

[25] AMMT Advanced Micromachining Tools GmbH, Anselm-Feuerbach-Str. 6, D-67227 Frankenthal, Germany, http://www.ammt.com/content/products/wetetching/index.html.

[26] Schultz, J. Ivanov, D.V. Farmer, K.R., "Meniscus interface etching of silicon in KOH in a hands-on introductory course in MEMS," University/Government/Industry Microelectronics Symposium, 2001. Proceedings of the Fourteenth Biennial, 2001, pp 153 – 157.

[27] Dhananjay S. Bodas, S.K. Mahapatra and S.A. Gangal, "Comparative study of spin coated and sputtered PMMA as an etch mask material for silicon micromachining," Sensors and Actuators A: Physical, May 2005, Volume 120, Issue 2, pp 582-588.

[28] Hsi-wen Lo, Wen-Cheng Kuo, Yao-Joe Yang, and Yu-Chong Tai1, "Recrystallized parylene as a mask for silicon chemical etching," 3rd IEEE International Conference Nano/Micro Engineered and Molecular Systems, 6-9 Jan. 2008, pp 881 – 884.

[29] J Dalvi-Malhotra, X F Zhong, C Planje, G Brand and K Yess, "A spin-on photosensitive polymeric etch protection mask for anisotropic wet etching of silicon," Journal of Micromechanics and Microengineering, February 2008, Volume 18, Number 2, Issue 2, pp 1-8.

[30] Brewer Science, Inc., 2401 Brewer Drive, Rolla, MO 65401 USA.

[31] G Canavese, S L Marasso, M Quaglio, M Cocuzza, C Ricciardi and C F Pirri, "Polymeric mask protection for alternative KOH silicon wet etching," Journal of Micromechanics and Microengineering, July 2007, Volume 17, Number 7, Issue 7, pp 1387-1393.

[32] Wenyu Song, "Two-dimensional Microscanners with T-shaped Hinges and Piezoelectric Actuators," http://etd.lsu.edu/docs/available/etd-02202009-182950/, Master's Thesis, 2009.

[33] Alvaro Mata, Aaron J. Fleischman and Shuvo Roy, "Characterization of Polydimethylsiloxane (PDMS) Properties for Biomedical Micro/Nanosystems," BIOMEDICAL MICRODEVICES, 2005, Volume 7, Number 4, pp 281-293.

[34] Miao Liu, Jianren Sun and Quanfang Chen, "Influences of heating temperature on mechanical properties of polydimethylsiloxane," Sensors and Actuators A: Physical, April 2009, Volume 151, Issue 1, pp 42-45.

[35] J B Orhan, V K Parashar, A Sayah, M A M Gijs, "Fabrication and Characterization of Three-Dimensional Microlens Arrays in Sol-Gel Glass," Journal of Microelectromechanical Systems, Oct. 2006, Volume: 15 Issue:5, pp 1159 – 1164.

[36] Myung-Geun Han, Yoon-Jung Park, Seoung-Hoe Kim, Byueng-Su Yoo and Hyo-Hoon Park, "Thermal and chemical stability of reflowed-photoresist microlenses," Journal of Micromechanics and Microengineering, March 2004, Volume 14, Number 3, pp 398 – 402.

[37] C. K. Chung and Y. Z. Hong, "Fabrication and analysis of the reflowed microlens arrays using JSR THB-130 N photoresist with different heat treatments," Microsystem Technologies, 2007, Volume 13, Numbers 5-6, pp 523-530.

[38] J J Bernstein, J Bottari, K Houston, G Kirkos, R Miller, B Xu, Y Ye, L E Cross, "Advanced MEMS ferroelectric ultrasound 2D arrays", IEEE Proceedings Ultrasonics Symposium, 1999, Volume : 2, pp 1145 – 1153.

[39] Sylgard 184® silicone elastomer, Dow Corning Corporation, http://www.dowcorning.com/applications/search/products/Details.aspx?prod=01064291&type=P ROD.

[40] Dow Corning Corporation, Corporate Center, PO Box 994, MIDLAND MI 48686-0994, United States.

[41] Curing temperatures for Sylgard 184®, Lindberg & Lund AS, Nygårdsveien 26, 1400 Ski, Norway. www.lindberg-lund.com/files/Tekniske%20datablad/DC-184-TD.pdf.

[42] Shuichi Takayama, Emanuele Ostuni, Xiangping Qian, J. Cooper McDonald, Xingyu Jiang, Phil LeDuc, Ming-Hsien Wu, Donald E. Ingber, and George M. Whitesides, "Topographical Micropatterning of Poly(dimethylsiloxane) Using Laminar Flows of Liquids in Capillaries," Advanced Materials, April 2001, Volume 13, Issue 8, pp 570–574.

[43] J. Garra, T. Long, J. Currie, T. Schneider, R. White, and M. Paranjape, "Dry etching of polydimethylsiloxane for microfluidic systems," Journal of Vacuum Science & Technology A, 2002, Volume 20, Issue 3, pp 975 – 982.

[44] Melvin Khoo and Chang Liu, "Micro magnetic silicone elastomer membrane actuator," Sensors and Actuators A: Physical, April 2001, Volume 89, Issue 3, pp 259-266.

[45] J. Brugger, G. Beljakovic, M. Despont, H. Biebuyck, N.F. de Rooij and P. Vettiger, "Low-cost PDMS seal ring for single-side wet etching of MEMS structures," Sensors and Actuators A: Physical, October 1998, Volume 70, Issues 1-2, pp 191-194.

[46] Paula Obreja, Raluca Muller and Elena Manea, "Silicon elastomer as a protective layer in 3D microfabrication of micro-opto-electro-mechanical systems," Sensors and Actuators A: Physical, April 1999, Volume 74, Issues 1-3, pp 24-26.

[47] N. S. Korivi, J. Hoffpauir, and P. K. Ajmera, "Texturing of silicon using a microporous polymer etch mask," Journal of Vacuum Science and Technology B, 2010, Volume 28, pp C6K8 – C6K12.

[48] Surebonder® industrial glue, FPC corporation, 335 Hollow Hill Drive, Wauconda, IL, US, 60084, http://www.surebonder.com/default.asp

[49] Kapton® tape, DuPont Electronic Technologies, US Rt. 23 South and DuPont Road, Circleville, OH 43113, USA, http://www2.dupont.com/Kapton/en\_US/

[50] Sung Chul Bae, Hyunjung Lee, Zhiqun Lin and Steve Granick, "Chemical Imaging in a Surface Forces Apparatus: Confocal Raman Spectroscopy of confined Poly(dimethylsiloxane)," Langmuir, 2005, Volume 21 (13), pp 5685–5688.

[51] Johannes G Smits, Koji Fujimoto and Vladimir F Kleptsyn, "Microelectromechanical flexure PZT actuated optical scanner: static and resonance behavior," Journal of Micromechanics and Microengineering, 2005, Volume 15, Number 6, pp 1285-1293.

[52] HMDS, Transene Company Inc., Danvers Industrial park, 10 Electronics Avenue, Danvers, MA 01923, http://www.transene.com/hexa.html

[53] Spincoater®, Model P6700, Specialty Coating Systems Inc., 7645 Woodland Drive – Indianapolis, IN 46278

[54] J Chung, M Jeng, J E Moon, A T Wu, T Y Chan, P K Ko, C Hu, "Deep-submicrometer MOS device fabrication using a photoresist-ashing technique," Electron Device Letters, IEEE, Apr 1988, Volume: 9 Issue:4, pp 186 – 188.

[55] Haiyang Mao, Wengang Wu, Yulong Zhang, Ge Zhai and Jun Xu, "Fabrication of highcompact nanowires using alternating photoresist ashing and spacer technology," Journal of Micromechanics and Microengineering, August 2010, Volume 20, Issue 8, pp 1 - 6. [56] Hard baking of the photoresist, MicroChemicals, http://www.microchemicals.com/technical\_information/hardbake\_photoresist.pdf.

[57] Munirathna Padmanaban, David Rentkiewicz, SangHo Lee, Chisun Hong, Dongkwan Lee, Dalil Rahman, Raj Sakamuri and Ralph R. Dammel, "Effect of Hard Bake Process on LER," Advances in Resist Technology and Processing XXII, Proceedings of SPIE, 2005, Vol. 5753, pp 862-869.

[58] Richard D. Peters, Kevin Lucas, Jonathan L. Cobb, Colita Parker, Robb McCauley, Kyle Patterson, Monique Ercken, Frieda Van Roey, Nadia Vandenbroeck, and Ivan K. Pollentier, "Line-edge roughness reduction and CD slimming using hardback processing," Proceedings of SPIE - The International Society for Optical Engineering, 2003, Volume 5038 II, pp 1131-1142.

[59] Manish Chandhok, Kent Frasure, E. Steve Putna, Todd R. Younkin, Willy Rachmady, Uday Shah, and Wang Yueh, "Improvement in linewidth roughness by postprocessing", Journal of Vacuum Science & Technology B, 2008, Volume 26, Issue 6, pp 2265 – 2270.

# VITA

Pradeep Pai was born in Mangalore, India, on October 18, 1984. He obtained his Bachelor of Engineering degree in Instrumentation Technology from Visveswaraiah Technological University, Belgaum, India, in Jun 2006. He is currently a candidate for Master of Science in Electrical Engineering at Louisiana State University, Baton Rouge, Louisiana.