# CONSIDERATIONS REGARDING THE USE OF SU-8 PHOTORESIST IN MEMS TECHNIQUE

Maria – Roxana Marinescu [1], Marioara Avram[2], Cătălin Pârvulescu[3], Corneliu Voițincu[4], Vasilica Ţucureanu[5], Alina Matei[6]

[1]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania; roxana.marinescu@imt.ro
[2]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania, marioara.avram@imt.ro
[3]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania, catalin.parvulescu@imt.ro
[4]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania, corneliu.voitincu@imt.ro
[5]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania, vasilica.tucureanu@imt.ro
[6]National Institute for Research and Development in Microtechnologies, IMT-Bucharest, Romania, alina.matei@imt.ro

ABSTRACT: Microelectromechanical systems (MEMS) represent the technology of microscopic devices. In this domain, photolithography is the most common method in order to obtain MEMS structures. The photoresist is the key element for microelectronic and MEMS technology. SU-8 is one kind of negative photoresist, used basically for microfluidics.
This paper presents experiments of SU-8 etching in $O_2/SF_6$ and $Ar/SF_6$ plasma. Monocrystalline silicon wafers with 3" diameter were used, which had native oxide preserved. The SU-8 was spin-coated on the wafers with a thickness of approximately 100 μm. The experiments were made with varying the gas flow rate and the pressure in the reactor. The etching depth and the underetching gave different values every time.
KEY WORDS: Photolithography, photoresist, SU-8, micro-electro-mechanical systems (MEMS), microfluidics.

## 1. INTRODUCTION IN PHOTOLITHOGRAPHY

In the miniaturization field, lithography is a very important technique used to transfer copies of a pattern onto the surface of a solid material (for example - a silicon wafer) [1]. Photolithography is the most used type of lithography. In the Integrated Circuit Industry (including microprocessing of micro-electro-mechanical systems and microfluidic systems) pattern transfer from masks onto thin films is accomplished almost exclusively via photolithography [1], [2]. There are known two classical types of photolithography: through etching and liftoff. The photoresist is the main element used in the technological process of making structures with different shapes and sizes [3].

SU-8 is a commonly used epoxy-based negative photoresist with excellent sensitivity and a very good aspect ratio. The main applications are found in MEMS and in other microsystems, e.g. microfluidic devices. In the last few years, SU-8 has been used in a large range of applications, namely in the fabrication of microchannels for microfluidic and lab-on-a-chip devices and as a structural component in MEMS applications [4].

### 1.1 Types of photoresist

There are 2 types of photoresist: positive and negative. When exposed to radiation, the photosensitive material (photoresist or resist) changes its chemical resistance due to the developed solution. Depending on the way the development is made, there can result two categories of lithographic layers: positive layers that retain the template configuration and negative layers when the radiation-affected areas are more resistant to the development substance (Figure1).

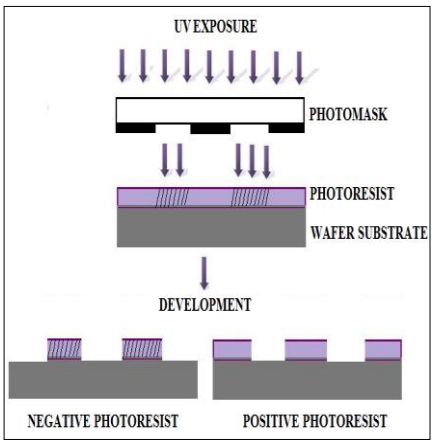


**Figure 1.** Negative and positive photoresist

The photoresist is composed of:

- **a polymer (base resin),** which ensures structural stability and engraving resistance;
- **a sensitizer** (also called the photoactive part);
- **a casting solvent**, which transforms the photoresist from solid form into liquid form [3].

Each one of these three categories has an important role. The polymer changes its structure when it is exposed to electromagnetic radiation. The solvent allows the spin-coating to make uniform layers on a flat substrate and the sensitizers controls the photochemical reactions in the polymerase phase.

Photoresists must meet certain conditions: good adhesion, high sensitivity, high contrast, good etching resistance (wet or dry etching), good resolution, easy processing, high purity, long shelf life, minimal solvent use, low cost, and high glass transition temperature [5].

## 2. SU-8 PHOTORESIST

SU-8 is a negative photoresist and its name derives from the presence of 8 epoxy groups (Figure 2). The SU-8 is thermally and mechanically stable and chemically inert [4], [5].

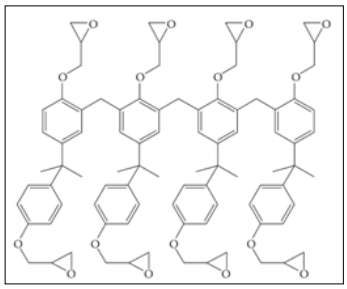

**Figure 2.** The SU-8 molecule [5]

When fully polymerized, it withstands nitric acid, acetone, and even sodium hydroxide (NaOH) at $90^0$C. The high epoxy content promotes strong SU-8 adhesion to many types of substrates and makes the material highly sensitive to UV exposure. From the microfluidics point of view, strong adhesion to the substrate and chemical inertness of the SU-8 are very desirable. The use of SU-8 photoresists allows for the coating of thick layers (up to 500 µm) on a single spin coat, or thicker layers in multiple spin coatings, and high-aspect-ratio structures with nearly vertical side walls [5]. Its photosensitivity allows its patterning by Ultraviolet Rays (UV) photolithography.

Due to its properties, SU-8 is an attractive material for a wide range of applications such as: microoptics, micromachining, packaging and analytical microfluidic applications. SU-8 offers a wide variety of applications in the fabrication of microfluidic systems. This photoresists transparency to visible light makes it compatible with optical detection and thus it helps identification biomolecules. It shows good biocompatibility offering the possibility to develop assay on the surface that allows the specific bending of biomolecules for analytical applications.

SU-8 is widely used to prepare the master molds for replication of PDMS (polydimethylsiloxane) microfluidic channels, but it is also directly used for fabricating microfluidic channels. Currently, SU-8 is available in various forms that cover thicknesses from 1 to 600 µm [4], [5], [6], [7].

### 2.1 Examples where SU-8 is used

#### *2.1.1 SU-8 as a Mold*

SU-8 can be used as a mold to fabricate PDMS microfluidic systems by soft lithography techniques since this material has been vastly employed in microfluidic devices due to its low-cost, transparency, and fast fabrication process. The SU-8 mold present the inverse structure of the purification of biomolecules in aqueous two-phase systems. In Figure 3 we have the SU-8 channels mold with a width of 200 µm spaced by 50 µm and a thickness of 120 µm and the PDMS microstructures resulted [4].

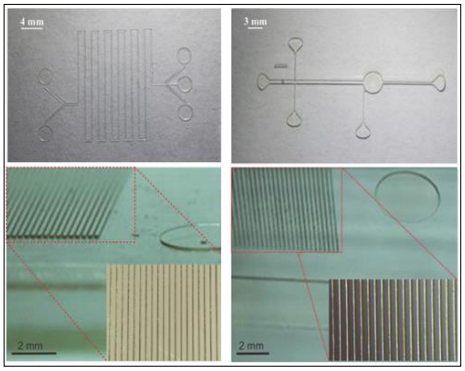


**Figure 3.** Molds made from SU-8 [4]

#### *2.1.2 SU-8 microfluidic devices*

The thick SU-8 films were spin coated on 4" silicon wafers and glass wafers using SU-8.They were soft baked at $65^0$C, exposed to UV light at a dose of 180-220 mJ/cm$^2$, and then post exposed at $95^0$C for 5 min. They were developed to create open microfluidic channels using an SU-8 developer and then dried in a vacuum oven at room temperature for 5h. Each obtained SU-8 microfluidic device was brought into close contact with a $N_2$ plasma-treated PDMS sheet and then treated at $100^0$C for 30 min to drive the reaction and seal the channels. The PDMS sheets (1 mm), were prepared by first casting and gelling via a 1 part curing agent to 10 part resin mixture in a flat mold at room temperature, and then curing them at 110 $^0$C for 3h [7].

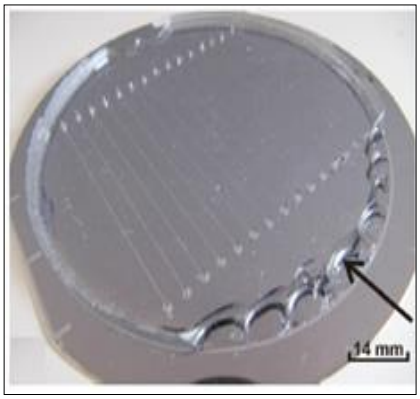


**Figure 4.** SU-8 microfluidic devices [7]

### *2.1.3 SU – 8 microfluidic device for detection and analysis of viruses*

This is a passive microfluidic device made with SU-8 photoresist. It represents a method that uses physical adsorption, bioparticles (specific antibodies) that can be fixed on the SU-8-based microstructures.
The required reagents have been introduced by way of pressure driven flow into the channel. When a contaminated fluid has been introduced through same procedure, viruses that are flowing through the microfluidic system bind with the immobilized biomolecules onto the SU-8 surface [6].

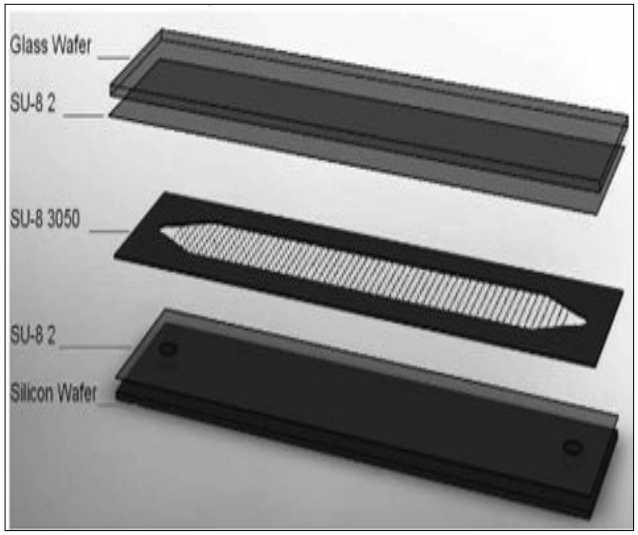


**Figure 5.** Design of the microfluidic device based on SU-8 [6].

## 3. EXPERIMENTAL DATA

We have the technological process for making SU8 molds for microchannel microfluidic platforms. The processing steps are presented in Figure 6:

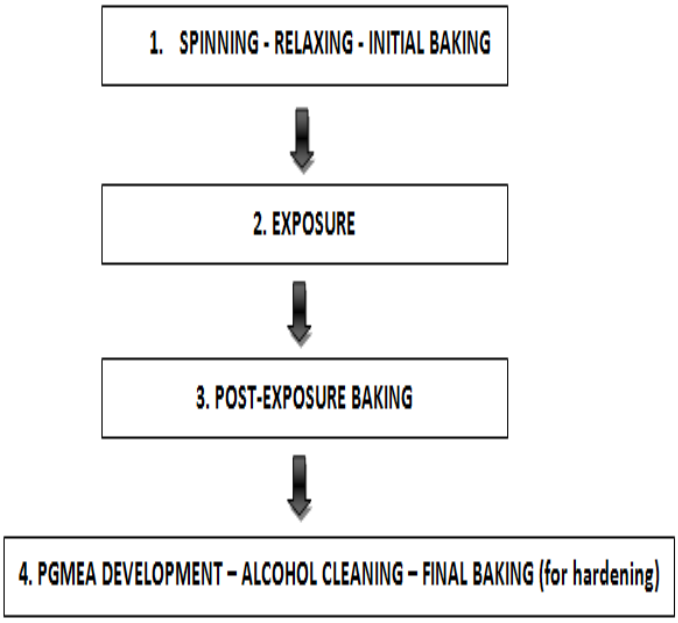


**Figure 6.** The steps for making SU8

### 3.1 Process description

**1.** The silicon wafer is placed on a hotplate (or in an oven) at 130°C for at least 20 minutes in order to evaporate the water absorbed by the substrate. The functionalization of the surface with HMDS (Hexamethyldisilazane) is not recommended.

**2.** After cooling the plate, SU8 is deposited using a spin-coaster for 40 seconds. The speed is chosen according to the layer thickness that we need (thicker or thinner). Acceleration and deceleration should not be more than 100 rpm/s.

**3.** If after spinning air bubbles appear in the SU-8 film, it is recommended to use a sharp, thin and clean tip to remove them. After removing all the bubbles, it is recommended to relax the film for 5 to 15 minutes.

**4.** The initial baking is made in order to evaporate the solvent from the SU-8. We place the SU-8 wafer on a plate which is warm-up very slowly, at $2^0$C/min, up to $65^0$C. The temperature is kept for 5-10 minutes, and then heated up to 95 $^0$C. This temperature is maintained until the solvent is completely evaporated. You can test the film with the tweezers. If it does not leave any marks on the tweezers it means that the solvent has been evaporated. The plate is switched off and the wafer will cool down gradually until it reaches room temperature.

**5.** After the hotplate is cooled, the photoresist is exposed. The exposure dose depends on the thickness of the film deposited.

**6.** After exposure, it is recommended to keep the wafer at room temperature for about 10 minutes to allow the chemical species to migrate.

**7.** A post-exposure baking is made to facilitate the polymerization of the exposed area. This step is critical, so it is recommended to use the hotplate instead of the oven.

**8.** After baking post-exposure, PGMEA is developed. Once the structure appears to be clear, another development in clean solution is recommended. The duration of the second development is 10% of the first development. The second development is done to complete the development of the walls.

**9.** After development, isopropyl alcohol is used for rinsing. If during this operation white streaks appear on the surface of the SU-8 film, it means that the development is not over yet.

**10.** The wafer drying is made outdoors at room temperature.

**11.** Final baking (optional step) is made in order to increase the strength of the SU-8 film. This step is recommended if cracks or areas of poor adhesion appear. Only small cracks can be removed.

All this operations take place inside a clean room, which is a specially constructed enclosed area environmentally controlled with respect to airborne particulates, temperature ($\pm 0.1^0$F), air pressure, humidity, vibration and lighting.

### 3.2 Removing SU-8

Due to its chemical resistance, the SU-8 can't be easily removed. For example, the commercial strip used, hot NMP (1-methyl-2-pyrrolidinone "1-methyl-2-pyrrolidinone"), softens the polymer by converting it into a consistent gel and then dissolves it so that the resulted gel in the intermediate phase can stretch over the structures until it is completely removed, contaminating them. The solution based on sulfuric acid and hydrogen peroxide ($H_2SO_4$: $H_2O_2$ (3: 1)) is much better for removing the SU-8, but it can attack the substrate, especially if it is a metal.

### 3.3 Experiments of SU-8 dry-etching

Experiments for SU-8 etching took place in $O_2/SF_6$ and $Ar/SF_6$ plasma. Oxygen plasma easily removes organic materials in general and photoresists in particular. It has been shown that the etching rates of SU-8 increase considerably if small amounts of fluorinated gases, such as $SF_6$, $CF_4$, $C_4F_8$, are added to the oxygen plasma. In our experiments we used $SF_6$ because SU-8 has carbon atoms in its molecule and using a gas that also has carbon in the molecule, the plasma will be rich in carbon and fluorine radicals, that can't be removed from the vacuum pump and will be deposited on the wafer surface, giving rise to thin films similar with teflon properties, inhibiting the SU-8 etching. The rate of SU-8 etching increases significantly, even if the amounts of $SF_6$ introduced into the oxygen plasma are very small, because F generates reactive sites in the main chain of the polymer so that small amounts of $SF_6$ lead to an increase in the number of oxygen atoms in the plasma.

### 3.4 Sample preparation and experiments

All the experiments were made on 3" diameter silicon wafers, which had native oxide preserved. SU-8 was spin-coated on wafers with a thickness of approximately 100μm. The standard procedures were followed:

**1.** Annealing on hotplates at 90°C for 30 minutes;

**2.** Exposure to UV light at 10mW $cm^2$ for 10';

**3.** SU-8 polymerization on hotplate at 90°C for 30';

**4.** Thermal treatment of SU-8 in the oven at $170^0$C for 60';

**5.** Cool down slowly in the closed oven to until it reaches room temperature.

**6.** Then, the wafer is keeping at room temperature for 10' to allow migration of chemical species;

**7.** Next, post-exposure annealing is done to facilitate the polymerization of the exposed area. This step is critical, so it is not recommended to use the oven but use the hotplate;

**8.** Development is made, and if the structure is not clear, further development in clean solution is recommended. The duration of the second development is 10% during the first development. The second development is done to complete the development of the walls;

**9.** After development the wafer is cleaning in isopropyl alcohol and is drying at room temperature.

Cu (200 nm) and cathodic Cr (20 nm) were sprayed over SU-8 for adhesion. Then a photoresist was deposited in which the mask configuration was defined, and the mask was etched Cu and Cr.

A series of SU-8 dry etching experiments were performed using a metal mask using the SENTECH SI 220 reactive ionization etching facility. The direct power was maintained at 200 W.

Experiments varying the gas flow rate were made ($O_2$: 20, 40, 60, 80 sccm; $SF_6$: 0, 2, 4, 6sccm; Ar: 0, 10, 20, 30sccm) and the pressure in the reactor: 10, 20, 30 Pa (75, 150, 225 mTorr). After 4 minutes of etching, were removed the photoresist residues and then Cu and Cr (masking layers) and the etching depth was measured using a profilometer and the underetching using SEM.

### 3.5 Experimental results

***Table 1****: Depth of etching and underetching of SU8 in oxygen plasma, 200W, 10Pa*

| $O_2$ | 20sccm | 40sccm | 60sccm | 80sccm |
|---|---|---|---|---|
| Depth (μm) | 5 | 4 | 5.5 | 5 |
| Underetching (μm) | 2 | 2.5 | 2 | 2 |

***Table 2**: Depth of etching and underetching of SU8 in oxygen plasma, 200W, 20Pa*

| $O_2$ | 20sccm | 40sccm | 60sccm | 80sccm |
|---|---|---|---|---|
| Depth (μm) | 5.2 | 4.2 | 6 | 5.5 |
| Underetching (μm) | 2.1 | 3 | 2.5 | 3 |

***Table 3**: Depth of etching and underetching of SU8 in oxygen plasma, 200W, 30Pa*

| $O_2$ | 20sccm | 40sccm | 60sccm | 80sccm |
|---|---|---|---|---|
| Depth (μm) | 6 | 5 | 7.5 | 6.5 |
| Underetching (μm) | 2.5 | 3.3 | 2.5 | 3 |

***Table 4**: Depth of etching and underetching of SU8 in $O_2$ plasma (60sccm)/$SF_6$, 200W, 10Pa*

| $SF_6$ | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 5.5 | 7.5 | 9 | 8 |
| Underetching (μm) | 2 | 3 | 2.5 | 2 |

***Table 5**: Depth of etching and underetching of SU8 in $O_2$ plasma (60sccm)/$SF_6$, 200W, 20Pa*

| $SF_6$ | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 6 | 8 | 9.5 | 8.5 |
| Underetching (μm) | 2.5 | 3.5 | 3 | 2.5 |

***Table 6**: Depth of etching and over-etching of SU8 in $O_2$ plasma (60sccm)/$SF_6$, 200W, 30Pa*

| $SF_6$ | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 7.5 | 8.5 | 10 | 8.5 |
| Underetching (μm) | 2.5 | 3.25 | 3.5 | 2.5 |

***Table 7**: Depth of etching and underetching of SU8 in $O_2$ plasma (60sccm)/$SF_6$(4sccm)/Ar, 200W, 10Pa*

| Ar | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 9 | 7.5 | 6 | 5 |
| Underetching (μm) | 2.5 | 2.5 | 2 | 1 |

***Table 8**: Depth of etching and underetching of SU8 in $O_2$ plasma (60sccm)/$SF_6$(4sccm)/Ar, 200W, 20Pa*

| Ar | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 9.5 | 8 | 7 | 6 |
| Underetching (μm) | 3 | 2.5 | 1.5 | 1 |

***Table 9**: Depth of etching and underetching of SU8 in $O_2$ plasma (60sccm)/$SF_6$(4sccm)/Ar, 200W, 30Pa*

| Ar | 0sccm | 2sccm | 4sccm | 6sccm |
|---|---|---|---|---|
| Depth (μm) | 7.5 | 6 | 4.5 | 3.5 |
| Underetching (μm) | 2.5 | 2.1 | 1.2 | 1 |

## 4. CONCLUSIONS AND ACKNOWLEDGEMENTS

Analyzing the data from the 9 tables, we can see that the optimal parameters for high-speed SU-8 etching in Reactive Ion Etching (RIE), slightly above 2μm/min, are: $O_2$ = 60 sccm, $SF_6$ = 4 sccm, Ar = 0 sccm and the reactor pressure: 20 Pa. If Ar is added to the $O_2$+$SF_6$ plasma, we observe that the rate of etching decreases with the Ar flow, while the underetching decreases. In conclusion, argon significantly improves etching anisotropy, but drastically decreases the etching rate.

This work was supported by a grant of the Ministry of Scientific Research, RDI Program for Space Technology and Advanced Research—Star, project number 639/2017. Also, this work was supported by UEFISCDI in the partnership framework: PN-III-P2-2.1-PTE-2016-0145 (project no. 46PTE/2016), PN-III-P1-1.2-PCCDI-2017-0214 (project no. 3PCCDI/2018). Authors would like to acknowledge and to thank Carmen Iorga, Tamara Dobre and Elene Gheorghe for cooperation.

## 5. REFERENCES

1. Madou Marc J, Fundamentals of Microfabrication and Nanotechnology, Volume II, Manufacturing Techniques for Microfabrication and Nanotechnology, Third Edition, CRC Press, (2011).
2. *Specialized Materials Meet Critical Packaging Needs in MEMS Devices, Master Bond Tech Spotlight,* white paper, (2008).
3. Oltean, I.D., *Tehnologie Electronica, Tehnologia Dispozitivelor Semiconductoare si a Circuitelor Integrat,* Ed. Matrixrom, Bucuresti, (2004).
4. Vania C. Pinto, Paulo J. Sousa, Vanessa F. Cardoso, Graca Minas; *Optimized SU-8 processing for Low-Cost Microstructures Fabrication without Cleanroom Facilities, Micromachines,* (2014).
5. Sushanta K. Mitra, Suman Chakraborty, *Microfluidics and Nanofluidics Handbook, Chapter: SU-8 Photolithography and Its Impact on Microfluidics* pp 231-268, CRC Press, (2011).
6. Claudia Garcia, Anna Schomacker, Ingo Klarholz, Carsten Harms, Walter Lang, *Fabrication of Microfluidic Devices using SU-8 for Detection and Analysis of Viruses,* SENSOR+TEST Conference (2009) - SENSOR Proceedings I.
7. Zhiyi Zhang, Ping Zhao, Gaozhi Xiao, BenjaminR. Watts, Changqing Xu., *Saeling SU-8 microfluidic channels using PDMS,* Biomicrofluidics 5, 046503(2011).