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Fabrication of micro-polarizer array with polymer thin film

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ABSTRACT

In this project, significant advancements have been made in creating an array of micro-polarizers using a polymer thin film in order to extract various polarization parameters about the imaged environment. The array of micro-polarizers is to be integrated with custom-made VLSI image sensor in order to create a complete low power real-time bio-inspired polarization sensitive imaging sensor. Two different photoresists, i.e. positive and negative photoresist, are used for patterning the polarization thin-film. The advantages and disadvantages of both procedures are analyzed in details. The isotropic properties of the etching procedure of the thin-film are compared between oxygen plasma and reactive ion etching. The optimum isotropic etching is outlined as a function of temperature, pressure and gases flow. Finally, we have summarized the complete procedure for creating a micro-polarize array with sub-μm precision.

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1. INTRODUCTION

Two characteristics of visible light: wavelength and intensity can be easily detected by unaided human eyes and present as color and brightness. The third important characteristic of light is its polarization, which our human eyes are not able to detect directly. However, the ability to see light polarization has been found in many animals for vital purposes such as navigation and detections of both preys and predators. By definition, visible lights (beams of electromagnetic radiation) have wave properties, in which possesses three-dimensional vectors, thus the presences of these vectors essentially cause the phenomenon of polarization. In most cases, the direction of polarization is the also the direction of electrical field. There are different ways a light source can be polarized, i.e. by double refraction, reflection, scattering, etc, where different vectors are absorbed and preserved.

The electrical field in unpolarized waves can be decomposed into two perpendicular linear components. The purpose for a single polarizer is to block one of these perpendicular components. Consequently, two of such polarizers position perpendicular to each other should block 100% of the light source. Any angle other than the perpendicular (90 degrees) and parallel (0 degree) ones let pass a certain amount of original light, from minimum in perpendicular up to the maximum value in parallel configuration. All four Stokes' parameters ($S_0 \sim S_3$) in Equations (1) can be used to describe full polarization information in an object.

$$
\begin{aligned}
S_0 &= I_t, \\
S_1 &= 2I(0^\circ, 0) - I_t, \\
S_2 &= 2I(45^\circ, 0) - I_t, \\
S_3 &= I_t - 2I(45^\circ, \pi/2).
\end{aligned}
\qquad (1)
$$

Equations (1) show that S_0 to S_2 can be obtained by measuring intensity of linear polarization of 0 and 45 degrees ($I(0^\circ, 0)$; $I(45^\circ, 0)$) without any phase change along with the total intensity (I_t). [1] Therefore, it's meaningful to fabricate a micro-polarizer array which can collect such information: I_t, $I(0^\circ, 0)$, $I(45^\circ, 0)$. One main future use for the array that this experiment will focus on is to be part of an overall sensor system: it will be placed on top of a custom made imaging sensor. Unlike other polarization detection systems available today, this particular system acts as a single unit, thus be able to display object's polarization, along with the conventional colors and brightness in real time settings. In addition, the small size of our array and chip (3mm by 3 mm in length and width) has low power consumption, which adds another advantage to the system.

Upon completion of this sensor system, it will be utilized in detection in light scattered environments related fields.

2. CHOOSING POLARIZER

There are two ways of obtaining polarizer in this experiment. One is to make a polarizer using procedures that are very similar to LCD monitor production, which is to align liquid molecules on rubbed surfaces. In many cases, a silica glass substrate is repeatedly rubbed toward one direction by a roller wrapped with velvet clothes. After the rubbing process is completed, a thin layer of polarizing material is then spin-coated onto the rubbed surface by an electric spinner. Molecules of the polarizing material would align themselves along the direction rubbing, thus making up the direction of light absorption. The biggest advantage for using such technique is that the thickness of polarizer can be easily controlled by spin speed and concentration of the polarizing material. Less than 1 μm layer of polarizer can be easily attained. Despite the fact that as the thickness varies, there is a trade off between the transmission coefficient and the polarization extinction ratio.

Although the expected data looks very promising, the actual rubbing process is a nontrivial task. Such process has become extremely refined over years of experience from multi-million dollars industries: high standards of rubbing pressure and uniformities are vital to the qualities of polarizer; close monitoring on the level of roller degeneration and dusts created during the process is absolutely required [2]. Therefore, with numerous factors that could affect the performances of polarizer, good polarization data and characteristics can not be guaranteed. Each individual polarizer made from this process will need to go through testing before proceeding to be used. Many sophisticated and expensive machineries have been designed just for the rubbing process, which are not practical to obtain in this experiment. However, simple rubbing process without precision involving dichroic dye solution (polarizing material) called *POLACOAT* is described below:

1) A cleaned glass substrate surface is rubbed repeatedly with a high speed drilling bits wrapped with cotton clothes (with no numerical measurements on pressure and uniformity).
2) The dichroic dye (4%) is then spin-coated on at 2000 rpm for 2 minutes
3) The surface is then heated to 140 Celsius for 14 minutes to evaporate the solvent.

Small amount of polarization effects on a layer of less than one micrometer are observed in the area that has been rubbed. This technique is proven that it can be effective if more sophisticated equipments are to be used in the process.

To get around these shortcomings, some commercially available polarizers are being considered. The advantage here is that, while very cheap and available to obtain, most of them have great proven data for the interests of this experiment. *TECH SPEC™ Linear Polarizing Laminated Film* is chosen for this experiment, it is a brand of polymer film that has shown great extinction ratio in the visible light spectrum.

3. TESTING POLARIZER

The most important characteristic to describe how polarizer performs is its extinction ratio. In ideal cases, one single polarizer should block 50% of the light intensity from an original un-polarized light source, while if two identical polarizers are to be placed at 90 degrees from each other, this resulting position should block 100% of the light. Base on these scenarios, the qualities of the actual polarizer depend on how close it can perform in such way. To measure this, a variable called *extinction ratio*, shown in Equation (2), is introduced. It's the ratio of amount of light that passes through with only one polarizer over when two polarizers that are crossed (90 degrees from each other):

$$\textit{Extinction Ratio} = \frac{\text{light intensity through one polarizer}}{\text{light intensity through crossed polarizers}} \qquad (2)$$

If the ratio unit is converted to units of dB in ideal case, it's obvious to see that the ratio will become infinite. In practical real life case, the result becomes a finite number, which the higher dB it possesses, the better the polarizer performs.

To validate the fact that the purchased polarizer is indeed able to match up data provided by manufacturer (figure 1) [3], a testing process goes as follow: light with different wavelengths are shining through the polarizer to measure the intensity. One narrow-banded green and one red LED are used as light sources. Either a single polarizer or two crossed polarizers are placed between the LED and the optical power meter. From the graph provided by manufacturer, it's clear to observe that the crossed transmission rate is significantly greater for red LED (700 nm) than to green LED (600 nm) due to higher energy emitted from red light. As result, from Equation (2), extinction ratio for the green LED is expected to be higher than red. Measured data from both LEDs are shown in table 1.

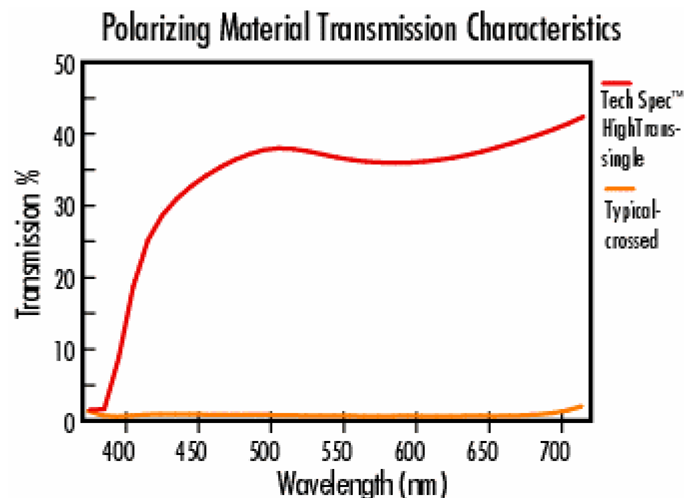

Figure 1: Extinction ratio data of the polarizer provided by TECH SPEC™

Table 1: This table shows the amount of light that has been permitted to pass through either single polarizer or cross polarizer (90 degree from each other). The Green LED shows a better extinction ratio than Red LED due to lower energy, which corresponds to the Figure 1.

	No Polarizer	Single Polarizer	Cross Polarizer	Extinction Ratio
Red LED (700 nm)	**480 mW/cm2**	**190 mW/cm2**	**2 mW/cm2**	**40dB**
Green LED (600 nm)	**480 mW/cm2**	**180 mW/cm2**	**0.2 mW/cm2**	**60dB**

4. POLARIZING THIN FILM STRUCTURE

The basic structure for *TECH SPEC™ Linear Polarizing Laminated Film* [4]has 5 layers: two outermost layers are protective plastic lamination layers, which can be easily peeled off. In the middle consists of a layer of doped Polyvinyl Alcohol (PVA) sandwiched between two Cellulose Acetate Butyrate (CAB) layers. PVA layer is the actual polarizing layer, and thus the main focus of this experiment. The CAB layers are hard and transparent. Their purpose is to give the polarizer a sturdy structure while attenuating a very small amount of light rays going through them. To expose the actual PVA layer, either one or both layers of CAB need to be removed.

100% pure acetone solution is used for the removal of CAB. The solution has no effect on PVA, and thus makes it an ideal chemical for CAB wet removal. When a sample film submerged into acetone solution, within 20 minutes, CAB starts to soften and peeling up from edges. After almost an hour, CAB is in gel-like state, and it turns into white solid when expose to air and water. Putting it back to acetone solution, the white residue will disappear and become gel-state again. If nothing is done to physically remove the CAB, after a period of 2-3 hours, the PVA is completely detached away from CAB acetone. However, due to small thickness of the PVA layer, it's extremely difficult to keep it undamaged.

One approach to solve film's fragility is to remove only one layer of CAB, while CAB from the other side is still thick enough for support. To prevent the CAB layer from being attacked by acetone, a brand of thermally and chemically resistant tape is used to tape one side of polarizer onto a glass substrate with the other side ready to be removed by acetone. To speed up the CAB removal process, as soon as CAB layer becomes gel-like state within an hour, lightly and gently rub the surface along with DI water rinsing. To avoid scratches on the film, most top portion of CAB is to be removed with physical rubbing, the rest is washed away with repeated DI water rinsing.

With a sample that contains one PVA layer and one CAB layer sample is obtained, a simple thickness measurement by a microscope is performed. It has been found that

most PVA appear dark (Shown in figure 2). One problem arises that originally PVA is expected to be rather uniform in thickness. In reality, the layer appears to have thickness ranging from 10 to 25 µm. Variation in thickness can be explained by the way the polarizer is made and manufactured: special machines are used to stretch the polymer film to very thin. As the result, due to different level of stress applying across the surface, the thickness varies. Due to this result, the polarizing capability changes from point to point. This variable presents a major obstacle in later part of this paper on how long the sample should be put in the dry etching process.

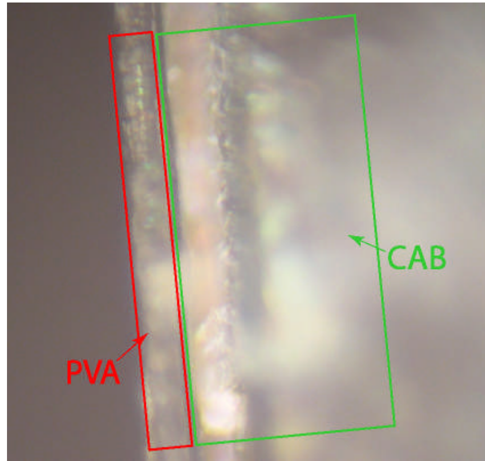

Figure 2: TECH SPEC™ Linear Polarizing Laminated Film when one layer of CAB has been stripped, it shows the clear interface between PVA and CAB layers.

5. POSITIVE PHOTORESIST MASKING

Patterns on the microscopic level need to be formed on actual PVA layer as the final product. The idea is to initially spin coat a layer of photoresist on top of PVA. The desired pattern is then put on photoresist through a conventional process called *photolithography*. This particular pattern is then transformed onto the PVA layer through designated etching procedures.

Positive photoresist is first being put to test first. It's more commonly used in semiconductor fabrication industry. *Shipley S1813* positive photoresist appears to possess a red-orange color, and it's slightly more viscous than regular water. To guarantee the uniformity of the photoresist layer, it will be spin-coated by an electric spinner. The desired thickness can be controlled by the spin speed.

An UV mask aligner is used to pattern the photoresist. The basic principle is that, when exposed under UV light for a certain period of time, energy exerted by UV would be enough to change properties of positive photoresist so to make it soluble to the photoresist developer [5]. The amount of time to be exposed is calculated by Equation (3):

$$\text{Power (wavelength)} = \text{Required photoresist energy} \times \text{Time} \qquad (3)$$

The entire photolithography process is described as follows:

1. Depending on the desired thickness, photoresist will be spin-coated with a different speed. (for 1.5 μm thickness of photoresist, spin 4000 rpm for 30 seconds, faster spin speed means thinner photoresist)
2. Undergo soft baking process to evaporate the solvent. (125 Celsius for 2 minutes)
3. UV Photolithography (with patterned masks), expose under the time that is necessary to change the exposed photoresist's structure completely with a *Kasper System™ 2001 Mask Aligner*.
4. Develop in the photoresist developer for certain period of time, depending on the expected thickness. (1 minute)
5. Hard bake for a period of time to harden the photoresist.

Figure 3 shows the positive photoresist UV photolithography process:

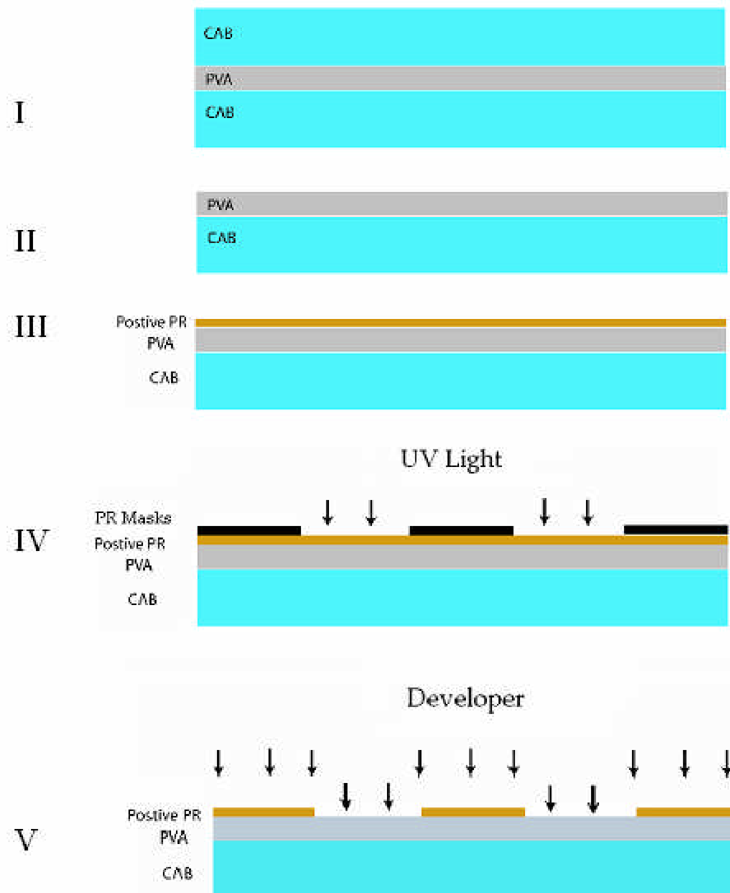

Figure 3: I: the original polymer film. II: one side of CAB is stripped by acetone. III: a layer of positive photoresist is spin-coated on top of PVA. IV: UV light shines on the masked photoresist. V: After development, the part of positive photoresist that has been exposed to UV has been dissolved.

The amount of time spent on hard and soft bake depends on the photoresist material and its thickness. The way to test relation between the spin speed and the

thickness of the photoresist is to go through the UV Photolithography process described above, then use *Tencor Instruments ™ Profimeter* to scan through the pattern to measure thickness.

5.1 Oxygen plasma etching and positive photoresist

Once a photo resist pattern is put on top of the PVA, to transfer the pattern, an etching technique is needed to etch both photo resist and exposed PVA simultaneously in order to achieve the purpose of pattern transferring. In best possible scenario, this technique is able to etch vertically in an anisotropic fashion. With the thicknesses of both photoresist and PVA are known, by the end of process, it's expected that both exposed photoresist and PVA should disappear at the same time, while the PVA underneath the photoresist remains to retain the original pattern from photoresist. The process is shown in figure 4:

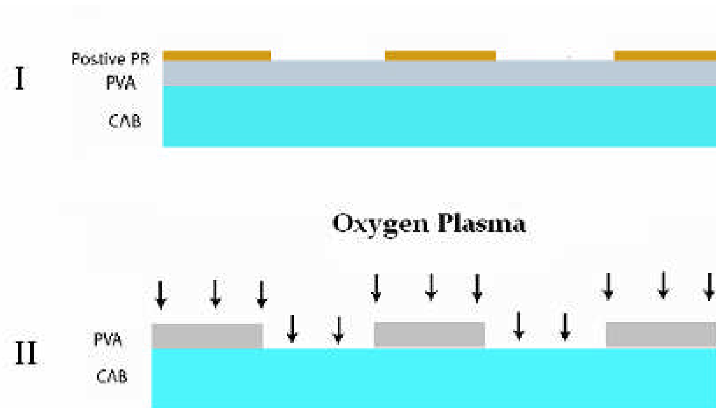

Figure 4: I: The sample after UV Photolithography. II: The oxygen plasma etching process is suppose to etch away the thickness of photoresist mask and the thickness of exposed

One commonly used dry etching method is the oxygen plasma etching (performed by *Technics PlasmaEtch™ II Oxygen Plasma instrument*). Gases such as oxygen are heated to extremely high temperature to their plasma forms. The electrons released in the process will give target surface the bombardment it needs to be etched away. In reality, it's more relevant to look at the etching rate ratio for both photoresist and PVA layers rather than the actual etching rates.

Among all the important factors that can affect the etching rate ratio, the most important one is the O_2 and CF_4 gas flow ratio [6]. Other factors such as amount of etching time and amount of power all have a linear relation with the etching rate, and therefore easy to predict. Figure 4 shows the etching rate for both PVA and photoresist layers, with power and time being fixed. One horizontal axis, O_2, CF_4 gas flows are in increments of 0.5 sccm.

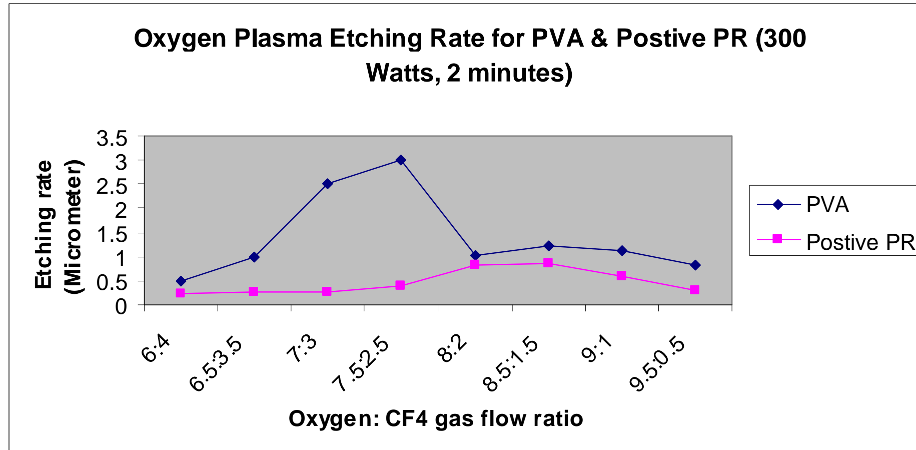

Figure 5: It's obvious to notice that the etching ratio (PVA over Positive PR) is at greatest when O_2 : CF_4 gas flow ratio is at 7.5:2.5.

As shown in figure 5, even if the right gas flow ratio to achieve the optimum etching rate ratio is attained, the maximum etching ratio (PVA over Positive photoresist) is approximately 7:1, much lower than the average thickness ratio(PVA over Photoresist) of 10:1. This indicates that at some point during the etching process, the protective photoresist layer will be long gone and PVA underneath will start to be etched away before the exposed PVA is etched away completely. As this paper emphasized earlier, changes in the thickness of the actual PVA can cause its polarizing performance to be altered, and thus undesirable in this experiment.

Besides the etching ratio, there is another important etching aspect that will directly affect how small of pattern's dimensions that can created, and it's called the isotropic effects of etching. Ideally, when both exposed PVA and photoresist pattern are etched away, an anisotropic etching fashion is being assumed. However, in real life, absolute anisotropic etching can hardly be achieved. It's only possible to find a technique with relatively less isotropic effects. In this case, with oxygen plasma etching process, it has been measured that for each PVA unit that has been etched vertically, the gas plasma also etches away half unit horizontally (see Figure 6), thus we conclude the vertical to horizontal etching ratio to be 2:1. With a simple calculation, if the PVA is at its thickest: 30 µm, by the time oxygen plasma completely etches away the exposed PVA, it will also etch approximately 7.5 µm from each side of the PVA that's under the photoresist patterns, with the desired PVA pattern to be in units of 10 µm, it will be impossible to create such patterns. A better technique is needed.

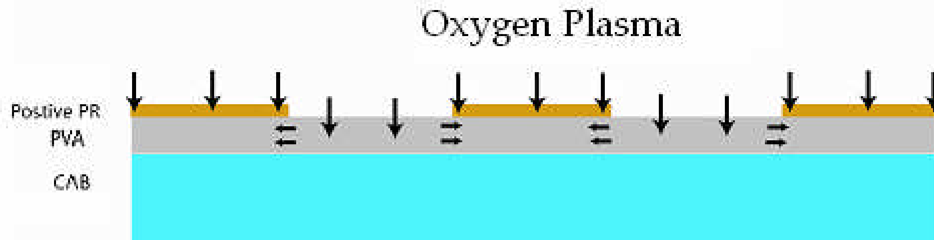

Figure 6: This is the actual Oxygen Plasma etching process. Ideally, it should only be etching vertically, but it does have the isotropic effects of etching all directions.

5.2 Wet etching

During the photoresist development process, once the developer completely dissolves the UV exposed positive photoresist, it gradually attacks the PVA layer underneath due to the similar structure shared by both positive photoresist and PVA. With this property, in theory, the developer can be used for wet etching. However, positive photoresist is etched away in a much more rapid rate than to PVA. The vertical to horizontal etching ratio (isotropic effects) for PVA could reach as low as 1:1, thus making it an undesirable etching technique also.

5.3 Problems with Positive photoresist and oxygen plasma etching

As Several shortcomings have been observed from using positive photoresist masking:

1. Because of the similar structure from both *Shipley S1813* and PVA layer in the polymer film, the developer will also etches away PVA underneath once the top photoresist layer is depleted. Due to the severe isotropic etching effect (as much as 1:1) from the positive photoresist developer, it is not desirable to use positive photoresist as the protective layer on top of PVA. Under-exposing the photoresist is tested to retain a layer of protection photoresist layer. However, the under exposed surface has high non-uniformity and roughness, and thus undesirable. See figure 7.

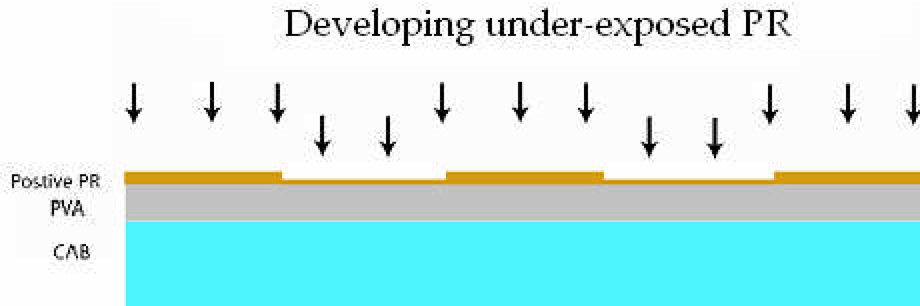

Figure7: For under-exposed PR, it would not be completely dissolved by the solution, and therefore leaves a thin protection layer, however, since this is the wet etching process, that layer is non-uniform.

2. The thickness ratio of PVA to positive photoresist could reach as high as 10:1, however, with the optimal O_2 and CF_4 gas flow ratio at 7.5 to 2.5, the oxygen plasma etching could only attain as high as 7:1 etching rate, which is much lower than the thickness ratio. In order to decrease PVA to positive photoresist thickness ratio, photoresist thickness has to increase. However, spin multiple layers of positive photoresist to increase the thickness is not possible because *Shipley S1813* is not designed for multiple spins.
3. With oxygen plasma dry etching, it shows a considerate amount of isotropic effects: as high as 2:1, which presents a problem in the compensation process of aligner mask making.

With these ideas in mind, a new type of photoresist that can be uniformly spun onto a surface with a larger thickness is needed since the thickness of PVA is fixed can't be uniformly decreases. It should also be made by much different (if not completely different) material from PVA, so that when it's undergoing development, PVA isn't to be affected at the same time. One final important improvement is that a better anisotropic etching technique is needed.

The following sections will describe how different approaches take place to solve these problems one by one.

6. NEGATIVE PHOTORESIST MASKING

Negative photoresist has opposite properties as the positive photoresist when exposed under UV light. Instead of exposed region being washed away by the developer like positive photoresist, it stays intact and the unexposed negative photoresist region is being washed away.

SU-8 is a series of negative photoresist that can be spin-coated on with a large range of thickness: from as thin as 1 μm to as thick as 1 mm.(depending on serial code of the photoresist) All products from this series have very transparent appearances and the viscosity increases with its serial number (the more viscous product is used to obtain a

thicker photoresist layer). Because *SU-8* is made up by completely different material as PVA, its developer has little or no effect on PVA.

One important property to notice when applying *SU-8* onto a surface is that the photoresist is highly hydrophobic. Even the humidity in the air condensed onto the PVA surface will decrease *SU-8*'s adhesion onto PVA dramatically. Therefore, in addition to heat the PVA before applying the photoresist, an extra thin layer of adhesion promoter will be put on top of PVA. It has been found that *AP300* series chemical solutions can be used. When spun on top of PVA, it reacted with moist to form a protective layer: TiO_2. The adhesion between TiO_2 and *SU-8* photoresist becomes much stronger.

The rest of negative photoresist masking process is similar to positive except for a couple of extra steps:

1. Spin coat the adhesion promoter
2. Immediately apply and spin coat desirable thickness of *SU-8* within half of a minute after adhesion promoter *AP300*.
3. Soft bake the photoresist to evaporate the solvent so to achieve higher density for the layer. It has two levels of temperature of 75 Celsius and 115 Celsius.
4. Expose through UV-photolithography with calculated time, which is based on the amount of energy needed to expose the photoresist completely
5. Post-expose bake is needed to cross-link the exposed portions selectively, it also has both 75 and 115 Celsius temperature levels.
6. Develop with MicroChem *SU-8* Developer

6.1 Negative photoresist gradients

For one particular trial in the experiment, after AP300 adhesion promoter is spun onto PVA layer (2000 rpm for 30 seconds), immediately afterward, a layer of 40 µm *SU-8* 2015 is applied on top of promoter (500 rpm initial spin speed for 10 seconds, then 10 seconds of constant acceleration up to 2000 rpm followed by 40 seconds of 2000 rpm). Soft bake process is then followed (1 minute at 75 Celsius, and 5 minutes at 115 Celsius), photoresist needs to be checked whether it's completely solidified. The sample is then going through UV photolithography (22 seconds at 11 ms per cm^2) with a mask pattern of 96 µms wide. A hard bake process is also added after the photolithography to cross-link the photoresist (1 minute at 75 Celsius, and 5 minutes at 115 Celsius). Finally, the pattern is formed when the sample is developed in the solution for 3 minutes. The vertical cross section of the pattern appeared to be a trapezoid shape instead of rectangle, shown in figure 8.

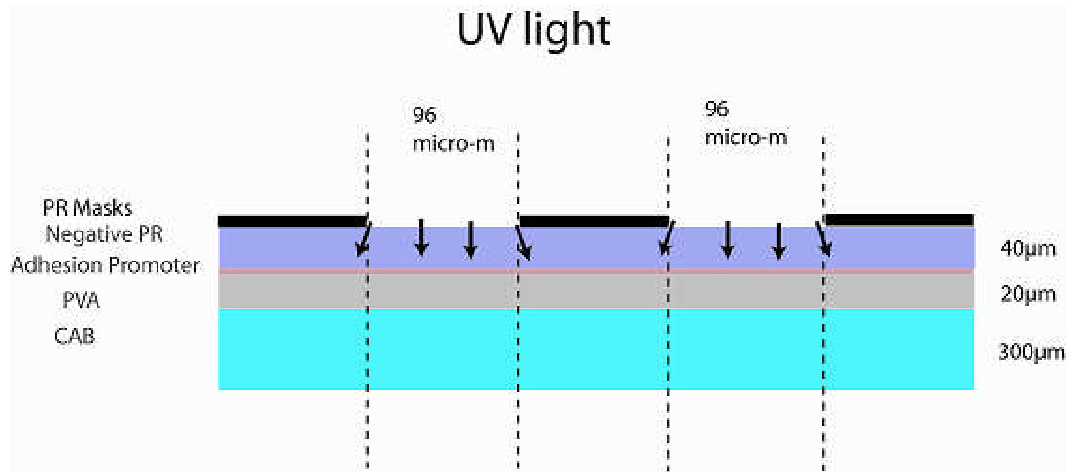

Figure 8: at the edges of the PR masks, the UV light rate does not travel straight as expected, instead, it gets refracted and penetrating PR through an angle.

Figure 9 shows the photoresist pattern formed after the UV-photolithography. The extra area UV light has covered is called the *gradient of photoresist.* In this case, due the 40 µms of thickness, *SU-8* layer has a gradient of 2 µm on each side. This is a significant length to overcome, considering the desirable size for each unit is only 10 µm. However, with a simple geometric calculation, if *SU-8* thickness can be controlled, so are gradients. Table 2 shows the size of gradients comparing to *SU-8* thickness.

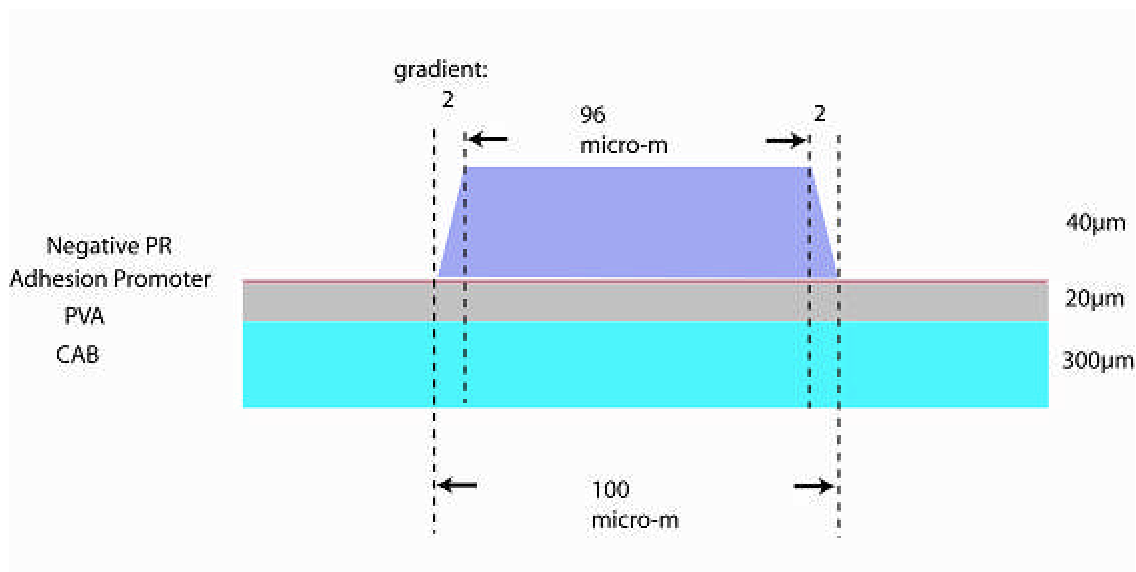

Figure 9: The gradients are shown between the dotted lines.

Table 2: it shows that when *SU-8* is around 1 μm, the gradients are negligible, which is the case when we used the positive photoresist.

SU-8 (μm)	Gradients (μm)
1	0.05
15	0.75
40	2

6.2 RIE (Reactive Ion Etching) process

It is mentioned earlier that an improved etching process with less isotropic effects is needed. One such effective etching process is called Reactive Ion Etching (Performed by *Plasma Tech. Plasmalab μp*). In addition to the mixture of O_2 and CF_4, Argon gas is added, which will provide more mechanical etching power vertically. With the optimal gas flow ratio of 7.5:2.5:2.88 (O_2 : CF_4 : Ar), the PVA over photoresist etching rate can be achieved as high as 3:1. Since *SU-8* photoresist can range from less than 5 μm up to 1 mm (in case of *SU-8 2015*, it can attain from 15 to 38 μm with 3000 rpm to 1000 rpm), *SU-8* can well accommodate the 3:1 etching ratio. The targeted photoresist will be around 15 μm ~ 20 μm to go with maximum possible PVA thickness of 40 ~ 60 μm, matching closely with etching rate of 3:1. In addition, for RIE process, the vertical VS horizontal etching ratio (isotropic effects) for PVA can reach as high as 10:1, which is a significant improvement over oxygen plasma etching. One undesirable properties of RIE is that the procedures blur both PVA and CAB surfaces, which will present problems in optical domain of the system operation.

7. CONCLUSION AND RECOMMENDATION

It has been found that *Shipley S1813* is not able to achieve the desired specifications as a mask layer for this experiment. With the thickness flexibility of *SU-8* combined with relatively good anisotropic etching from RIE process, all the comparisons and calculations are drawing the conclusion that it's feasible to fabricate polarizing array with each individual unit size at 10 μm with these procedures. The future plan for the continuation of this experiment will include:

1) Explore different ways to treat the blurry surface left by RIE etching process.
2) Use a test mask that has features with desired dimensions (close to 10 μm) and go through the fabrication procedures described in this report. Scan the entire structure profile of the test product with Scanning Electronic Microscope.
3) Two-layers configuration so to collect both $I(0^\circ, 0)$; $I(45^\circ, 0)$.

8. ACKNOWLEDGEMENT

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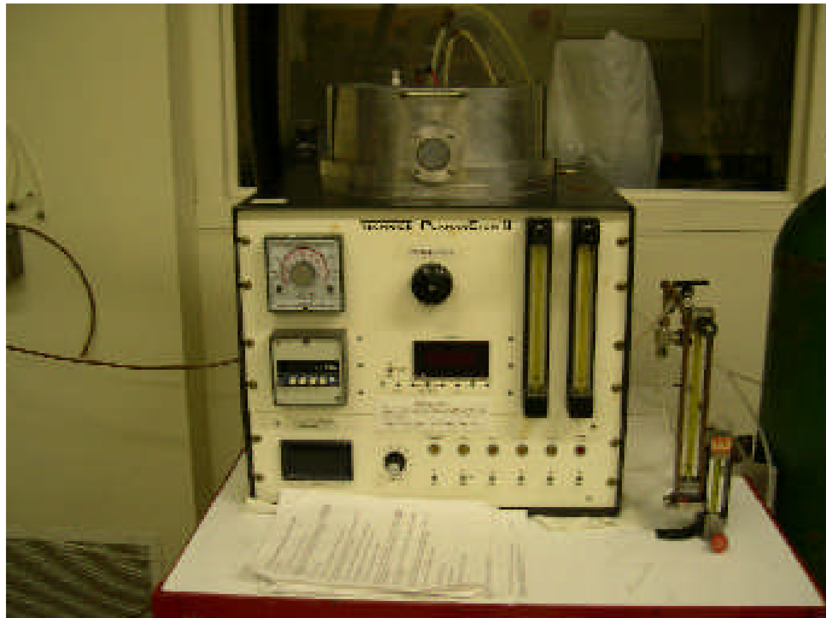
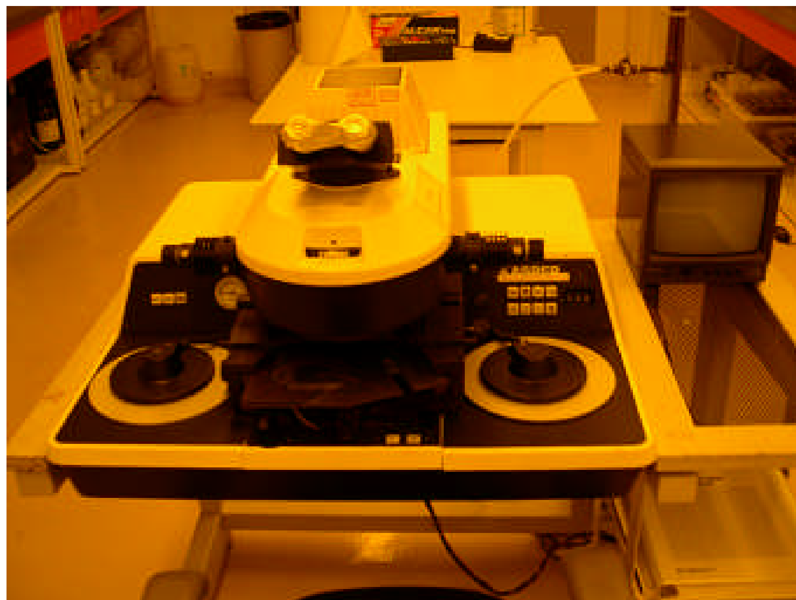
APPENDIX A

INSTRUMENTS

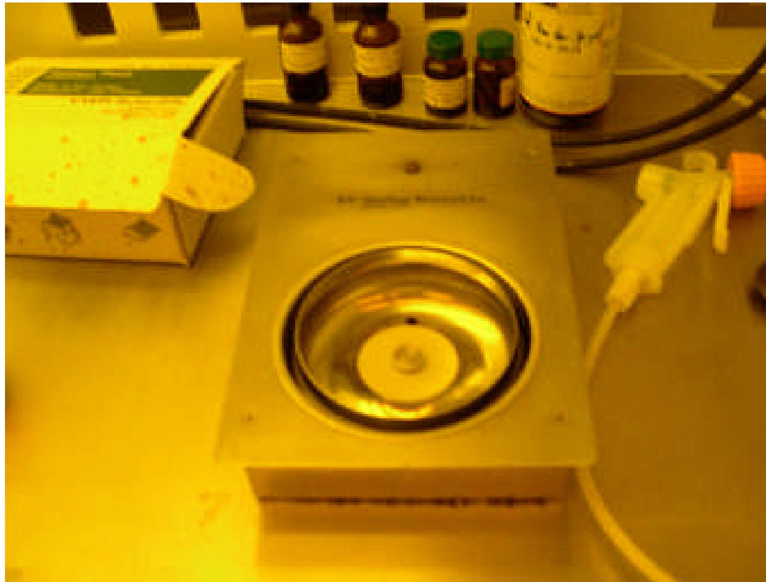


Technics PlasmaEtch™ II Oxygen Plasma instrument

Plasma Tech. Plasmalab µp



Kasper System™ 2001 Mask Aligner



Spinner

Tencor Instruments ™ Profimeter