NANOIMPRINT FABRICATION OF WIRE-GRID POLARIZERS USING DEEP-UV INTERFERENCE LITHOGRAPHY

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NANOIMPRINT FABRICATION OF WIRE-GRID POLARIZERS USING DEEP-UV INTERFERENCE LITHOGRAPHY

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ABSTRACT

NANOIMPRINT FABRICATION OF WIRE-GRID POLARIZERS USING DEEP-UV INTERFERENCE LITHOGRAPHY

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Wire-grid polarizers in the visible and near-IR spectra have a number of interesting applications in imaging because they can be made in pixel-sizes and at different orientations. They are most easily fabricated by lift-off lithography, but this reduces the wire thickness resulting in low aspect ratios and the poor polarizer extinction ratios. Alternative methods such as the damascene process have also proven to be difficult.

In this thesis, we demonstrate a nanoimprint technique where a polymer film on glass is used as the substrate for imprinting the grooves, followed by metallization. A high resolution 220nm periodic stamp, with feature sizes of the order of 100nm, is fabricated on silicon using deep-UV (266nm) interference lithography and directional plasma etching. The interference lithography process was developed and optimized for the fabrication of these nanostructures. This nanostructure is transferred onto a patternable epoxy (SU-8) using vacuum thermo-compression and in-situ UV exposure. SU-8 was chosen because it is optically clear and easily imprinted. A new in-situ UV illumination system was designed and built for the imprint. The imprinted structure also enables a unique glancing angle deposition method that is much easier for the
fabrication of wire grids than lift-off or damascene. A polarizer extinction ratio of 90 was measured at 1064nm wavelength. In this thesis we will show the results from these processes, including process details, SEM images and performance data.
Dedicated to my dear parents.
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INTRODUCTION

Human eyes are only sensitive to the intensity and color rather than polarization. Many objects around us also produce polarized light that contain useful and distinct information just like color. A number of applications have emerged recently that uses polarimetric sensors: contrast enhancement in foggy environment [1], underwater imaging [2], pollutant classification in atmosphere [3], skin cancer detection [4], surveillance, and search and rescue efforts. However, today’s polarimetric image sensors are prohibitively expensive and are used only in very specific applications. The main problem is fabrication complexity. All of the nanoscale patterning has been developed for silicon. But silicon is opaque to visible light. Glass is an ideal substrate material but it is more difficult to be patterned at the nanoscale. Regular nanofabrication methods such as lift-off have been proven to be difficult for achieving metal wire of high aspect ratio. The purpose of this research is to develop wire-grid micro-polarizers in near infrared spectra in pixel-sizes using nanoimprint. The fabrication process can be broken into three steps. Firstly, we used interference lithography followed by plasma etching to fabricate silicon stamps. Secondly, the stamps are imprinted onto thin SU-8 films to create grating structures. Thirdly, the sample is coated with aluminum at a glancing angle to form metal wires. Compared to the reported fabrication method of wire-grid polarizers, our research is a more efficient and easier process in the fabrication of micro-polarizers for real-time polarimetric imaging in near infrared spectra. Chapter 1 discusses the principle of interference lithography, plasma silicon etching, and nanoimprint. Chapter 2 gives a general overview of recent literatures of the fabrication of metal wire-grid micro-polarizers. A comparison between their fabrication methods and our method is
given. Chapter 3 introduces our experiment of stamp fabrication using interference lithography and deep silicon etching, including the experimental configuration, the solutions and inspection of the common issues, and the SEM images of grating structures of nano-scale featuresizes. In Chapter 4, nanoimprint fabrication using SU-8 for submicron structure patterning is demonstrated. The final metal wire fabrication is described in Chapter 5. Three different metal wire fabrication methods are introduced and compared. In Chapter 6, COMSOL simulations of extinction ratio as well as optical performance measurements of fabricated polarizers are given. The conclusion and the future direction of the research are discussed in Chapter 7.
CHAPTER 1
INTRODUCTION TO NANOIMPRINT TECHNOLOGY
—STAMP FABRICATION AND IMPRINT

Nanoimprint technology is a novel fabrication technology, which can be broken into two main processes, stamp fabrication and imprint. In our research project, we used interference lithography followed by plasma etching to fabricate the stamp and we used thermal compression for the imprint. In this chapter, theories are presented, and their applications related to the fabrication of the wire-grid polarizer are introduced.

1.1 Interference lithography

1.1.1 Interference of electromagnetic plane waves

In conventional mask lithography, the intensity modulation is produced by passing the light through a binary mask. In interference lithography (a nonconventional lithography), optical intensity modulation is produced by interfering two or more beams together.

Interference is the superposition of electromagnetic waves in the same region of space and time. Three conditions have to be satisfied for wave interference to occur: 1. the source must be coherent; 2. the source must be monochromatic (same frequency); and 3. the source must have the same phase relationship to each other.

As an additional condition, the phase delay between the beams must be smaller than the coherence time of the light source.

Here, a theoretical deviation of interference patterns produced due to two plane electromagnetic waves is presented:
The two plane waves can be mathematically present in the flowing form:

\[ E_1(r, t) = \text{Re}(E_{01}e^{ik_1r-\omega t}) \]

\[ E_2(r, t) = \text{Re}(E_{02}e^{ik_2r-\omega t}) \]

where \( k_1 \) and \( k_2 \) are the wave vectors and \( E_{01} \) and \( E_{02} \) are the corresponding electric field amplitude vectors. The superposition carries of the two fields will be:

\[ E_T(r, t) = E_1(r, t) + E_2(r, t) = \text{Re}[(E_{01}e^{ik_1r} + E_{02}e^{ik_2r})e^{-i\omega t}] \]

The intensity of the resultant wave is given by

\[ I(r) = |A(r)|^2 = A(r) \times A^*(r) = (E_{01}e^{ik_1r} + E_{02}e^{ik_2r}) \times (E_{01}^*e^{-ik_1r} + E_{02}^*e^{-ik_2r}) \]

which is equal to

\[ I(r) = |E_{01}|^2 + |E_{02}|^2 + \text{Re}(E_{01} \cdot E_{02}^*) \cos[(k_1 - k_2)r] - 2\text{Im}(E_{01} \cdot E_{02}^*) \sin[(k_1 - k_2)r] \]

where \( \text{Re} \) and \( \text{Im} \) respectively denote the real and imaginary part of the complex number. To observe the interference effects, \( k_1 - k_2 \) cannot be zero. In other word, the directions of propagation of the waves must be noncollinear. Additionally, the term, \( E_{01} \cdot E_{02}^* \), cannot be zero either, which means the electric field amplitude vectors (i.e. the polarization states) of the individual plane waves must not be perpendicular.

Specifically, for two plane waves interfering at the same but opposite incident angles, the deviation can be expressed as shown below:

![Figure 1-1 Schematic of two plane waves interference with the same incidence angle](image-url)
where beam 1 and beam 2 are the incident plane waves and \(x\) and \(y\) denote the horizontal and vertical directions, respectively. The electric field of plane wave can be written as:

\[
E = A e^{i(kx - \omega t + \delta)},
\]

where \(A\) is the amplitude of electric field; \(k\) is wave number \(\frac{2\pi}{\lambda}\); \(\omega\) is the frequency, and \(\delta\) is the initial phase.

The intensity distribution due to the interference along the horizontal direction of the electric field can be derived. The horizontal electric field of beam 1 is

\[
\vec{E}_{1x} = A e^{i k x} e^{-i\omega t} = A e^{ik \sin \theta_1 x} e^{-i\omega t}
\]

Equivalently, the horizontal electric field of beam 2 is

\[
\vec{E}_{2x} = A e^{-i k x} e^{-i\omega t} = A e^{-ik \sin \theta_2 x} e^{-i\omega t}
\]

The total horizontal electric field function is then

\[
\vec{E} = \vec{E}_{1x} + \vec{E}_{2x} = (Ae^{ik \sin \theta_1 x} + Ae^{-ik \sin \theta_2 x}) e^{-i\omega t}
\]

Similar to the principle we described above, the intensity of the resultant wave is

\[
I_x = |\vec{E}|^2 = I_0 \left( 2 + e^{i[k (\sin \theta_1 - \sin \theta_2)]} + e^{-i[k (\sin \theta_1 + \sin \theta_2)]} \right) = 2I_0 + 2I_0 \cos \left( \frac{2\pi (\sin \theta_1 + \sin \theta_2)}{\lambda} x \right)
\]

Let \( \frac{1}{d} = \frac{(\sin \theta_1 + \sin \theta_2)}{\lambda} \), and \(d\) is the period of our electric field intensity distribution. Therefore, the period versus angle function can be described below:

\[
d = \frac{\lambda}{\sin \theta_1 + \sin \theta_2} \quad \text{equation 1-1}
\]

where \(\theta_1\) and \(\theta_2\) are the two incidence angles. This optical intensity distribution can produce a periodic pattern.

Similarly, the vertical vectors of the two waves are also able to interfere. The intensity in vertical direction can be expressed as:

\[
I_r = 2I_0 + I_0 \left[ (e^{2i k \cos \theta_1 y} + e^{2i k \cos \theta_2 y})e^{i\Delta \delta} + (e^{-2i k \cos \theta_1 y} + e^{-2i k \cos \theta_2 y})e^{-i\Delta \delta} \right]
\]

where \(\Delta \delta\) is the phase shift caused in the reflection from substrate.
1.1.2 Photoresist

To transfer the intensity pattern onto a substrate, a photoresist is used as an intermediate medium. A photoresist is made of photoactive compound, resin, and solvent. The solvent is only to change the viscosity and transfer the photoresist onto a substrate by liquid dispensing and spin coating. It will usually be expelled during a soft bake. The resin is the “resist” part in a photoresist. It is the main structural element of the photoresist and it also forms the bulk of the remaining component. Novolac is the most common used resist. The photoactive compound (PAC) is UV sensitive and it’s the “photo” part that it will undergo a chemical reaction once exposed to UV.

A photoresist can be basically categorized into two types: positive and negative. An introduction to the chemical reaction among the PAC, resin, and developer is reviewed in [5]. Most commonly used positive photoresists contain diazoquinone (DQ) and matrix material (N). Novolac is a polymer matrix material whose monomer is an aromatic ring with two methyl groups and OH group and it is easily dissolved in aqueous solutions. The PAC is the inhibitor within the matrix material. Some have suggested that the inhibitor mechanism will become active only after thermal cycles making soft bake a necessary step [6], and that the mechanism is due to the interactions between the resin and the PAC [7]. Once exposed to UV light, the DQN will react with water and produce carboxylic acid and nitrogen. The carboxylic acid binds with the Novolac and increases the dissolution rate of the resist in the developer by a factor of 10 or more. As a result, the solubility of the resin changes after UV exposure and the exposed regions will be dissolved while the unexposed regions will remain. Photoresist which shows an increase in solubility after exposure is known as positive-tone photoresist. Since the developer does not penetrate the resist, the unexposed area of DNQ photoresist is essentially unchanged during the development. Thus the imaged small pattern will keep its shape and size. Additionally, Novolac is a long-chain aromatic ring polymer which has a low etch rate under plasma etching.

On the other hand, the working mechanism of most negative photoresists is cross-polymerization [5] – a process where large resin molecules attach to each other and become less soluble. A
popular negative resist is an azide-sensitized rubber. When it is exposed to UV light, the solubility is significantly reduced. This results in the unexposed areas being developed away and the exposed area remaining. Compared to positive photoresist, the most distinct advantage of negative photoresist is its strong adhesion to the substrate without any pretreatment.

1.2 Plasma etching

Etching can be categorized as dry, wet, physical, and chemical. Wet etching is a purely chemical process that has high selectivity and produces nearly no damage to the substrate. However, it is also limited by several drawbacks: a lack of anisotropy, poor process control, and excessive particle contamination [5]. It is isotropic, which will cause a lateral undercut below the resist. It cannot be precisely controlled because the etching rate depends on many factors such as temperature, concentration, agitation and the removal rate of gas byproducts. Additionally, the reacted particles may remain in the wafer after the etching and result in a contamination. Reviews of wet etching can be found in [8]. Ion milling or ion-beam etching is a purely physical etching because it involves no chemical reactions. It is a strictly mechanical process which is analogous to sputtering, the micromechanical analog of sandblasting. Compared to wet etching, it has a very good directionality. This is because the ions are accelerated by the electric fields in the plasma towards the cathode, and the low chamber pressure strongly reduces atomic collisions [5]. The etching method we applied in our research project is a dry chemical etching method. After the photoresist image has been transferred onto a wafer, the next step is to transfer the image pattern to the layer under the resist by etching. The photoresist acting as a mask should be highly resistant to the etching chemistry so that the areas covered by the photoresist will be protected from being etched while the uncovered areas would be etched away.

1.2.1 Reactive-ion etching (RIE)

We used plasma enhanced chemical vapor etching (PECVE) for silicon etching. Plasma is introduced to the substrate surface to decrease the temperature required for the chemical reactions
and to increase the etch rate. The working mechanism of CVE is similar to chemical vapor deposition (CVD) which uses precursor gases to synthesize the film by chemical reaction. Instead of generating a nonvolatile film reacted by the chemical gases for deposition, in CVE, it generates volatile byproduct of the material being etched. This etching is most commonly known as reactive-ion etching (RIE).

The construction of a plasma etching system is similar to plasma enhanced chemical vapor deposition. A vacuum chamber with controlled gas flow in and flow out maintains the working pressure. The sample is placed on the cathode. The cathode electrode is energized by a radio frequency (RF) source to generate plasma. The gases decompose in the plasma and generate free radical species to etch the substrate.

Since there are too many factors involved, including polymerization, thin film passivation and scavenging effects of various gas components in the plasma, accurate mathematical models of etch processes are rarely done. Most of the thermal dynamic descriptions have to be determined experimentally rather than analytically [9]. However, a crude model based on a simple thermodynamic analysis was developed for the etch rates quantitative analysis. Similar to low pressure chemical vapor deposition, the reaction rate can be expressed the surface reaction rate.

\[ J_s = k_s P_s \] (equation 1-2), where \( k_s = A e^{-(E_a/kT)} \) \( E_a \) is the activation energy and \( P_s \) is the partial pressure of the reactant species on the substrate surface. Generally speaking, increasing the temperature, power and pressure will increase the etch rate. As shown in the equation 1-2, the etch rate is exponentially dependent on the substrate temperature and a higher temperature will result in a higher etch rate. Increasing the RF power will result in an increasing percentage of free radical generation and therefore increase the etch rate. Increasing gas pressure will only increase the etch rate to a certain limit. This is because a higher pressure will scatter the ions in the plasma and to degrade its directionality.

Another important key to determine the etch rate is the ions. The primary role of ion is to sustain the plasma by bombarding the cathode and releasing secondary electrons. However, it also causes
ion sputtering on the substrate which is a mechanical etching process. The etch rate due to this process depends on the property of the film on the substrate and the sputtering yield. Although the etch rate of the ion sputtering is much lower than that of the free radicals since the ion density is smaller than the density of free radicals by many orders of magnitude, it plays a very important role in the overall etch rate when it is used to remove the nonvolatile byproduct coating which is produced in carbon containing plasmas such as the CF$_4$ plasma. Additionally, the ion sputtering can also break the surface bonds of the surface atom to increase its reaction with the free radicals. The ion density can be controlled by the working pressure and the RF discharge power.

1.2.2 Inductively coupled plasma etching (ICP)

Apart from RIE, ICP is another common but different mechanism for etching. Compared to RIE that has electric field from cathode to energize the plasma in the parallel plate configuration, the plasma can be energized by a magnetic field and this plasma is called inductively coupled plasma. A large coil is used to induce a magnetic field inside the chamber instead of connecting the cathode to the RF source to provide power. The resultant plasma will travel in a circular path around the magnetic field instead of going in a straight path. Therefore, the substrate will not experience any ion bombardment. However, the free radicals generated by ICP have the same function for etching. Without ion bombardment, this etching becomes an isotropic etching, which is not desirable. Combining the ICP and RIE into a single chamber results in a more versatile reactor. An example of the configuration is given in figure 1-2. The ICP will be generated by one RF source and the RIE plasma will be generated by a second RF source. By choosing different powers, one can achieve a purely isotropic etching without any ion bombardment to a more conventional RIE anisotropic etching, or any combination between them. The most widely used process involving ICP+RIE plasma is the Bosch etch process. It was first developed and patterned by German Bosch Electronics Company for deep silicon etching technology. Figure 1-3 shows a
deep silicon etching using this technique [10]. The ICP and RIE sources are used to sequentially repeat an ion sputter etch step for isotropic passivation deposition and an isotropic etch.

![Etching tool combined ICP and RIE plasma.](image)

**Figure 1-2** Etching tool combined ICP and RIE plasma.

![Deep silicon etching using Bosch etch](image)

**Figure 1-3** Deep silicon etching using Bosch etch [10].

### 1.2.3 Deep silicon etching

Sulfur hexafluoride (SF$_6$) plasma is the most commonly used plasma etching for silicon. The silicon reacts very quickly with the fluoride free radicals. The resultant silicon fluoride has a boiling temperature much lower than room temperature so that it becomes gas and the silicon is etched. The chemical reaction is shown in equation 1-3 and 1-4. The free radical will etch equally in all directions, resulting in an isotropic etching. Although a residual sulfur layer will form on the silicon surface, it is not a passivation. Therefore, the sulfur layer will slow down the etching instead of stopping the etching. In RIE, the ion bombardment will break the surface bond and
remove the sulfur layer to accelerate the reaction. Since the ion is driven only vertically, the vertical etch rate will be faster than the horizontal etch rate.

\[ \text{SF}_6 + e^- \rightarrow \text{S}_x\text{F}_y + \text{S}_x\text{F}_y + F + e^- \quad \text{equation 1-3} \]

\[ \text{Si} + F \rightarrow \text{SiF}_x \quad \text{equation 1-4} \]

On the other hand, the photoresist does not react with fluorine but it will be etched by the ion sputtering. Greater ion energy, greater power and higher ion density, or higher pressure, will produce a faster etching in photoresist. This is the reason that dry etching has a lower selectivity than wet etching. The sulfur will also cause some polymer crosslink that will make some photoresists become harder during etching, but the crosslink occurs only in a small range that will not be easy to control.

In common applications, researchers will add some oxygen in SF\textsubscript{6} to increase the etch rate. It is believed that the O\textsubscript{2} can combine with the S-F radicals in the plasma and prevent them from scavenging the useful F radicals and reforming SF\textsubscript{6}[9]. But too much O\textsubscript{2} will etch photoresist and decrease the selectivity. Typical SF\textsubscript{6} and O\textsubscript{2} mixture ratio is 10:1 [9].

CF\textsubscript{4} is also commonly used in deep silicon etching. A CF\textsubscript{4} plasma works similar to the SF\textsubscript{6}. However, the carbon is more robust than the sulfur and it can react with silicon and fluorine to form compounds. The compound it forms with silicon is silicon carbide (SiC) which is a hard dielectric film. The reaction is shown in equation 1-5. The compounds it forms with fluorine are fluorocarbon polymers which are hard plastic and inert coatings. These robust thin films will decrease the etch rate, therefore the etch rate of SF\textsubscript{6} is faster than that of CF\textsubscript{4}. However, ion bombardment can facilitate the etching by breaking the surface bonds so that the etching profile of CF\textsubscript{4} is more anisotropic than SF\textsubscript{6}. This is the tradeoff of CF\textsubscript{4}. It has a slower etch rate but more anisotropic etching than SF\textsubscript{6}. Similar to SF\textsubscript{6}, O\textsubscript{2} is used with CF\textsubscript{4} to reduce the C-F radicals from scavenging other F radicals and increase the etch rate. By choosing the suitable gas mixture of SF\textsubscript{6}, CF\textsubscript{4}, and O\textsubscript{2}, scientists are able to perform a fast and anisotropic etching for a deep silicon etching.
1.3 Principles of nanoimprint lithography

1.3.1 Introduction of nanoimprint

Lithography technologies have been massively deployed in the microelectronics industry. Conventional lithography involves exposure to UV light, although charged beams such as electrons and ions can also be used in e-beam lithography and ion beam lithography. However, these patterning methods are facing challenges due to resolution and throughput. In 1995, an alternative lithography, nanoimprint lithography (NIL), was proposed and demonstrated by Stephen Chou[11]. This method is completely different from the conventional photolithography methods. It is a mechanical patterning method instead of inducing a chemical reaction by radiation. The idea of nanoimprint is simple (see figure 1-4): The desired nanostructure is first fabricated in a mold or template using a high-resolution lithography method. This then acts as the mask for subsequent processing. Then, nanostructures are mechanically duplicated from the mold surface to a layer of deformable materials. To facilitate nanoimprint, the deformable material is usually in a liquid phase during the imprint and becomes fully or partially solidified before the mold is removed. This is a high throughput, high resolution and low cost mechanical patterning method which can be used in large area manufacturing. Therefore, it is also being called the next generation lithography.

Figure 1-4 Schematic of NIL: (a) deposit imprinting material on substrate, (b) using mold to transfer desired patterns to the material, (c) release mold from the material.
1.3.2 Key technologies in NIL

For a successful nanoimprint manufacturing, several key steps are required. In this section, the mold (mask), imprinting materials, and imprinting process will be introduced.

1.3.2.1 Mold (Mask)

An imprint mold is required to have (a) desired nanostructures, (b) good fidelity in pattern transfer, and (c) low defect density. In the above requirements, (a) is related to the patterning of the mold, and the requirements (b) and (c) are primarily related to mold materials and structures [12]. The molds and stamps are typically made of materials that have a high Young modulus.

An imprint mold can also be categorized as hard or soft based on its material. To increase individual pattern transfer and overlay fidelity, and to reduce distortion during the imprint, hard molds are preferred. Typical molds with high mechanical hardness are made of quartz, silicon, silicon dioxide, silicon carbide, silicon nitride, metals, sapphire, and diamond film. Plenty of works from other research groups have demonstrated that Si and SiO$_2$ have sufficient hardness and durability properties for nanoimprint application. During the nanoimprint process, thermal expansion plays an important role. There should be minimal thermal mismatch between the mold and substrate, for avoiding pattern distortions or stress build-up. Several room temperature NIL techniques have also been developed, including UV-assisted NIL, room temperature NIL and Step-and-Flash Imprint Lithography (SFIL) process. On the other hand, a soft mold is able to cover an uneven surface and reduce the damages caused by dust on surfaces. Typical soft mold are polymers, including polydimethylsiloxane (PDMS). Another candidate for soft mold is Teflon AF 2400. It is a copolymer of 2, 2-bis-trifluoromethyl-4, 5-difluoro-1, 3-dioxole and tetrafluoroethylene. It has a low energy of 15.6 dyn/cm (cf. PDMS, ca. 19.6 dyn/cm), which makes it easy to release from substrates without any surface treatment [13].

Nanostructures can be patterned using conventional or nonconventional lithography. The conventional patterning methods include electron beam, ion beam, scanning laser beam, and
photolithography. The nonconventional patterning methods include interference lithography, edge lithography, self-assembly and guided self-assembly methods, as well as combining these with conventional lithography. Factors such as resolution and cost determines the lithography technology used in the mold fabrication. For example, compared to interference lithography, electron beam lithography can make various high resolution structures, but it is also an expensive and time consuming method. Austin et al. have also demonstrated a high resolution NIL (14nm pitch pattern) using molecular beam epitaxy to grow a superlattice structure on GaAs [14].

A release agent is necessary to prevent the mold from permanently adhering to the substrate. There are several approaches, which can also be used in combination. One method is to coat a release agent on the mold surface. The most widely used method is a self-assembled monolayer of a fluorosilane release agent (e.g., 1H,1H,2H,2H-perfluorodecyl-trichlorosilane) [15]. Another approach is to add a release agent into the imprint material, called internal release agent. Another one is fluorinated mold materials. The release agent should have: ultrathin thickness or monolayer so as to not deform the nanostructures, good release property, and good durability [12]. By using atomic force microscopy, ellipsometry, IR spectroscopy, and contact-angle measurement for the comparison of the above process, Jung et al. have demonstrated that vapor-phase coating method provided a superior surface-release property [16]. Based on the material, a release agent on mold surface can be separated into two classes: fluorined polymers and diamond-like carbon. The fluorocarbon rated material has a low surface energy and a weak interaction with the imprint material. Because of the saturated carbon bonds, the carbon-like material also has low surface energy [12]. Recently, Schift et al. demonstrated that the substrate coated with fluorinated trichlorosilanes can be further improved by co-deposition of monochlorosilanes [17].

1.3.2.2 Imprinting materials

A good imprinting material has the following properties: good flow properties during the imprint, good shape fidelity after imprint, small shrinkage, and good demolding property. In addition, the
imprinting material can act as a part of the device. Therefore, the imprinting material is also determined by its functionality in the device. There are 3 general types of imprinting material: thermal plastic materials, curable UV or thermal materials, and hybrid material.

Thermal plastic is a material that is solid state under its glass transition temperature while it turns to liquid phase when heated above its glass transition temperature. When the resist is heated above its glass transition temperature, both Young’s modulus and viscosity will drop by several orders of magnitude compared to their values at room temperature. The chemical reaction is that, due to the weak bonding between the linear polymer chains, the chains can slide past each other at high temperature, making the material flow under pressure. Practically, it is usually to raise the temperature 70-90 degree above the glass transition temperature (Tg) to obtain a viscous flow rate [15]. Nowadays, there are new photoresists that require low operating temperatures; however, there is a trade-off between the imprinting temperature and the thermal stability. Although it is not difficult to obtain high fidelity nanostructure from both low Tg and high Tg resist materials, the nanostructures from each show different pattern relaxation at room temperature [18]. Nanostructure on lower Tg resist material will relax faster than those on higher Tg resist material resulting in pattern distortion in a shorter time period.

The resist material in our research is SU8 patternable epoxy. SU8 is a permanent epoxy which is cured by exposure to UV and subsequent thermal cross-linking. Therefore, it is a negative-type resist.

1.3.2.3 Nanoimprint process

The process steps in nanoimprint are simple and straight forward. As shown in figure 1-4, during the imprint, the resist material underneath the protrusions must be squeezed and transported to nearby holes or cavities. This is a so called squeezed-flow model. However, the cavity-fill process is rather complicated. The duration of the imprint step depends on the mold geometry, resist material properties, and the process conditions.
The name “nanoimprint” is an accurate representation of the process because it takes longer to pattern large structures than nanostructures. Hirai et al. have investigated the pressure required for complete imprinting and filling in the grooves of the mold as a function of the aspect ratio of the pattern, the initial thickness of the polymer, and the duty cycle of the structure for a periodic pattern [19]. They demonstrated that the required pressure is high for both high aspect ratio structures as well as low aspect ratio structures. The reason is because the pressure is not evenly distributed for wide trenches, resulting in some resistance to polymer flow. Simulation results are shown in figure 1-5 [21]. The filling rate of the resist at the center is faster than at the edge of the cavity. The required pressure for imprint also depends on the initial thickness of the resist. The pressure is increased when the initial thickness is less than twice the mold depth. Rowland et al. have also found that the polymer can deform as single peak and dual peak closed to the vertical sidewall when it is filling into the cavity. Normally, the resist deforms as single peak when the ratio of half width to initial polymer film thickness is less than unity. The cavity will be filled fastest when for a geometry at the dual/single peak threshold [20-22].

Figure 1-5 Schematic of polymers deformation process during nanoimprint [21].
Other imprint process includes reverse-nanoimprint (figure 1-6). It is based on making the surface energy of the mold lower than the substrate. Jay Guo et al. have demonstrated this technology in [23]. This technology allows transferring pattern onto substrates that are not suitable for spin-coating or have high surface topographies.

Figure 1-6 Schematic of reverse-imprinting nanostructures over an existing topography [23].
CHAPTER 2

CURRENT WIRE-GRID MICRO-POLARIZER FABRICATION METHODS REVIEW

The wire grid structure size and the fabrication methods of wire grid polarizers are different based on the working spectral region. In this chapter, some fabrication methods for micro polarizers from selected works would be introduced to give a general idea about the nanofabrication techniques which had been used. These techniques would be compared with our fabrication methods.

2.1 Current wire-grid micro polarizer fabrication methods

Our research group has reported a high performance wire-grid polarizer for mid infrared applications [24]. The technique used in fabricating the wire-grid structure is projection lithography. This technique enables the wire-grid in four different directions to be fabricated at one time and on a large area (up to 26mm x 32mm). Using the lift-off method, 200nm thick aluminum was deposited on a silicon wafer and patterned. The resultant micropolarizer array shows a high extinction ratio exceeding 30dB. It is the highest reported micro-polarizer performance in the MWIR band.

Jian Jim Wang et al. reported a high-performance, large area, deep ultraviolet to infrared polarizers based on 40 nm line/78 nm space nanowire grids [25]. Because the spectral region is from UV to IR, fused silica is chosen to be the substrate material. They first deposit a layer of aluminum by e-beam evaporation on the fuse silica. Then a layer of SiO₂ is deposited by e-beam evaporation. To build the wire-grid structure, they use full-wafer immersion interference lithography [26]. This technique reduces the period by a factor of the fluid’s refractive index.
After the wire line is patterned, RIE is used to etch the SiO$_2$ and Al anisotropically. The resultant micro-polarizer shows an extremely high contrast from 10000:1 to 50000:1 across the whole visible and near-infrared wavelength range and a good TM wave transmittance (85%-90%).

A wire-grid polarizer specific for the UV spectral region was reported by Thomas Weber et al. [27]. They introduced a technique called the spatial frequency doubling [28]. The whole process can be described in the figure 2-1. They used e-beam lithography to create a 200 nm period grating pattern into the electron-beam resist layer, which was previously spin coated on a layer of chromium. After development the chromium layer was etched by ICP. In the next step the chromium acted as a hard mask and the photoresist was etched by a O$_2$ plasma, resulting in a 160nm high and 100nm wide high aspect ratio grating structure. A sputter process under an oblique incidence angle was used for deposition of aluminum with a thickness corresponding to the ridge width of the grating. The smooth sidewall of the photoresist grating structure was used as a carrier for the aluminum grating. After the aluminum deposition, they used an ion beam etching process to remove the metal from the bottom of the grating and the top of the ridges, followed by a dry etching process to remove the remaining photoresist. The result is an aluminum grating with half of the period of the polymer carrier grating [30].

Figure 2-1 Schematic and the SEM of Al grating structure fabricated in spatial frequency doubling method [27].
Fabrication of a 50 nm half-pitch wire grid polarizer using nanoimprint is reported by Seh-Won Ahn et al. [29]. Similar to our method, they used nanoimprint to fabricate a stamp and used it to form a grating structure on an imprint resist as shown in figure 2-2 (a), (b) and (c). The structured imprint resist (mri-8020) on the aluminum layer deposited earlier became a mask in the later etching process. The grating structure was transferred to the aluminum layer by the Al etching performed in BCl$_3$/Cl$_2$ plasma enhanced by a magnetic field. After Al etching the residual photoresist was stripped. The resultant aluminum grating structure had an aspect ratio of 4 and the polarization extinction ratio achieves 2000 with transmission of 85% at a wavelength of 450 nm.

Figure 2-2 Schematic of Al grating structure fabricated using nanoimprint (left) and the SEM of the result (right) [29].

In 2013 Chia-Meng Chen et al. reported fabrication of bi-layered metallic wire-grid polarizers by nanoimprint and O2 plasma etching [30]. The newly designed bi-layered structure (figure 2-3) gives more degrees of freedom to optimize the impedance matching for one polarization. The wire-grid structures provide birefringence, which provides the opportunity to make a thin layer with different property for two orthogonal polarizations. The double layers provide more degrees of freedom to achieve this desired performance. In the fabrication method, similar to the previously discussed article, they used nanoimprint to fabricate the grating structure. Interference
lithography and RIE were employed in the stamp fabrication. The stamp was used to transfer the structure to the epoxy PMMA. After the imprint, aluminum was deposited onto PMMA and a chemical mechanical polishing is used to planarize the metal surface. The resultant bi-layered wire-grid structure with wire gratings showed 100 nm in linewidth, 240 nm in pitch, and 300 nm in total height. The performance of the polarizer had an extinction ratio of 497 and a brightness gain of 1.18.

![Schematic of Al grating structure fabricated using nanoimprint](image)

Figure 2-3 Schematic of Al grating structure fabricated using nanoimprint [30].

2.2 The significance of proposed research

Basically, the approaches people have used are based on interference lithography or electron beam lithography to make patterns on a photoresist film followed by chlorine plasma etching or, deposit aluminum to fill the spaces between the photoresist and then remove the unwanted metal by polishing.

There are several limitations to these approaches. Some works have used silicon as a substrate because it is the most developed material for nanofabrication processes. This works in the infrared spectrum because silicon is transparent. But silicon is opaque to visible and ultraviolet light. Therefore, the techniques developed for silicon cannot be used to make polarizers for
visible light. The other drawback is that these methods use expensive processes, and unique facilities, such as e-beam lithography and chlorine etching, to fabricate just one micro-polarizer array. Only one imaging array can be built from this array. The next micro-polarizer array will require the same number of resources and processing steps. For instance, to improve metrics of optical performance such as the extinction ratio, one has to increase the aspect-ratio, reduce the line width and increase the depth of the wires. However, it is exceedingly difficult to make wire lines with a pitch under 300 nm, especially if their thicknesses are also of the same order of magnitude as their width.

Therefore, in this thesis we demonstrate a new method by using nanoimprint lithography and a different metallization method. The grating structures were fabricated on a silicon stamp and then nano-imprinted onto a transparent epoxy (SU-8) on glass. Then the wiregrids were fabricated directly on the SU8 structures by shadow masked deposition of the metal. These polarizers are being fabricated on a glass substrate to make it possible to use them in visible applications.

2.3 Conclusion

We have summarized some nanofabrication methods of wire-grid polarizer from the selected publication. Comparatively speaking, our fabrication methods show a great advantage in the fabrication efficiency and potential for high extinction ratio polarizers. In the next chapter, we will introduce our fabrication of silicon stamp for the nanoimprint of grating structures.
CHAPTER 3

STAMP FABRICATION USING DEEP ULTRAVIOLET INTERFERENCE LITHOGRAPHY

To fabricate the periodic nanometer feature size structure of the proposed polarizer using nanoimprint, the first and the most important step is the stamp fabrication. As mentioned in Chapter 1, the quality of the stamp determines the quality of the imprinted structure as well as the performance of the polarizer. Deep ultraviolet (DUV) interference lithography (IL) is able to create structure size from a few microns to a few nanometers. Although interference pattern is easy to create with two coherent laser beams, to capture the optical pattern and transfer it to photoresists are difficult since there are many unknowns to cause defects. Our research group has spent a number of years to perfect this process and we have developed a new method for achieving feature size of 110nm. In this chapter, the developed fabrication procedure and recipe of interference lithography as well as the consequent plasma etching and the SEM data will be presented.

3.1 Substrate preparation

We have developed a new preparation method to address a common issue that comes from the substrate preparation. A standard method and the new developed method will be discussed, and a comparison will be given.

Standard method: Prime grade silicon <100> three inch wafer is chosen to be the sample wafer. The naturally formed oxide layer on the silicon is removed by a buffered oxide etch (BOE) with 50:1 Ammonium Fluoride to hydrofluoric (HF) acid ratio. It usually takes less than one minute to
completely remove the thin oxide layer. After that the wafer is rinsed by deionized water. Pure silicon is hydrophobic, and this property can be used to check whether the silicon wafer is oxide-free. To increase the adhesion between the photoresist and the silicon wafer, the wafer is put into a Vacuum Bake/Vapor Prime (figure 3-1) system for a vapor priming with hexamethyldisilazane (HMDS). The system is first set to 150 degree C and pumped down to a few milliTorr pressure. Three pump and dry-nitrogen purge cycles is performed to dehydrate the wafer before flooding with HMDS. HMDS vapor pressure is maintained for five minutes. Then, the chamber is pumped down and nitrogen purged again repeatedly to remove all traces of the HMDS before venting. After the HMDS treatment, the wafer is ready for photoresist spin coating.

![Figure 3-1 YES HMDS Vapor Prime System.](image)

New method: The same wafer is used. To clean the organic particles on the wafer, the AMI (acetone, methanol, and Isopropyl alcohol) cleaning is applied. The wafer is then treated with oxygen plasma for organic removal and increasing the surface energy. This makes the silicon surface become hydrophilic. After that, the wafer is spin coated with bottom anti-reflection coating (BARC), BSI.N0889A, by 1500 ramp per minute. The wafer is then baked for one minute at 190 C. The BARC is an organic layer to absorb the UV and reduce the reflection from the substrate. The exposed area is soluble in the developer and the solubility depends on the soft bake
temperature [31]. A higher soft bake temperature will result in a more robust film and harder to be developed.

In Chapter 1, we discussed that vertical interference generates ripples on the side wall of the grating structure. When the vertical interference grows in amplitude, it can create regions of under- and over-exposed layers within the photoresist. Figure 3-2 (a) is fabricated using the standard preparation method. The image shows that adjacent gratings are being connected to each other due to the standing wave. Figure 3-2 (b) shows that SEM image of the sample fabricated using the new preparation method discussed. The grating structure is greatly improved and the substrate is clean, which shows the improvement of using BARC compared to the standard method.

![Figure 3-2 SEM image of grating structure pre-treated with (a) and without BARC (b).](image)

Furthermore, we found the BARC is an ideal intermediate material for interference lithography on different substrates. Because of the reflectivity and the adhesion property on materials, such as conductors, dielectrics, and semiconductors, are various; the recipes for these materials vary widely. However, by applying the BARC, we can use one recipe for fabricating structures with 200nm feature size on these three types of material.

In the experiment, we have fabricated 2D cross grating structure on different substrate material with UVN 30. The reason we fabricated 2D structures instead of 1D is because it is easier to create small structures in 2D pattern than in 1D pattern. The 2D pattern on silicon (b),
molybdenum (c), and sapphire (d) shown in figure 3-3 were fabricated in a very similar recipe. The cross-section profiles of pattern on each material are similar as shown in figure 3-3 (a). We believe the BARC has a strong enough adhesion to different substrate and this property would allow it to become an intermediate layer for the photoresist and different substrates for interference lithography.

![SEM images of 2D grating structure on silicon (a) and (b), molybdenum (c), and sapphire (d).](image)

Figure 3-3 SEM images of 2D grating structure on silicon (a) and (b), molybdenum (c), and sapphire (d).

### 3.2 DUV photoresist

The photoresist we used in our experiment is the UVN30 negative photoresist. It is a deep-UV chemically amplified photoresist. It creates a photoacid upon exposure to a deep-UV radiation. The acid acting as catalysis then destroys the inhibitor at an elevated temperature (during post exposure bake) [32]. There are three parameters to control the thickness of the photoresist in the process of spin coating: spin coating speed, viscosity of the photoresist, and spin coating time.
The film thickness is a function of spin coating speed, higher spin coating speed resulting in a thinner film. The dilution makes the UVN30 become less viscous and therefore the spin coated film becomes thinner during the spin coating. One minute of spin coating time ensures to spin coat a uniform thin film on the wafer surface. We have found that the photoresist with an aspect ratio too high will collapse easily during the development. Figure 3-4 shows the photoresist with 4.5:1 aspect ratio collapses. Therefore, after large number of experiments, we set the spin speed to 2000 rpm, spin coating time to one minute, and dilute the photoresist (with Rohm & Haas thinner P) in 3:1 ratio to have 300nm thick UVN30.

Figure 3-4 High aspect ratio structure collapses [33]

The photoresist is spin coated on a Laurell WS400-LITE Spin Coater (Figure 3-5). The wafer is fixed on the vacuum-secured rotating chuck. The UVN30 is transferred from the chemical bottle to the wafer surface and it is uniformly spin coated on the wafer. Since the solvent in the photoresist is only used to transfer the photoresist, the wafer is put on a hot plate at 90 C to evaporate the solvent. This process is also called the soft bake.
3.3 Experimental configuration

An interference lithography system has been designed and set up by our research group in the previous years. We have optimized the setup to obtain stable laser output since the stability of the laser determines the quality of the pattern. The laboratory configuration is shown in figure 3-6. The system is set on a floating table and it is equipped with a deep-UV laser system which consists of two parts, a solid-state diode-pump 532nm Coherent Verdi laser which has a maximum output of 2W and a MBD-266 resonant frequency doubling unit. The output laser is a coherent deep-UV beam at 266nm with a maximum power of 200 mW. However, the output beam does not have a HG$_{00}$ profile. A Thorlab KT310 Spatial Filter is placed after the laser system to clean up the output laser. Because the output laser beam size is small and has a Gaussian intensity distribution, the laser beam is then fed into a Newport GBS-UV-H UV beam shaper and a beam expander lens. The laser beam is flattened and expanded towards the rotating stage. Additionally, to monitor the output laser power, a beam splitter is used to split a small fraction of the power for real time monitoring, and a shearing plate with a microscope camera is used to check the stability of the output laser. Finally, a Lloyds Mirror configuration for interference exposure is setup on the Newport 495CC rotating stage. The expended laser can be seen as two laser beams. Therefore, the laser reflected by the mirror will interfere with the one directly expose to the sample.
3.4 Common issues due to exposure and development

The structure made by interference lithography depends on several parameters: incident angle between the two beams, exposure dose, post bake temperature (PEB), and development time. For the Lloyd’s mirror interference configuration, the resulting grating period can be calculated using the equation below:

\[ \Lambda_{grating} = \frac{\lambda_{laser}}{2 \sin \theta_{mirror}} \]  

equation 3-1

Although all the suggested fabrication parameters of UVN30 have been given in the specification sheet, it took some time for our group to optimize the recipe. Common issues in interference lithography include over exposure, under exposure, low adhesion on silicon surface, under development, and vertical standing waves due to back reflection.
Figure 3-7 Typical issues of interference lithography; over-exposed (a), under-exposed (b), weak adhesion between BARC and UVN 30 (c), and under-developed (d).

Figure 3-7 (a) shows an SEM image of a typical over-exposed sample. The sample is exposed to UV light for too long so that even those areas where the intensity is low trigger the chemical reaction and stay on the substrate during development. Figure 3-7 (b) shows a sample which is under exposed. It is clear that the small amount of photoresist is patterned and left on the BARC. However, due to insufficient exposure, most of the photoresist is dissolved during the development leaving a layer of BARC. The disordered structure shown in figure 3-7 (c) is due to low adhesion on the silicon surface. The small structures tend towards each other and cause clutters when the surface adhesion is low. Figure 3-7 (d) is a sample structure which shows an issue of under development (5 seconds). As seen in the SEM picture, the photoresist is not fully developed to the BARC. This is because the development time is too short to remove all the exposed photoresist.
The fabrication process has been optimized as shown in the table below. The UVN30 negative photoresist is diluted in the solvent (Shipely’s thinner P) in a 3:1 ratio and spin coated at 2000 rpm for 60 second. The sample is then exposed to deep-UV light at certain degree for an exposure dose of 5 to 7 mJ. It is found that 50% duty cycle of the grating structure is achieved at this dose range. Then the sample is placed on a hot plate for post exposure bake (PEB) at 95 C for 60 seconds. This temperature triggers the chemical amplification process in the photoresist to change the solubility. However, a temperature too high can cause thermal diffusion of the photoacid, resulting in a diffused pattern. Finally, the sample is immersed in a Rohm and Hass MF-319 developer (dilute Tetra Methyl Ammonium Hyroxide) for 10 to 30s and then rinsed in DI water. Although the development time is usually one minute, in our experiments we found that the photoresist will eventually all dissolve in the developer when the development time is one minute. This is at least partly attributable to the grey-scale nature of interference lithography. Furthermore, the BARC is not easy to be dissolved in the developer in a short time (10s). On one hand the robust BARC plays an important role in supporting the grating structure, keeping them from collapse during the development. On the other hand the etch rate of silicon will be decreased due to the BARC because the BARC will be etched first. Therefore, we optimized the development time to 30s to develop the BARC. The structures which have been developed for 30s in figure 3-8(a) show clean silicon surface on 77nm thick BARC. Comparatively, the residue BARC is clear shown the structure in figure 3-8 (b) which have been developed for 10s.

![Figure 3-8 BARC structure after developed for 30s (a) and 10s (b).](image-url)
3.5 Visual examination of the structural quality

The structures we fabricated are in a nanometer scale which cannot be seen even by the most powerful optical microscope. However, we use some methods to easily and visually check the quality of the exposed patterns.

A very simple method is to observe the optical diffraction. A periodic structure will show diffraction if the grating period is larger than, but of the order of half a wavelength of visible light. This approximately corresponds to a grating period of 250nm. By applying this simple method, we can roughly assess the depth of the grating, uniformity and whether the sample has been over-exposed and under developed. A good sample, such as shown in Figure 3-9, shows uniform diffraction while an over-exposed sample will show no diffraction.

![Figure 3-9 Well patterned structure shows uniform diffraction.](image)

In fact, our samples show that only those samples which have period above 250nm show diffraction that we are able to observe. A structure with larger period would show a stronger diffraction and a smaller period would show a weaker diffraction. For those samples with smaller periods, we can observe the diffraction in a beaker of water, which effectively raises the grating period by about 35%. This allows a structure with a period smaller than 230nm to also show diffraction. This is the most common way we used in our experiment to check the sample quality, before examining them under a SEM.
Another issue that affects overall quality is the missing photoresist due to the weak adhesion between the BARC and the UVN30. A uniform structure will show a uniform diffraction. A non-uniform structure will also show diffraction. However, there is a distinct difference in the diffraction between a good sample and a sample with defects. As we can see in figure-3.10 (a), compared to the sample in figure 3-9, this sample is non-uniform with very weak diffraction although both samples have the same period. The missing pattern of the photoresist is due to the non-uniform of the exposure beam. As shown in figure 3-10 (c), the profile shaped laser beam has an uneven intensity distribution. The photoresist on the area where the beam power is small will be easier to dissolve in the development while the photoresist on the area with high power will remain. When observed in the SEM, the missing photoresist result in missing structure. Therefore, the integrity of the photoresist is very important for the nano-scale structure.

![Figure 3-10 Sample of missing pattern (a) and laser beam profile for exposure (b).](image)

Last but not the least, the develop time is also used as a check. Since the BARC is harder to dissolve in the developer then photoresist, it is difficult to distinguish whether the sample with periods under 220nm is over-exposed because, even in the water, the diffraction for this type of sample is very weak. Although the BARC is developer soluble, when the sample is over-exposed, the photoresist covered on the BARC would significantly impede the BARC from dissolving. Normally, the BARC would be almost dissolved in developer in one minute. Comparatively, it
will take much longer time for an over-exposed sample to dissolve. Therefore one minute of development is used to check whether the high resolution sample is over-exposed.

3.6 Optimized recipe and SEM data

The optimized recipe for the silicon stamp fabrication is listed in table 3-1. Based on this recipe, we are able to consistently fabricate samples at incident angles from 15 degrees to 35 degrees. SEM images in figure 3-12 show that our structure sizes are high consistent with the simulated results shown in figure 3-11 which is based on equation 3-1, meaning our samples were able to capture the interference pattern and transfer the pattern to the photoresist without distortion. Furthermore, the line width of our fabricated structure has exceeded the limitation of the photoresist shown in the specification data sheet. The smallest line width demonstrated in the specs is 150nm [35] while our structures have 110nm wide lines as shown in figure 3-12. Although the specs sheet does not state the highest aspect ratio that UVN30 can achieve, the optimized experiment procedure produced grating structures with aspect ratio closed to 3. This high aspect ratio structure as well as the nearly 50% duty cycle shown in the SEM images are very desirable. Because in our design the metal wires will eventually take over this feature size and the polarizer is able to achieve a high extinction ratio.

Figure 3-11 Grating period versus mirror angle.
<table>
<thead>
<tr>
<th>Preparation</th>
<th>Photoresist</th>
<th>Exposure dose</th>
<th>PEB</th>
<th>Development</th>
</tr>
</thead>
<tbody>
<tr>
<td>BARC 1500rpm/190 C</td>
<td>UVN30 diluted in 3:1</td>
<td>5-7mJ</td>
<td>95C/60s</td>
<td>10-30s</td>
</tr>
<tr>
<td>2000 rpm, 90C/60s</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 3-1 Optimized recipe for interference lithography

Figure 3-12 SEM images of grating structure at the incident angle of 35 degree.

3.7 Plasma etching of Silicon

Plasma etching is a step to transfer the pattern of the photoresist to the silicon substrate. The dehydrated photoresist acts as a mask and it has some resistance to the plasma and chemical etching. Therefore, the area without photoresist on top is etched faster than the areas with photoresist. To increase the resistance of the photoresist, the sample is put on a hot plate at 100 C for at least one hour. This step is known as the hard bake.

A straight side wall of the sample is important in the later metal planarization. To obtain an anisotropic etching for straight side walls, the sample is etched by RIE. However, the gas combination of CF₄ and SF₆ has to be optimized. Too much CF₄ will slow the etching too much and therefore the etching selectivity between the photoresist and the silicon will decrease. However, too much SF₆ will cause unfavorable isotropic etching. Figure 3-13 shows a typical sample under isotropic etching, or we can say the CF4 in this etching is not enough to protect the
side walls from etching. After some trials, the optimal recipe for anisotropic etching was developed (table 3-2).

<table>
<thead>
<tr>
<th>Gas flow rate (SCCM)</th>
<th>RIE power (W)</th>
<th>Etch time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CF₄</td>
<td>SF₆</td>
<td>RF</td>
</tr>
<tr>
<td>49</td>
<td>1</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 3-2 Recipe for deep silicon etching

Figure 3-13 SEM picture of isotropic deep etched silicon sample.

Figure 3-14 is a SEM picture of the sample with photoresist after the deep silicon etching. The picture shows the etching is anisotropic, resulting in a straight side wall. The etching depth is deep enough to produce a near 100nm high grating structure. This etching is desirable because the aspect ratio of the resultant grating structure is near to 1. This structure will consequently transfer to the imprinted SU-8 and the later metal wire will have such high aspect ratio, which is much higher than lift-off method can achieve.

Figure 3-14 SEM picture of anisotropic deep etched silicon sample.

36
In addition, it is found that the profile of the photoresist after etching is not flat. This is because the sputtering yield depends on the plasma incident angle. Based on Sigmund’s theory, the peak sputtering yield occurs at the incident angle of 66 degree. Two competitive effects determine the peak of angular dependence of sputtering yield: the increased energy deposited on the surface by the incident ion and the decreased depth travelled by the sputtered atom [36].

The photoresist is then stripped and the stamp is finally fabricated. Due to the uniformly imprinted structure, at certain angles, the imprinted sample will diffract white light from the fluorescent lamp and show a rainbow of colors which can be used as a first indicator of the samples. Figure 3-15 shows the diffraction due to uniform structure and the SEM picture of a finished stamp.

![Figure 3-15 Diffraction from a silicon stamp (a) and SEM images of the silicon stamp, (b).](image)

3.8 Conclusion

In conclusion, we used interference lithography followed by plasma silicon etching to fabricate silicon stamps of 220nm pitch and 110nm line width with an optimized procedure and recipe. This high aspect ratio structure shows a potential high aspect ratio in the subsequent imprinted SU-8 structures. In addition, we have found the BARC is a desirable intermediate material for different substrates to reduce the influence due to material properties in DUV-IL. In the next chapter, we will introduce our nanoimprint technique using the silicon stamp fabricated in this chapter.
Glass, or fuse silica, is a perfect substrate material for polarizers for the visible and near infrared spectrum. However, the etching selectivity between glass and photoresist is very low resulting in a low aspect ratio grating structure, which detrimentally decreases the extinction ratio of polarizers. Creating SU-8 grating structures on glass is an alternative. SU-8 is an ideal optically clear patternable epoxy that can be applied on glass slides using nanoimprint technique. In this chapter, we will describe our SU-8 nanoimprint technique, including working mechanisms of related chemicals, our self-customized in-situ UV illumination system, and an optimized experiment procedure.

4.1 Principle of nanoimprint using SU-8

The formal name of SU-8 is epoxy-Novolack Epon SU-8. It was originally developed and patented by IBM-Watson research center in 1989 and first adapted for MEMS applications by EPFL-Institute of microsystems and IBM-Zurich [37]. In this section, a brief introduction of SU-8 as well as the working mechanism of nanoimprint using SU-8 is given.

SU-8 consists of three basic components as shown in figure 4-1: 1) an epoxy call Epon SU-8, 2) a solvent called gamma-Butyrolactone (GBL), and 3) a photoacid generator taken from the family of the triarylium-sulfonium salts. SU-8 is favorable for MEMS applications because of its low molecular weight, good solubility, high optical transparency, low glass transition temperature before exposure an excellent chemical resistance, and good biocompatibility [40]. The chemical reaction of SU-8 is similar to any negative type photoresist. Photoacid is produced when it is
exposed to UV light, which catalyzes the cross-linking reaction during the post exposure bake (PEB). The exposed areas cross-link while the unexposed areas remain without change. The PEB is necessary for the cross-linking because the reaction kinetic of the cross-linking mechanism is very slow at room temperature. When the temperature is raised above its glass transition temperature (Tg=65°C) the molecular motion is increased and therefore assists the cross-linking process [40]. This creates a dense network that is insoluble in the developer. The PEB must be carried out at a temperature above Tg, typically 90°C. During the cross-linking, some shrinking will be inevitable, which can cause considerable film stress and lead to adhesion failure. The cross-linking will proceed in the film until the film is completely cured. The reaction will slow down and eventually stop. The Tg of the cross-linked SU-8 is above 200°C.

![Chemical structure of SU-8 molecules](image)

Figure 4-1 The chemical structure of the SU-8 molecules [38].

### 4.2 Experimental configuration

One of the challenges is to figure out the process optimization for the SU-8 nanoimprint step. We chose SU-8 as our imprint material because of its excellent optical clarity and structural properties. Although SU-8 has been used widely in Micro Electro Mechanical Systems (MEMS), it is a novel material in nanoimprint. A special nanoimprint machine that can provide UV light, high temperature and pressure at the same time is necessary in the nanoimprint process. Our laboratory has a thermo-compression bonding tool originally designed for bonding wafers. It can also be used to compress a stamp to a soft film under vacuum, high temperature and pressure.
However, it doesn’t have a UV source for the SU-8 cross-linking process. Figure 4-2 is a photo of our thermo-compression tool and a sample being put in the chamber of the tool. Due to the high vacuum conditions inside the chamber and the configuration of the bonder plate, there is no space for inserting a conventional UV lamp around the sample. Therefore, we designed and built three high-power UV LED light sources and also custom designed corresponding clamping fixtures. Furthermore, for the higher resolution structures as we mentioned above, to fully fill the cavities in the stamp with SU-8 is a challenging task since it takes longer exposure. The UV LED’s also require cooling capability to protect them from overheating for long exposures. We spent significant effort in designing this UV illumination system inside the thermo-compression vacuum chamber.

![Figure 4-2 Thermo-compression tool in our laboratory [39].](image)

We had several iterations of the design before converging on the configuration that worked. In the first design we used a UV LED strip fitted around a ¼” thick quartz glass plate. As shown in figure 4-3 a SNFLS series 30 Side emitting LED flexible light strip is used and wrapped around a glass plate. This LED strip has 1.1W output power at 395nm. To increase the back reflection a folded aluminum foil is placed behind the glass plate. The SU-8 is coated on a 3inch x 2inch microscope glass slide and it is placed on the glass plate. Several drops of IPA were injected between the glass slide sample and the glass plate for increasing the coupling between two glass pieces. However, in subsequent nanoimprint tests we found that the power output from this UV
LED strip was far too low to produce sufficient exposure. The nanoimprint was conducted in a vacuum pressure at $8 \times 10^{-4}$ T, temperature at 95°C, tool pressure at 500Torr, for 10 minutes. Figure 4-4 (a) is the silicon mold, $3\mu m \times 3\mu m$ squares, after the nanoimprint. It is obvious that some SU-8 is stuck into the hole and not cross-linked. Figure 4-4 (b) is the imprinted SU-8. We believe this structure is a further evidence of insufficient UV exposure. The SU-8 is a viscous and sticky fluid before curing. We believe the SU-8 shown in figure 4-4 is not fully cured so that the imprinted structure is deformed and a part of SU-8 is stuck to the silicon stamp.

Figure 4-3 UV LED strip retrofitted to a glass plate.

Figure 4-4 Microscope image of silicon stamp after imprint (a) and the imprinted SU-8 (b).

In the second version we used three new high-power LEDs. The idea was to generate intense UV light from the top imprint membrane and use a scattering sheet behind the quartz plate to reflect and scatter the UV to the SU-8 sample. These LEDs were rated for 1W at 394nm each. The LED was embedded on a high thermal conductive package for heat diffusion. To further address the overheating problem, we used thermal paste to attach the LED package onto a thermoelectric
module. This product can stabilize the temperature of sensitive optical components such as high power LED. The maximum temperature difference between both sides was 65°C. We machined a thin titanium plate as a spacer and carrier between the top silicon carbide imprint membrane and the heater. Three LED/TEC combos were connected in series and clamped on the titanium plate by spring steel and screw fixed stand. A single LED/TEC combo and the whole illumination system are shown in figure 4-5.

![Figure 4-5 Module of LED package with clamping fixture (a) and the whole system (b).](image)

Additionally, we placed a cleanroom wipe at the bottom of the glass plate to increase the UV scatter. We spin coated a generic photoresist SPR955 on a silicon wafer to test the exposure performance. 15 minutes of exposure would fully cure the SPR955, but the SU-8 would require more exposure than the SPR955. A longer exposure would cause more overheating. The output power of the LED is a function of temperature, with higher temperature resulting in lower dose. Even the TEC could not significantly diffuse the heat generated by the LED. The overheating strongly decreases the efficiency of the nanoimprint and the high temperature may also lead to premature failure of these expensive LEDs. Therefore, the UV source had to be redesigned.

We designed and built a new stainless steel substrate plate to replace the original one and embed a new LED inside the substrate plate. This new design enables the UV light to directly reach the SU-8 film. We connected the LED and an external power supply through electrical feedthroughs. The UV LED in this design is a LZ4-00U600 LED. It contains four LEDs in series, providing
11W at 365nm. We attached the LED to the bottom plate with a thermal paste. The bottom plate temperature is air cooled by compressed air, which can effectively stabilize the LED temperature. A bead blasted glass was placed between the sample glass slide and the stainless steel plate for scattering to make the illumination uniform. Figure 4-6 is gives an overview of the in-situ UV illumination system.

Figure 4-6 High performance UV LED embedded into the stainless steel plate (a) with external power supply (b). The schematic of the UV illumination system fixture (c). The scattering glass slide made by sand bead blast for UV scattering (d).

We coated SU-8 on glass slide to measure and characterize the UV dose. Again we used the same silicon stamp for the exposure performance study. We used the same parameter listed in table 4-1 except for the imprint time reduced to 10 minutes. Figure 4-7 is the microscope image of the silicon stamp, 3μm × 3 μm squares, (a) and the imprinted SU-8 (b). Although partial SU-8 was still left on the stamp, most of the structure of the stamp was transferred onto the SU-8 film. A potential concern was whether the LED can sustain for a long periods of exposure. We continued
some experiments by increasing the exposure time and we found the temperature and the output power of the LED are both stable. This shows our in-situ UV illumination system can satisfied the requirement for long and stable exposure.

![Figure 4-7 Microscope image of the silicon stamp (a) and the imprinted SU-8 (b).](image)

4.3 Release agent

The release agent applied in our nanoimprint is the SigmaCote. This is a clear and colorless solution of a chlorinated organopolysiloxane in heptane. It reacts with the silanol group of the natural formed silicon oxide layer on the silicon surface and produces a neutral and hydrophobic microscopically thin film [40]. The SigmaCote is coated on the silicon stamp surface as the release agent.

Our silicon stamp is first cleaned in the AMI method and dried by nitrogen. After that we immerse the silicon stamp into the SigmaCote solution and the chemical reaction is immediately. The stamp is dried by nitrogen again and rinsed by DI water since SigmaCote reacts with water to produce HCl. The water bead is the key to determine whether the stamp is hydrophobic. We usually put the SigmaCote coated stamp on a hot plate at 130 degree C for 30minutes. This is to strengthen the thin film to be more durable.

We have mentioned in chapter 1 that the release agent plays an important role in the nanoimprint. Our experiments show that SigmaCote can satisfy the requirement. Figure 4-8 shows the SEM images of the stamp and the imprinted SU-8 pre-treated with and without SigmaCote, respectively. As we can see from the figure 4-8 (a) and (b), without SigmaCote the SU-8 stick to
the silicon stamp after the imprint and the imprinted structure is very poor. Figure 4-8 (c) and (d) show that the structure of the stamp is completely transferred to the SU-8 and no obvious SU-8 remains in the stamp.

![Comparison of nanoimprint with and without release agent (SigmaCote). SEM images of the silicon stamp after imprint without SigmaCote (a) and with SigmaCote (c). SEM images of the SU-8 after imprint without SigmaCote (b) and with SigmaCote (d).](image)

**4.4 Optimization for nanoimprint recipe**

We have optimized all the parameters in the nanoimprint, including SU-8 film thickness, imprint pressure, imprint temperature, exposure time, and imprint time.

Thickness of the SU-8 film is important in the nanoimprint. A film too thick would result in a long exposure for a sufficient dose while a film too thin cannot satisfy the requirement for a high aspect ratio structures. The thickness of SU-8 depends on the viscosity of the material and the spin coating speed. To make the desired structures with high aspect ratios, we have diluted the
SU-8 and figured out the optimum spin speed for spin coating the SU-8. The SU-8 in our experiment was diluted in a 1:5 ratio, 1 SU-8 2015:5 MICROCHEM SU-8 thinner. The diluted SU-8 was spin coated at 2000RPM on a cleaned 2inch ×3inch microscope glass slide. We have measured the thickness of the diluted SU-8. The SU-8 was patterned using photolithography and its thickness was measured by an Ambios XP-1 profiler (figure 4-9). The measurement is taken by a sharp tip stylus which is kept in contact with the sample and the sample is translated laterally. A laser is illuminates at the end of the stylus arm, and the intensity of reflection of the laser beam is measured as the stylus moves up and down. The resolution of the measurement can be down to 10 Angstroms. The diluted SU-8 is measured as 360nm thick. This thickness is desirable for short exposure and high aspect ratio.

![Image](image.jpg)

Figure 4-9 Ambios XP-1 profiler.

The procedure of the nanoimprint has been developed and listed below. We used double-sided tape to attach the stamp on the top imprint membrane. This is to hold the stamp so that the space between the stamp and the SU-8 can be vacuumed. The temperature is set to 135 degree Celsius. It is believed that the imprint temperature should be 60-70 degree higher than the glass transition temperature [41]. The stamp is heated to 135 C and the chamber is pumped down to $5 \times 10^{-4}$ Torr. After that the stamp is pressed to the SU-8 with 500 Torr for 10 minutes. This is to make sure the SU-8 is also heated by the stamp to 135 C. With this temperature and pressure the SU-8 becomes a liquid and completely reflows into the groove of the stamp. In Chapter 1 we have
introduced that the required nanoimprint time is a function of stamp geometry. Figure 4-10 is a SEM image of a poorly imprinted SU-8. The deformed structure can be attributed to insufficient reflow time. That means the time for the heated SU-8 to reflow into the grating structure is important. Our experiments show that it takes at least 10 minutes for the SU-8 to reflow for the nano-scale structure. The UV LED is then turned on. With desirable temperature and exposure, the cross-linking begins. This process takes 20 minutes to fully cross-link the SU-8. Finally, the chamber is vented and the imprint membrane with the stamp will be released at the same time. The optimized nanoimprint procedure is listed in figure 4-11.

![Figure 4-10 SEM image of poorly nanoimprinted SU-8.](image1)

**Figure 4-10 SEM image of poorly nanoimprinted SU-8.**

**Figure 4-11. Optimized procedure for nanoimprint.**

Based on the discussion above, we optimized the recipe of the nanoimprint shown in table 4-1.

```plaintext
Chamber pumped down to 5 \times 10^{-4} \text{Torr}

\downarrow

Stamp heated to 135 degree C and compressed to SU-8 with 500 Torr for 10 minutes

Continue imprint with UV exposure and the same pressure and temperature for 20 minutes

Vent and release SU-8 from the stamp
```
<table>
<thead>
<tr>
<th>Chamber pressure</th>
<th>$5 \times 10^{-4}$ Torr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tool pressure</td>
<td>500 Torr</td>
</tr>
<tr>
<td>Imprint temperature</td>
<td>95 degree Celsius</td>
</tr>
<tr>
<td>Exposure time</td>
<td>10 minutes</td>
</tr>
</tbody>
</table>

Table 4-1 Recipe of nanoimprint for release agent test.

Our experiments have shown the nanoimprinted structure is highly consistent with the structure on the stamp. Figure 4-12 shows the SEM images of the silicon stamps and the imprinted SU-8 samples. Our nanoimprint technique can completely transfer the structure even with the feature sizes on the order of 100nm. the imprinted sample will diffract white light from a fluorescent lamp and show a rainbow of colors as shown in figure 4-13.

![Figure 4-12 SEM image of silicon stamp of 220nm pitch structures (a) and the corresponding imprinted SU-8 (b).](image1)

Figure 4-13 Strong diffraction due to uniform structure.
4.5 Conclusion

In conclusion, we have developed a nanoimprint tool based on our thermo-compression bonder and self-customized in-situ UV illumination and employed it for imprinting SU-8 films using silicon stamps. The nanoimprint technique for fabricating high resolution periodic structures has been demonstrated. The optimized process procedure of nanoimprint for nano-scale structure is given. SEM images show that imprinted SU-8 structures are highly consistent with structures of silicon stamps which have feature sizes of 110nm.
CHAPTER 5
METAL WIRE FABRICATION

The last step of fabricating a wire-grid micro-polarizer is to build the metal wire. In this chapter, we will introduce three methods of metal wire fabrication. These three fabrication methods are: 1) Chemical Mechanical Planarization (CMP), 2) spiral nanorod growth, and 3) glancing angle deposition. A comparison of these three different fabrications will be given.

5.1 Chemical Mechanical Planarization Method

The principle of CMP is using corrosive and abrasive chemical slurry in conjunction with a polishing pad and a retaining pad to remove and even out the protruding and irregular topography [42]. Although our lab does not have a CMP tool, based on the principles we developed a similar polishing technique. Similar to the slurry, we used a diluted aluminum etchant as the catalyst to soften the metal and a grinding sheet, with grain size of 100nm, to perform the polishing. The combination of the diluted etchant and polishing sheet has the same function as the slurry. The process can be broken into two steps: metal deposition and chemical assisted planarization. As shown in figure 5-1, the first step is to deposit aluminum on the SU-8 imprinted surface, filling the grooves and tops. The subsequent polishing will planarize the surface to clean the redundant aluminum on top of the SU-8. This is known as the damascene process.

Figure 5-1 Schematic of CMP process, metal deposition followed by CMP.
We used RF sputtering using the Denton Vacuum Explorer 14 (figure 5-2) to deposit aluminum. We placed our sample into the chamber and pumped it down to $1 \times 10^{-6}$ Torr. Argon was the working gas, and the pressure was set to 4m Torr, and the RF power was set to 100w. The argon plasma is ignited by the cathode. The material to be sputtered is called the target. The material being coated is called the substrate [43]. The kinetic energy of the ion is delivered to the aluminum target atom. The ejected target atom will then land on the substrate to form a thin film.

In the earlier experiments as shown in figure 5-3 (a), we found that the aluminum was deposited in large grains, which prevented them from filling the grooves of SU-8. This is also unfavorable for the subsequent planarization step since the layer of aluminum was found to be easily peeled off from the SU-8 surface as shown in figure 5-3 (b). The main question here is how to modify the deposition method to produce aluminum with fine grains into the grooves of SU-8. In the polishing process, one of the unknown factors is the relative difference in hardness between aluminum and SU-8 so that the aluminum can be planarized without destroying the SU-8 film. We expect the diluted chemical etching will assist the planarization since the aluminum will be softened but the SU-8 is resistant to the aluminum etchant.

Figure 5-2 Denton Vacuum Explorer 14
Figure 5-3 SEM image of aggregation of aluminum particles (a) and an aluminum layer is peeled off (b).

To solve the problem of large aluminum grains, we then switched to electron beam evaporation (figure 5-4) to deposit the aluminum on SU-8 samples. This change allowed the aluminum layer to fill into the grooves of SU-8 as shown in figure 5-5 (a). Some of the aluminum was deposited into the groove of the SU-8 and others were deposited on the top of the groove. This change of aluminum grain size is due to their different deposition mechanisms. E-beam evaporation deposition is a thermal evaporation process. It uses an electron beam to heat an aluminum pellet and increase its vapor pressure so that it can condense on the substrate as a thin film. We placed our imprinted sample into the chamber and pumped it down to 0.5 μT and deposited 100nm aluminum.

Figure 5-4 Torr E-beam Evaporator
As we can see from the SEM image in figure 5-5 (a), the aluminum was evenly distributed on the imprinted SU-8 structure, filling the grooves and the tops. This structure was favorable for the later polishing. After the metal deposition, we diluted the aluminum etchant and used the grinding sheet for planarization. We diluted the aluminum etchant in a 1:20 ratio with DI water. This ratio makes the etchant act as catalyst to soften the metal instead of dissolving the aluminum. The SEM image in figure 5-5 (b) shows a typical profile of the polished sample. It shows the SU-8 mechanically hard enough to survive during the polishing as we expected. The surface is flattened but there is still a thin layer of aluminum covering the top of the SU-8. The main difficulty in this process is the determination of the end-point. That is because there is nothing we can infer from the sample visually during the removal process. It is nearly impossible to control the removal rate since it was a function of mechanical pressure, etchant concentration, and polishing speed. The lack of process control due to these unknowns makes a uniform polishing very difficult to achieve.

![SEM image of aluminum deposited on the imprinted SU-8 sample by e-beam evaporation (a) and the polished SU-8 sample (b).](image)

5.2 Spiral nanorod growth

Another method to accurately control the deposition of the metal wire structure is spiral nanorod growth. During the normal deposition, the metal growth direction is vertical to the substrate surface. When the substrate is deposited at an oblique angle, the photoresist grating acts as shadow masks and the metal will grow at the oblique angle on the grating structures.
As shown in figure 5-6 (b), the side wall and the top will get coated and the bottom will be uncoated. The oblique angle can be optimized for each structure based on its aspect ratio. The aspect ratio of the imprinted structure is the tangent of the oblique angle. Furthermore, by rotating the substrate we can produce the result as shown in figure 5-6 (c). In other words, the metal will grow around the protruding structures, and it forms a metal cladding around each photoresist grating line.

To perform the rotating deposition at a glancing angle, our group members designed and built a glancing angle substrate holder that can be rotated by a vacuum-compatible electric motor. As we can see from figure 5-7, a 2 inch steel plate is attached to the motor shaft and the motor is fixed to an adjustable arm. The angle of the substrate can be adjusted from 90 degree to 65 degree. The motor is energized by an external DC power supply connected through electrical feedthroughs. The rotating speed can be controlled by the voltage.
Figure 5-7 Motor based fixture for deposition in the e-beam evaporation chamber.

In this process we used an angle of incidence of 85 degrees. The power supply voltage was set to 2.5V to produce 1rpm, and the thickness of the metal thickness was set to 300nm. We chose chromium as the metal because it has very low transmission over a broad spectrum, from 200nm to 1000nm. It also has high adhesion to the substrates and the straight structure is relatively easier to grow on the sample. Figure 5-8 shows an SEM image of the metal structure after deposition at the oblique angle of 85 degrees. The metal on top of the imprinted SU-8 has columnar structures of nanorods, which is the expected results from glancing angle deposition [44]. The rotation enables the nano-rods to grow straight. Therefore, the combination of rotation and glancing angle builds the spiral nano-rod structure.

Although the straight structure is favorable, in later measurement we found the extinction ratio of this polarizer was very low, near 2:1. This is unexpected because based on the simulation results from the thin film design software, Openfilter, a 250nm thick chromium layer should have transmission lower than 0.001%. However, the transmission of the spiral nanorod structure we measured was beyond 30%. We believe the high transmission is due to the loosely packed nanorod structure as shown in figure 5-8 (b). Now, the question becomes how to grow a similar straight structure and avoid the nano-rod structures.
Figure 5-8 SEM images of chromium rotating deposition at a glancing angle. Cross-section images (a), and top view image (b).

To avoid the nano-rod formation, we decreased the deposition angle to 68 degrees. Nano-rod structures become denser as the angle decreases. Figure 5-9 is the SEM image of a structure formed by using the rotating deposition method at the oblique angle of 68 degree. We found that the metal columns were growing larger connecting adjacent columns together. This is an unfavorable condition that will strongly decrease the transmission of TM waves, which is an important criteria to determine the performance of a polarizer. We can decrease the metal thickness to avoid the metal wires from connecting, but the thin metal layer will result in a low extinction ratio. Therefore, the structure made by the rotating glancing angle deposition does not satisfy our requirements.

Figure 5-9 SEM images of chromium rotating deposition at a small glancing angle.
5.3 Glancing angle deposition without rotation

Metal deposition at an oblique angle without rotation is also an alternative to fabricate the metal wires. By choosing an optimum angle, the nanorod structure can be avoided. The expected metal wire is similar to figure 5-6 (b). Again, we used the motor based fixture to hold the sample at an angle without rotation. The maximum glancing angle is determined by the aspect ratio of the imprinted structure. Figure 5-10 is the SEM image of the sample. Compared to figure 5-8 (a), figure 5-10 (a) shows the metal wires are thin film instead of nano-rod, and it shows the uniformity of the structure. Figure 5-10 (b) shows there is a distinct separation between each metal wire.

![Figure 5-10](image)

Figure 5-10 SEM image of metal deposit on imprinted SU-8 at the angle of 30 degree.

We have improved the nanoimprint quality and increased the metal thickness. We increased the deposition thickness, which would give lower transmission of TE waves. As we can see from the SEM images of figure 5-11 (a), the aluminum is a desirable solid thin film. The topography shown in figure 5-11 (b), (c), and (d) shows the uniformity of the imprinted structure and the consequent deposition. Due to the oblique deposition, the metal layer covers the top and right side of the imprinted SU-8. This changes our original design that we purposed the metal would be only on top of the SU8 and the fill factor of the metal wire is not the same as the imprinted SU-8. However, in our simulation we found the increasing fill fact would provide higher polarization extinction ratio. We will discuss the influence of fill factor in the following chapter.
Figure 5-11 SEM image of metal deposit on imprinted SU-8 at the angle of 70 degree. Cross-section images (a). Top view images (b), (c) and (d).

5.4 Conclusion

What we can conclude based on the SEM image is that, among three fabrication methods, the glancing angle deposition produces the best structure. It was difficult to determine the end of the polishing by referring the SU-8 when we used the CMP method. In the spiral nanorod method, the loose nanorod structure is not able to completely reflect the TE wave, which results in a low extinction ratio. Comparatively, the glancing angle deposition gives the most desirable structure. The aluminum thin film has low transmission in a broad spectrum. The solid aluminum thin film has an aspect ratio larger than 1 and this relatively high aspect ratio metal wire structure results in the extinction ratio In addition, this structure can be easily fabricated in a time and cost saving manner.
CHAPTER 6

COMSOL SIMULATION AND THE OPTICAL PERFORMANCE MEASUREMENTS

6.1 COMSOL simulation

The polarization extinction ratio (PER) and the TM wave transmission are the two important criteria for a polarizer. To better understand the influence of the structure on the optical performance, we used COMSOL Multiphysics version 4.1 to simulate the PER and TM transmission of our design. The design of our grating structure can be mainly broken into three parts: 1) the metal wire structure, 2) the imprinted SU-8 structure, and 3) the substrate glass slide. In the simulation, the thickness and the optical properties of the glass slide are constant so we focus on the structure of metal wire and the structure of imprinted the SU-8. An idealized one period structure is shown in figure 6-1. The fill factor of the imprinted SU-8 is 50% and the metal is on top of the SU-8 grating. The SU-8 is on the glass substrate and the metal wires and half imprinted SU-8 are exposed to the air. The glass thickness is set to 2mm and the air layer is set to 2mm. Based on the SEM of the imprinted SU-8 structure, the SU-base 8 is set to 800nm thick and the imprinted SU8 is set to 250nm thick. We used these data in all the simulations. Since SU-8 is a highly transparent material, the period, metal thickness, and the fill factor of the metal wire become the most important three factors to determine the optical performance of our micro-polarizer.
Our simulation is first carried out to examine the influence of the period and metal thickness to the PER. The incident wavelength is set to 633nm. Our simulated results are highly consistent with simulated results using the rigorous-coupled wave analysis developed by Zhi Wu [36]. The simulated results are listed in table 6-1. As we can see from the table the PER is not simply linearly proportional to either the period or the metal thickness.

<table>
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<td>2.28</td>
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<td>3.07</td>
</tr>
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</table>

Table 6-1 The simulated PER corresponding to the metal wire thickness and period.
We are also interested in the dependence of PER with metal wire fill factor. As we can see from the SEM images in figure 5-11, the metal is not exactly on top of the SU-8 grating but also on the right side wall. To better simulate the structure, the structure in our simulation is shown in figure 6-2. Compared to the metal wire structure in figure 6-1, the metal covers the right side of the imprinted SU-8. We change the fill factor from 0.5 to 0.7. We set the incident wavelength to 633nm, period to 375nm, metal thickness to 180nm, and imprinted SU-8 line width to 160nm. The simulated PER are shown in figure 6-3. It shows that the PER is proportional to the metal wire fill factor, larger fill factor corresponding to higher PER. However, our further simulations show that TM transmission is not proportional to the metal wire fill factor. Therefore, in our metal wire-grid design, we should take the TM transmission into consideration while increasing the metal fill factor for a larger PER.

Finally, we used the data measured for the size of the structure shown in figure 5-11 to simulate the optical performance of the sample. We set the incident wavelength to 1064nm, period to 400nm, metal thickness to 180nm, metal wire fill factor to 0.7, and SU-8 line width to 176nm. The simulated PER are consistent with the measured result shown in the following section.

![Figure 6-2 Schematic of an actual Al wire/SU8/glass structure.](image)
6.2 Optical performance measurements

Polarizer extinction ratio (PER) is defined as the ratio of optical powers of perpendicular polarizations, Extinction Ratio $= \frac{\text{Power}_{\text{Transmitted TM}}}{\text{Power}_{\text{Transmitted TE}}}$, which is a significant criterion to determine the optical performance of polarizers. Based on the definition, we setup a measurement system to measure the PER of our polarizers. The experiment setup was established by Shiyi Wang in Dr. Qiwen Zhan’s research lab. A photo of our experimental setup is shown in figure 6-4 (a). We used a Neodymium doped YAG laser as the light source, which can output 1064nm. Since we used a Spiricon CCD camera to capture and measure the intensity of the transmitted light from our polarizer, an attenuator was used and a spatial filter was placed after the attenuator to decrease the intensity and clean up the laser spot. A linear polarizer was placed after the laser to further polarize the light source. A half-wave plate was placed after the polarizer to adjust the polarization direction to our sample which was fixed before the CCD camera. The CCD camera was connected to a computer and we observed the transmitted spot and measured the intensity with the Spiricon software. Since our sample was perpendicular to the laser beam, there was only one polarization direction on our sample, as shown in figure 6-4 (b). When we rotate the half-wave plate, the ratio between the highest and the lowest measured power is the PER. We

![PER vs. Fill factor](image)
measured the maximum and minimum transmitted power with our sample for the PER. The sample we measured is shown in figure 5-11. The PER was measured to be 90:1.

![PER Measurement Configuration and Schematic](image)

Figure 6-4 A photo (a) of the PER measurement configuration and a schematic of laser polarization on sample (b).

The 90:1 extinction ratio of our polarizers in near infrared region is acceptable. It is easy to find that the commercialized holographic wire grid polarizers have achieved an extinction ratio of 300:1 [45]; the nanoparticle linear film polarizers have achieved 100000:1 [46]; and Glan-Thompson Polarizers have achieved 1000000:1 [47]. However, regular polarimetric imaging would require at least three orientations of wire-grid and another one for increasing the extinction ratio. These commercial polarizers are either centimeter scale bulks or sheets which cannot be fabricated in pixel size with different orientations for CCD or COMS cameras to obtain real-time imaging. Therefore, the polarization extinction ratio of our polarizer is acceptable.

### 6.3 Conclusion

In this chapter, we simulated PER using COMSOL and the results are consistent with the results simulated by RCWA as well as the measurement results. The fabricated wire-grid micro-polarizer has a PER of 90:1 in 1064nm, which is acceptable for the near infrared polarimetric imaging applications.
CHAPTER 7
CONCLUSION AND FUTURE WORK

We have developed a technique for fabricating wire-grid micro-polarizers using nanoimprint on SU-8 thin film and glancing deposition of metal thin films. This technique allows us to utilize silicon’s processing advantages such as high-resolution lithography and deep etching to realize high-aspect ratio metal wiregrids on a transparent substrate. The resulting wire grids also have high aspect ratios and consequently high extinction ratios.

Compare to other fabrication methods using e-beam lithography and CMP, the techniques we used in our research greatly improve the fabrication efficiency. The interference lithography can create the periodic pattern for grating pattern in few seconds. The nanoimprint technique can quickly transfer the pattern to a resist film with a stamp and the stamp is reusable. Finally, the glancing angle deposition for the metal wire fabricated is more convenient than regular damascene process such as CMP.

Our future research will focus on wire-grid polarizers for the visible spectrum. To obtain high extinction ratios, we propose to use chemical vapor etching for the metal wire fabrication. The principle is similar to CMP. We will use plasma etching to planarize the metal surface. The etching selectivity between SU-8 and aluminum using BCl3 and Cl2 will be studied.
REFERENCES


[42] Zhengfeng Wang, Yin Ling, Ng Sum Huan, Teo Phaik Luan, “Chemical Mechanical Planarization,” SIMTech Technical Report (PT/01/003/JT)


