Fabrication of Plasmonic Nanostructures using Electron Beam Lithography

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Abstract

The need for more sustainable sources of energy has become apparent in recent years. There has been much research pertaining to photovoltaic technology, not only in areas of increasing the efficiency of solar cells themselves, but also into methods of coating the cells with various materials in order to increase absorption and efficiency. One such method is the fabrication of plasmonic nanostructures on the surface of cells to reduce reflectivity and increase absorption. This project investigates fabrication of plasmonic nanostructures on silicon using electron-beam lithography. Dot array patterns were exposed consistently in resist. Silver evaporation and lift-off were successful and resulted in arrays of nanostructures, but the quality was not consistent. Silicon wafers either retained too much silver in areas surrounding the dot arrays and appeared dirty, or too much silver was removed and the nanostructures were fragmented. Further work might investigate ways to attune the lift-off process and therefore fabricate consistent arrays that are clean with nanostructures that are whole.
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Table of Contents

Abstract ........................................................................................................................................... 3
Acknowledgements ........................................................................................................................ 4
Chapter 1: Introduction .................................................................................................................. 6
  1.1. Motivation ............................................................................................................................ 6
  1.2. Solar Cells ............................................................................................................................. 7
  1.3. Plasmonics ............................................................................................................................ 8
  1.4. Prior Work ............................................................................................................................ 9
Chapter 2: Theory ........................................................................................................................ 11
  2.1. Solar Cells ........................................................................................................................... 11
  2.2. Plasmonics .......................................................................................................................... 13
  2.3. Lithography ......................................................................................................................... 15
Chapter 3: Experimental Methods and Design ........................................................................... 18
  3.1. Materials and Equipment ................................................................................................... 18
  3.2. Methods ............................................................................................................................. 18
  3.3. Design ................................................................................................................................ 21
Chapter 4: Results ....................................................................................................................... 22
  4.1. Lithography ......................................................................................................................... 22
  4.2. Evaporation ....................................................................................................................... 23
Chapter 5: Conclusions ................................................................................................................ 25
  5.1. Summary of Results .......................................................................................................... 25
  5.2. Future Work ....................................................................................................................... 25
References ..................................................................................................................................... 27
Chapter 1: Introduction

1.1. Motivation

Dependence on fossil fuels presents us with a complex assortment of global problems. Diminishing reserves threaten us with economic catastrophe and destabilization unless we make proactive steps toward a transition to renewable energy sources. In addition, the dangers of climate change caused by greenhouse gases released by the burning of these fuels make it critical that we improve renewable energy technologies. Photovoltaics are one such technology, and with 174,000 TW of power being delivered to us by the sun, solar power clearly has the potential to produce more than enough energy to sustain our current consumption of 17 TW. However, it is currently responsible for a mere 1% of global energy production.

The price of solar cells has been steadily decreasing as efficiencies have improved, though the most common PV technologies in use today are still relatively costly and resource intensive due to the way high-quality silicon cells are manufactured. Many cells in development make use of alternate materials and methods that allow for much thinner cells. These cells vary widely in their efficiency, depending on the type, from 12% for certain thin-film cells to over 40% for multijunction cells (Figure 1.1). Organic thin-film solar cells show great potential due to their potential low cost and rapid gains in efficiency. These cells are still much less efficient than traditional PV, but there are clear ways to improve this. One such method is to fabricate plasmonic nanostructures on the cell surface. These increase light absorption by reducing the amount of reflected or transmitted light, and while this can be done on any type of solar cell, it is most advantageous to find ways to improve inexpensive types like thin film cells.
1.2. Solar Cells

A typical solar cell consists of n-type and p-type silicon layers sandwiched between electrical contacts. The n-layer has some of the silicon atoms replaced with elements with one additional valence electron, and these electrons act as negative charges that are free to move in the silicon. This is the same for the p-layer except that the silicon is doped with an element with one less valence electron, acting as positively charged ‘holes’ that are also free to move about the silicon. The boundary between the n- and p-layers is called the pn junction. The electrons and holes will move together and cancel each other out, creating a depletion zone that no longer has free charge carriers. This exchange in charge also creates an electric field across the depletion zone. When a photon strikes the silicon, the energy will excite an electron, leaving a hole, creating what is called an electron-hole pair. The electric field will then separate the electrons and holes. If there is an external load connected to the electrodes of the cell, then the
buildup of charge will drive electrons through the circuit, and this current can be harnessed to generate electricity (Figure 1.2). (Kasap)

![Figure 1.2. Basic solar cell function.](http://www.electricaltechnology.org/2015/06/how-to-make-a-solar-cell-photovoltaic-cell.html)

1.3. Plasmonics

Plasmonic nanostructures are metal particles with diameters on the order of tens of nanometers. They assist in the excitation of free conducting electrons by incident light waves, resulting in collective oscillations referred to as surface plasmons (Barnes). This property can be utilized for a variety of applications, including optics, sensors, and data storage, but relevant to this project is the application to solar cells.
These nanostructures can increase the efficiency of solar cells in two ways. First, certain properties of the nanostructures allow them to scatter light from relatively large surrounding areas; due to the properties of the air and the silicon, light scattered by the nanoparticles in this way is preferentially scattered into the silicon, enhancing light absorption by the energy-generating portion of the cell. Second, these nanoparticles have strong fields around them which essentially store energy from incident light. This energy increases the amount of electron-hole pair creation around the nanoparticle, which contributes to the current through the solar cell and the generation of electricity. (Catchpole)

1.4. Prior Work

1.4.1. A Brief History

Plasmonics emerged as a field in the early 1900s, with R. W. Wood’s observations of uneven light distribution by diffraction gratings. In particular, when polarized light passed through the grating at certain angles of incidence the patterns were extremely well-defined. These anomalous diffraction gratings could not be accounted for with classical theory. (Wood) Research began in earnest in the 1950s with the work of Ritchie, which brought the cause of Wood’s results, surface plasmons, into the field of surface science. Research into the properties of these surface plasmons and applications of plasmonic nanostructures on solar cells has emerged in the last several years as ways to increase the efficiency of cells without large increases in cost.

1.4.2. Prior implementation on solar cells

Other labs have experimented with the application of plasmonics technology to solar cells. Various shapes of nanostructures have been fabricated, including concentric circles as well as arrays of dots similar to what I plan to fabricate (Chiu). An oft targeted problem is the absorption of light by the nanostructures themselves, rather than being scattered into the substrate. This phenomenon, known as parasitic absorption, can reduce efficiency by absorbing energy into the metal and reducing the amount of light that can reach the conducting layer and
generate electricity. The shape and material of the nanostructure has been found to be crucial in determining the amount of this absorption.

The Tanenbaum lab has had minimal plasmonics fabrication experience. Victoria Kim was able to successfully expose stencil-like patterns in resist, but was unable to use those to create arrays of silver nanoparticles (Kim). This project seeks to build on this prior experience in order to successfully fabricate and test plasmonics in the lab.
Chapter 2: Theory

This chapter seeks to explore the properties of solar cells and plasmonic nanostructures and how they relate to each other. This chapter also discusses benefits and drawbacks of different methods of lithography, as well as some of the issues that must be taken into account when using electron-beam lithography.

2.1. Solar Cells

The pn junction is the boundary between an n-layer and a p-layer. n-type and a p-type materials are silicon doped with elements with one more and one less valence electron, respectively. The extra electrons in the n-layer act as free electrons that can move in the silicon. The electron shortage in the p-layer essentially acts as ‘holes’ which can also move around. When these two layers are put together, the free electrons will move to fill the holes, creating a net negative charge on the p-layer and a positive charge on the n-layer. This creates an electric field perpendicular to the boundary (\(E_o\) in Figure 2.1), resulting in a voltage called the built-in potential. Electrons will stop traveling across the pn junction when the electric field is strong enough to prevent further diffusion of electrons. This field also causes remaining free charges to be concentrated at either end of the material, outside the junction. The depletion zone is the charge carrier-free area surrounding the pn junction (between the dotted lines in Figure 2.1).

*Figure 2.1. Free charge carriers move across the depletion zone, resulting in an electric field \(E_o\).*

A basic silicon solar cell consists of an n-layer and a p-layer sandwiched between electrodes, with a thin anti-reflection coating on the surface. When photons strike the silicon, an electron is excited and an electron-hole pair is created. If this pair is created in or near the depletion zone, the built-in potential will force the electron to the n-side and hole to the p-side. This results in the n-side becoming negatively charged and the p-side positively charged, creating an open circuit voltage between the terminals. If a load is connected, electrons will flow from the n-layer to the p-layer, do work, and fill a hole.

Long-wavelength photons are absorbed in the neutral p-side of the cell photogenerate electron-hole pairs that diffuse as if there is no electric field (Figure 2.2). The electron diffuses a mean distance \( L_e = \sqrt{2D_e\tau_e} \) before recombining with a hole, where \( D_e \) is its diffusion coefficient in the p-side and \( \tau_e \) is the recombination lifetime. Electrons within \( L_e \) of the depletion region can readily reach it and be pulled by the built-in electric field to the n-side. Electrons further than \( L_e \) will recombine with a hole before reaching the depletion zone. This phenomenon is also important for holes on the other side of the depletion zone. Holes generated by short wavelength photons within diffusion length \( L_h \) of the depletion zone will be swept to the n-side electrode. There is a resulting volume of thickness \( L_h + W + L_e \) in which

\[\text{Figure 2.2. Different wavelengths of light generate electron-hole pairs at different depths in the cell.}\]

photons can be absorbed and contribute to the photovoltaic effect. If the terminals are shorted or a load is connected, then the flow of electrons resulting from the incident photons is called the photocurrent.

Crystalline silicon has a bandgap of 1.1 eV. This corresponds to a threshold wavelength of 1.1 um, meaning that incident energy from photons with wavelengths greater than 1.1 um does not form electron-hole pairs and is wasted. This is approximately 25% of the incoming energy. The other major loss is from high energy photons. These photons create electron-hole pairs very near to the surface of the cell which recombine before they can reach the depletion zone.

2.2. Plasmonics

The addition of plasmonic nanostructures to solar cells can increase the efficiency by reducing the reflectivity of the surface of the cell as well as increasing the amount of light that remains trapped in the cell.

Plasmonic nanostructures support the propagation of surface plasmons, which are the excitations and collective oscillations of free conducting electrons at the boundary between a metal and a dielectric. If nanostructures are fabricated with the appropriate size and shape they can trap light in the cell, increasing the path length of light and thus the rate of absorption, since light spends more time in the cell. Both localized surface plasmons excited in the nanostructures and surface plasmon polaritons at the interface of the metal and the dielectric are relevant. (Atwater)

This allows for an increase in efficiency in multiple ways. These nanoparticles can scatter sunlight into the semiconductor and trap it there, effectively folding the beam into the semiconductor layer (Figure 2.3), causing the light to travel a longer distance and have a higher chance of being absorbed. This is due to scattering properties of a metal nanostructure at the boundary between two dielectrics. When light strikes such a structure, the light scatters preferentially into the material with the higher permittivity. Silicon and air have relative permittivities of approximately 12 and 1 respectively, so in the case of a structure on the
surface of a silicon solar cell, incident light will scatter into the cell. This anisotropic scattering means that the nanostructures also function as an anti-reflection coating by decreasing the amount of light that is reflected off of the surface and unable to contribute to the generation of electricity. (Spinelli)

For nanoparticles with diameters well below the wave length of incident light, the scattering and absorption can be described as follows:

\[ C_{scat} = \frac{1}{6\pi} \left( \frac{2\pi}{\lambda} \right)^4 |\alpha|^2, \quad C_{abs} = \frac{2\pi}{\lambda} \text{Im}[\alpha] \]

where

\[ \alpha = 3V \left[ \frac{\varepsilon_p/\varepsilon_m - 1}{\varepsilon_p/\varepsilon_m + 2} \right] \]

is the polarizability of the particle. Here \( V \) is the particle volume, \( \varepsilon_p \) is the dielectric function of the particle and \( \varepsilon_m \) is the dielectric function of the embedding medium. The surface plasmon resonance is the collective oscillation of electrons at the boundary between the silver and the silicon, and occurs when the polarizability is very large. This is the case when \( \varepsilon_p = -2\varepsilon_m \). The scattering cross section of the particle is maximized at the plasmon resonance. Silver nanoparticles at resonance have a scattering cross-section approximately ten times the size of the particle (Catchpole).
Another way that these nanostructures can increase efficiency is through the strong local field enhancement around the nanoparticles. This is the most effective for small particles around 5-20 nm in diameter. The nanoparticle acts as an antenna that stores the energy from incident photons in a localized surface plasmon mode. This leads to increased electron-hole pair creation.

2.3. Lithography

Lithography is the process of transferring patterns from one medium to another, such as from a digital format to an exposure on a silicon wafer. Electron-beam lithography has an extremely high resolution limit, making it useful for nanostructure fabrication applications.

There are two methods of e-beam lithography: projection printing and direct writing (Figure 2.4). Projection printing uses a thin mask or stencil to print a set image in a single exposure. The electron beam passes through the mask and scatters the electrons to varying degrees depending on the properties of the mask at each point. The mask acts as a stencil, letting electrons pass through unhindered if in an area to be exposed, and scattering the rest. The
beam then passes through an aperture that filters out the highly scattered electrons, allowing a high contrast image to be printed on the substrate.

In the direct writing process, the method used in this thesis, the pattern is written one pixel at a time. The beam is focused directly on the surface of the substrate, which allows for extremely fine pattern writing with features as small as 10 nm. Since no projection setup and mask are needed, direct writing is the most useful process for the fabrication of micro- and nanostructures. However, because direct writing works by exposing one pixel at a time, it is a relatively slow process and thus not adequate for mass production applications. Instead it is important in prototyping, maskmaking, fabrication of small numbers of special products, and research and development for advanced applications.

The lift-off process is the most popular direct writing process, and involves coating the substrate with resist, exposing the pattern, and developing the exposure (Figure 2.5). A resist such as polymethyl methacrylate (PMMA) is a substance that breaks down when exposed to a beam of electrons. This is what allows precise patterns to be written with the electron beam in the exposure process. A developer such as methyl isobutyl ketone (MIBK) is used to wash out the exposed resist, and then a layer of a metal such as silver it deposited on the substrate. The last step of the lift-off process is to soak the substrate in a solvent bath to remove the remaining resist and unwanted metal.

![Figure 2.5. E-beam lithography and liftoff process](image)

*Victoria Kim, Fabrication of Plasmonic Nanostructures using Lithography, Scripps College, 2015*
PMMA is a positive resist, which means that exposure to electrons breaks down the resist so that it is removed by a developer. This is in contrast to a negative resist, which is strengthened by the electron radiation, and the unexposed areas are what is removed by the developer. PMMA has very high resolution, demonstrated to be under 10 nm. This makes it a good choice for use in the nanostructure fabrication process. The drawbacks of PMMA include relatively low sensitivity, poor dry-etch resistance, and moderate thermal stability.

One issue in electron beam lithography that must be taken into account is the proximity effect. When electrons in the beam strike the resist or the underlying substrate, they experience small-angle forward scattering, widening the beam slightly. As they continue through the resist, some electrons scatter at large angles leading to backscattering, in which the electrons travel through the resist far from the intended exposure area. This causes additional exposure and is called the proximity effect, the strength of which is dependent on the voltage used to accelerate the electron beam (Figure 2.6). As the electrons from the beam slow down, much of their energy is dissipated to secondary electrons, a small number of which have high energies on the order of 1 keV. These ‘fast electrons’ are responsible for much of the actual exposure of the resist. (Tseng)
Chapter 3: Experimental Methods and Design

3.1. Materials and Equipment

3.1.1. Substrate and Resist

Silicon wafers are used as the substrate on which to research nanostructure fabrication because it is similar to fabrication on a silicon solar cell, but more costly solar cells are not used up while determining ideal parameters for consistent fabrication.

The resist used during pattern generation is polymethyl methacrylate (PMMA), diluted to 4% in anisole. Methyl isobutyl ketone (MIBK) diluted 1:3 in IPA is used as the developer, removing resist that has been exposed to the scanning electron beam, as well as to remove PMMA/ excess silver after evaporations.

3.1.2. Microscopes

The Atomic Force Microscope (AFM) is used to determine PMMA and silver thicknesses, and the Hitachi SU-70 Scanning Electron Microscope (SEM) is used for lithography and for examining samples post-evaporation. Two programs are used for lithography: DesignCAD LT is used to design the patterns that will be exposed on the sample. The Nanometer Pattern Generation System software imports DesignCAD files and controls the electron beam in order to expose the patterns.

3.1.3. Evaporator

The evaporator consists of boats which hold the silver pellets, a movable shutter that blocks evaporation onto samples until desired, and a support platform which holds the samples above the boats. Everything is enclosed under a bell jar, which is vacuum pumped during evaporation runs to lower the evaporation point of the silver.

3.2. Methods

3.2.1. Sample Preparation

To prepare a sample for plasmonics fabrication a silicon wafer was cut to approximately 15x20 mm, using a diamond scribe and a pair of tweezers. The sample is placed in a spin coater
and cleaned with acetone followed by isopropyl alcohol (IPA). The sample is then coated with 3-4 drops of 4% PMMA in anisole, and is spun at 7000 rpm for 40 seconds, bringing the thickness to approximately 140 nm. The sample is then baked on a hot plate at 180°C for 60 seconds. Guidelines are then scratched onto the surface by using tweezers to scrape off thin lines of resist down the middle of the sample (Figure 3.1). These mark locations for pattern exposure.

![Resist-coated sample with scratched guidelines, set up in SEM sample holder.](image)

*Victoria Kim, Fabrication of Plasmonic Nanostructures using Lithography, Scripps College, 2015*

### 3.2.2. Lithography

The sample is placed in the SEM and the beam is focused on the edge of the sample. The guideline scratches are then followed and the end points saved so they can be accessed quickly during pattern generation. The beam blanker for the SEM is then turned on and set to be controlled by the NPGS software. A guideline edge location is selected, and the NPGS runs an exposure 1 mm to the side, so as not to create a pattern on an area that was exposed while following the guidelines. This is repeated for each saved location.
The pattern used was sixteen 20x20 arrays of dots, as shown in Figure 3.2. Point doses ranging from 15-90 fC were tested. Beam current was typically measured to be 400 pA, though it was occasionally found to be as low as 140 pA or as high as 600 pA.

After running all exposures, the sample is removed from the SEM and placed in an MIBK:IPA developer for 30 seconds and then swirled in DI water for 30 seconds.

![Figure 3.2: CAD file of dot arrays. Scale bar is 7.7 µm.](image)

3.2.3. **Silver Evaporation and Lift-off**

A glass slide is cleaned in the spin coater with acetone and IPA. After pattern development, one or two samples are attached to the glass slide with copper tape. The slide is then placed in the center of the evaporator, and a silver pellet is placed in one of the boats.

After pumping the evaporator chamber down to a pressure of $1.0 \times 10^{-6}$ torr, the current through the boat is slowly increased to about 120 A. The shutter is then opened, and the silver is allowed to evaporate onto the sample for 2.5 minutes, resulting in a silver film thickness of approximately 50 nm. The sample is allowed to cool, and is then removed from the evaporator and from the glass slide.
Liftoff is performed by placing the sample in MIBK at 90°C until all PMMA and excess silver have been removed. The sample is then rinsed in DI water and blown dry with compressed CO₂.

3.2.4. Quality Characterization

The sample is then examined in the SEM to determine the quality of the dot fabrication, and in the AFM to determine the height of the dots. The area should be clean and clear, similar to those in Fig 3.3. The dots should be 50 nm tall and 100 nm in diameter, at a pitch of 380 nm.

3.3. Design

3.3.1. Metal

Silver was chosen for the nanostructure material because for dots over 70 nm in diameter, silver has the lowest absorption cross-section efficiency of comparable metals (Hylton et. al.). This means that silver will absorb very little light in a way that prevents it from generating electricity. Silver was also used in previous plasmonics research in this lab, so using silver made it easier to incorporate existing knowledge.

3.3.2. Pattern

The dot pattern used was 42 µm across. A dot array is an efficient use of space on the surface of the cell. This pattern leaves more space open than is covered, allowing light to both hit the cell directly as well as scatter off the nanostructures into the cell.
Chapter 4: Results

4.1. Lithography

The resist is consistently 140-150 nm thick when a silicon wafer is spin coated with 3-4 drops of 4% PMMA at 7000 rpm for 40 seconds. This thickness is ideal because a small amount of resist will be removed in the exposure and development process, and the holes must be deeper than the desired height of the nanostructures — roughly 50 nm — in order to have successful silver lift-off.

I was able to consistently develop dot arrays in the resist. I found the optimal dosage to be between 47-62 fC/dot. The beam had an accelerating voltage of 5 kV and a current of 400 pA. The exposure was done at 1900x magnification. An accelerating voltage of 2 kV was also used, but the exposures were not well-defined, or the exposures resulted in holes that were connected to each other vertically. An AFM scan of the exposures in the resist is shown in Figure 4.1.

![AFM image of dot array exposure in resist. Scale bar is 7 µm.](image)

Figure 4.1. AFM image of dot array exposure in resist. Scale bar is 7 µm.
4.2. Evaporation

I was able to consistently evaporate a layer of silver 50 nm thick. This was achieved when the current through the boat reached 120-140 mA (when the silver pellet had visible surface evaporation) and then opening the shutter for 2 minutes 30 seconds. An SEM image of a dot array pre-lift-off is shown in Figure 4.2.

![Figure 4.2. Dot array following silver evaporation, prior to lift-off.](image)

Though the silver evaporation was consistent, I have not been able to determine the optimal parameters for successful lift-off during my time working on this project. Many samples were not clean following development, though the dot structures are apparent (Figure 4.3). Other evaporations were clean, but each nanostructure was in several pieces rather than a solid particle (Figure 4.4).
Figure 4.3. Nanoparticles following lift-off. The sample has an excess of residual silver surrounding the patterns following development.

Figure 4.4. Nanoparticles following lift-off. The nanoparticles are not the desired solid structures.
Chapter 5: Conclusions

5.1. Summary of Results

This project was able to build on the existing work done in this lab. Victoria Kim was able to consistently expose regular dot arrays in the resist (Figure 5.1), but she found her designs small and therefore hard to locate. She also was unable to successfully retain silver particles on silicon following lift-off.

![AFM scan of Victoria Kim’s array exposure](image_url)

*Figure 5.1. AFM scan of Victoria Kim’s array exposure. Two squares of exposure are present due to insufficient beam blanking.*

Victoria Kim, *Fabrication of Plasmonic Nanostructures using Lithography*, Scripps College, 2015

Progress on this project was achieved in multiple ways. First, silver is evaporated at a consistent thickness, and silver is present on the samples after lift-off. However, the resulting silver nanoparticles are not of consistent quality, so more research has to be done to find the best process for removing excess silver and leaving the sample clean.

Second, the nanostructure pattern design used here was changed from the one used in the previous project. Because it was 16 sets of 20x20 dots, the pattern was larger and was easier to locate under a microscope. This made qualitative characterization of the exposures and evaporations a smoother process.

5.2. Future Work

Future research in this lab should focus on improving lift-off quality. Being able to consistently create uniform nanostructures is crucial if their effectiveness at enhancing
efficiency is to be tested. One step would be to use MIBK:IPA 1:1 for the final removal of excess silver, rather than MIBK:IPA 1:3. Higher concentrations of MIBK have lower resolution and higher sensitivity, but the resolution of the developer is no longer critical at this stage of the lift-off process.

Once nanoparticle fabrication on silicon wafers is consistent, fabrication and testing on solar cells can be included in the lab’s research. This will require investigation into any potential differences in the process of fabrication on solar cells, rather than on plain silicon wafers, and determining how to best cover the relatively large surface of a test cell with nanostructures. Other expansions to research could include fabrication and comparison of aluminum nanostructures. Aluminum is much cheaper than silver, and is another ideal material for this purpose, as aluminum has a high cross-sectional scattering area and minimal parasitic absorption.
References
8. P Spinelli, Plasmonic light trapping in thin-film Si solar cells, IOPscience, 2011