Fabrication and Characterization of Plasmonic Nanophotonic Absorbers and Waveguides

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Plasmonics is a promising field of nanophotonics dealing with light interaction with metallic nanostructures. In such material systems, hybridization of photons and collective free-electron oscillation can result in sub-wavelength light confinement. The strong light-matter interaction can be harnessed for, among many applications, high-density photonic integration, metamaterial design, enhanced nonlinear optics, sensing etc. In the current thesis work, we focus on experimental fabrication and characterization of planar plasmonic metamaterials and waveguide structures. The samples are fabricated based on the generic electron beam lithography and characterizations are done with our home-made setups. Mastering and refinement of fabrication techniques as well as setting up the characterization tools have constituted as a major part of the thesis work. In particular, we experimentally realized a plasmonic absorber based on a 2D honeycomb array of gold nano-disks sitting on top of a reflector through a dielectric spacer. The absorber not only exhibits an absorption peak which is owing to localized surface plasmon resonance and is insensitive to incidence’s angle or polarization, but also possesses an angle- and polarization-sensitive high-order absorption peak with a narrow bandwidth. We also demonstrated that the strong light absorption in such plasmonic absorbers can be utilized to photothermally re-condition the geometry of gold nanoparticles. The nearly perfect absorption capability of our absorbers promises a wide range of potential applications, including thermal emitter, infrared detectors, and sensors etc. We also fabricated a plasmonic strip waveguide in a similar metal-insulator-metal structure. The strip waveguide has a modal confinement slightly exceeding that of the so-called plasmonic slot waveguide. We further thermally annealed the waveguide. It is observed that the propagation loss at 980 nm has been decreased significantly, which can be attributed to the improvement in gold quality after thermal annealing.
Acknowledgements

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Last but not least, thanks to my family, your love and care for me are always an motivation for me in my study and life.

Yiting Chen
2013 – 12
**Acronyms**

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
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<tbody>
<tr>
<td>2D</td>
<td>Two Dimensional</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscope</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
</tr>
<tr>
<td>CCD</td>
<td>Charge Coupled Device</td>
</tr>
<tr>
<td>DI</td>
<td>Deionized</td>
</tr>
<tr>
<td>EBL</td>
<td>Electron Beam Lithography</td>
</tr>
<tr>
<td>EUV</td>
<td>Extreme Ultraviolet Lithography</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width at Half Maximum</td>
</tr>
<tr>
<td>HMDS</td>
<td>Hexamethyldisilazane</td>
</tr>
<tr>
<td>ICP</td>
<td>Inductive Coupling Plasma</td>
</tr>
<tr>
<td>IPA</td>
<td>Isopropyl Alcohol</td>
</tr>
<tr>
<td>ITO</td>
<td>Indium Tin Oxide</td>
</tr>
<tr>
<td>IR</td>
<td>Infrared</td>
</tr>
<tr>
<td>LRM</td>
<td>Leakage Radiation Microscopy</td>
</tr>
<tr>
<td>MM</td>
<td>Metamaterial</td>
</tr>
<tr>
<td>MPA</td>
<td>Metamaterial Perfect Absorber</td>
</tr>
<tr>
<td>MIM</td>
<td>Metal–Insulator–Metal</td>
</tr>
<tr>
<td>NIL</td>
<td>Nanoimprint Lithography</td>
</tr>
<tr>
<td>NIR</td>
<td>Near Infrared</td>
</tr>
<tr>
<td>OSA</td>
<td>Optical Signal Analyzer</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical Vapour Deposition</td>
</tr>
<tr>
<td>PECVD</td>
<td>Plasma–Enhanced Chemical Vapor Deposition</td>
</tr>
<tr>
<td>RIE</td>
<td>Reactive Ion Etching</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SERS</td>
<td>Surface–Enhanced Raman Spectroscopy</td>
</tr>
<tr>
<td>SNOM</td>
<td>Scanning Near–Field Optical Microscope</td>
</tr>
<tr>
<td>SPP</td>
<td>Surface Plasmon Polariton</td>
</tr>
<tr>
<td>STM</td>
<td>Scanning Tunneling Microscope</td>
</tr>
</tbody>
</table>
TE  Transverse Electric
TEM  Transmission Electron Microscopy
TM  Transverse Magnetic
UV  Ultraviolet
WF  Writing Field
List of Publications

List of papers included in this thesis

I Yiting Chen, Jin Dai, Min Yan, and Min Qiu, “Influence of lattice structure on metal-insulator-metal plasmonic absorbers,” manuscript.


List of papers not included in this thesis

(VII) Xi Chen, Yiting Chen, Jin Dai, Min Yan, Ding Zhao, Qiang Li and Min Qiu, “Ordered Au Nanocrystals on Substrate Formed by Light-Induced Rapid Annealing,” Nanoscale, 6, 1756-1762(2014).


(X) Wei Wang, Ding Zhao, Yiting Chen, Hanmo Gong, Xingxing Chen, Shuowei Dai, Yuanqing Yang, Qiang Li, and Min Qiu, “Grating-assisted enhanced optical transmission through a seamless gold film,” submitted for publication.

(XI) Ding Zhao, Lijun Meng, Hanmo Gong, Xingxing Chen, Yiting Chen, Min Yan, Qiang Li, and Min Qiu, “Ultra-narrow-band light dissipation by a stack of lamellar silver and alumina,” submitted for publication.

(XII) Hanmo Gong, Yuanqing Yang, Xingxing Chen, Ding Zhao, Xi Chen, Yiting Chen, Min Yan, Qiang Li, and Min Qiu, “Large-scale gold nanoparticle transfer through photothermal effects in a metamaterial absorber by nanosecond laser,” submitted for publication.

List of conference proceedings not included in this thesis


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Chapter 1

Introduction

1.1 Background

Plasmonics is a very important part of nanophotonics, a new promising field of science and technology that exploits the optical properties of metallic nano-structures to confine and manipulate light at nanometre length scale, beyond diffraction limit [1]. Plasmonic devices take the advantage of sub-wavelength confinement of light field by means of surface plasmon polaritons (SPPs), a mixed wave of light and collective free electron oscillation on the metal surface. In recent decades, plasmonics undergoes tremendous development with various types of plasmonic devices designed and realized with their versatile applications in different fields. Physicists are not only attracted to investigate the fundamental physics involved, but also excited to design sub-wavelength plasmonic devices leading to miniaturized photonic circuits and metamaterials to obtain exotic electromagnetic properties. Besides, plasmonic devices also find their applications in medical and biological fields such as biosensing and cancer curing. Especially, surface-enhanced Raman spectroscopy (SERS) [2, 3], a technique based on the electric-field enhancement effect around metallic nanostructure, has become a wide-spread technique to detect single molecules and analyze material components. Besides, plasmonics devices based on graphene have also been a research focus in recent years [4, 5, 6].

For researchers in the field of optics, one of the most attractive aspects of plasmonic devices is their capability to confine and channel light in sub-wavelength structures, which offers the possibility to realize miniaturized plasmonic circuits with a feature size close to electronic circuits. To realize such kind of plasmonic circuits, it would require a variety of components, including waveguides [7, 8], couplers [9], switches [10], lasers [11, 12], antennas [13, 14] and so on. So far, a great endeavor has been dedicated to developing such plasmonic devices. For example, various types of plasmonic waveguides has been realized with excellent sub-wavelength field confinement [15, 16].

Plasmonic metamaterial is also a flourishing field in plasmonics. Metamaterials
are artificial materials with exotic electromagnetic properties usually unattainable in nature, due to their unique geometry rather than their material. By engineering the shape, size and period of metallic nanostructures with dielectric materials, metamaterials with specific effective electric permittivity ($\varepsilon$) and magnetic permeability ($\mu$) can be realized [17]. Metamaterials have many important applications, such as negative refractive index material [18, 19, 20], invisibility cloak [21, 22, 23], perfect lenses [24, 25], and perfect absorbers [26, 27, 28, 29, 30]. In the past years, much effort has been devoted to realizing different plasmonic absorbers in the interest of various applications like solar cells [31], thermal emitters [32, 33], imaging [34, 35] and so on. Plasmonic absorber will be one of the main topics we will discuss in the thesis.

Together with the advantage of sub-wavelength confinement of the light field, plasmonic nanostructures also suffer from the drawback of heat dissipation loss originating from the imaginary part of the refractive index of the metal. Therefore, there is usually a trade–off between better field confinement and larger loss for the plamonic waveguide. However, strong absorption from the metallic nanostructures can also bring in new opportunities in photothermal applications, such as metallic particle reshaping, thermal emitters, cancer curing. Therefore, it is one of our interest to study the thermal effects on the plasmonic devices. In this thesis, besides propagation loss issue of plasmonic waveguide and thermal annealing effect will be investigated, photothermal reshaping of nanoparticles will be experimentally demonstrated.

Meanwhile, development of nanofabrication techniques, such as electron beam lithography (EBL), focused ion beam (FIB), self–assembly, together with nanocharacterization technique such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), leakage radiation microscopy (LRM) and scanning tunneling microscope (STM) and so forth, has indispensable contribution to the prosperity of plasmonics research. Especially, EBL has become particular important state-of-the-art nanofabrication technique, with the advantage of allowing for full control of the shape, size and distribution of the nanostructures in nanometer order precision. Therefore, in this thesis, we will introduce the fabrication and characterization techniques involved in my work.

1.2 Thesis outline

This thesis is organized as follows:

In first chapter, I will introduce the background and motivation of my research work.

The second chapter discusses about fabrication of the sub-wavelength plasmonic devices. The fabrication process mainly includes three parts: sample preparation, pattern generation and pattern transfer. Pattern generation is realized through EBL, and is the most important part of the process. To obtain precise nano-patterns by EBL, various parameters such as proximity effect, resist thickness and so on
should be taken care of. Besides, discussions about material deposition technique and lift-off process are also presented.

In chapter 3, two types of home-made optical characterization experiment setups, transmission/reflection measurement setup and nanowire propagation length measurement setup, are demonstrated. By means of those two setups, we can acquire the absorption characteristics of our metamaterial absorbers and the propagation loss of various types of sub-wavelength waveguides, or of other waveguide-related devices.

Chapter 4 discusses about our research results on metamaterial absorbers. Metamaterial absorbers with different lattices including square, triangular and honeycomb lattice, are demonstrated and the influence of lattice on the absorption properties of absorbers is investigated. Special attention is paid to the honeycomb-lattice plasmonic absorber, which possesses an anomalous high-order resonance at near-infrared regime. This high-order resonance is different from the fundamental resonance due to its narrow bandwidth and angle dependence. Besides, we also present the experimental results of photothermal reshaping of the gold nanoparticles from cuboids to spherical domes.

In chapter 5, we explore the thermal annealing effect on our plasmonic analog of microstrip transmission line. It is experimentally presented that the propagation loss is alleviated dramatically after the plasmonic strip waveguide is put in a 300°C oven for 18 hours with slow heating and cooling process, due to the quality improvement of the gold layers.
Chapter 2

Fabrication of plasmonic devices

In this chapter, we discuss the fabrication process of our plasmonic devices with sub-wavelength structures. The process can be divided into three steps, which are sample preparation, EBL and pattern transfer. As a major part, we will cover the key principles of EBL and explore the effects of various factors (dose, proximity effect, resist thickness and so on) and their impacts on the resolution, precision, shape of pattern generated. Details about fabricating plasmonic absorbers, and quarter-wave plate are also presented.

2.1 Overview

As shown in Fig. 2.1, the nanofabrication mainly includes three steps. Firstly, the design must be drawn in a software, and the substrate must be prepared, such as cleaning the substrate with plasma, depositing desired materials onto the substrate. Secondly, the pattern is generated by different techniques depending on specific circumstances, including EBL, optical lithography, FIB etching and so on. In my research work, EBL is the main technique adopted, because it fulfills the condition of nanometer order precision and big fingerprint exposure. Thirdly, the pattern is transferred onto the functional layers by means of etching or liftoff. When dealing with metal deposition, liftoff is the more common method, in which I use

![Diagram](https://via.placeholder.com/150)
CHAPTER 2. FABRICATION OF PLASMONIC DEVICES

to fabricate the metamaterial absorbers, plasmonic waveguides. While etching here mainly means dry etching, including plasma etching, inductive coupling plasma (ICP) and reactive ion etching (RIE). Dry etching is a very common technique in silicon or semiconductor industry, together with plasma-enhanced chemical vapor deposition(PECVD).

2.2 Electron beam lithography

Lithography is a process whereby an arbitrary (usually 2D) pattern can be accurately and reproducibly generated in a specialized layer of material called the resist [36]. Optical lithography (or photolithography) is a microfabrication technique widely used in electronic industry to produce printed circuit boards, by means of exposing the resist through a mask by UV (ultraviolet) light. Even though the resolution of optical lithography has been improved via using deep UV source or immersion lens, it is still difficult to achieve nanometer precision due to diffraction limit. When an electron beam is accelerated by a high voltage such as 100 keV, its wavelength can reach as small as 3.9 pm [37]. Thereby diffraction limit will not be an obstacle for electron beam microscopy and nanometer order resolution is realizable. While EBL is such kind of direct-writing technique based on scanning a focused electron beam with designed pattern on the substrate covered with an electron-sensitive resist. Now EBL has become one of the major nanolithography techniques to fabricate plasmonic devices with sub-10 nm precision. Hereby, we will introduce the EBL system we utilized in our lab.

![Figure 2.2: (a) Photo of the Raith 150 EBL system. (b) Schematic of the main components of the Raith 150 EBL system.](image)

The EBL system we use is Raith 150, which is shown in Fig. 2.2(a) [38], located in KTH nanofabrication lab. One computer is connected to control this system. Fig. 2.2(b) [39] presents the schematic of the major components of the Raith EBL system. Basically it is an upgrade version of an SEM system by adding a pattern...
generation system, which helps to control the electron beam to scan along desired path to generate pattern on the resist. Usually the EBL system is comprised of the following components: column, chamber with interferometer-controlled laser stage, objective lens and other electronics such as power supply, vacuum pump, control units and computers.

In the EBL system, the part that generates the electron beam is referred as the column. The electrons are generated by the electron gun usually with a tungsten filament and accelerated with acceleration voltage ranging typically from 1 keV to 100 keV. Larger acceleration voltage will produce electron beam with smaller wavelength. A beam blanker is employed to switch the beam on or off, together with an aperture to define the beam. The aperture helps to set the beam convergence angle and beam current, control the lens aberrations and resolution. Smaller aperture size provides pattern with better quality and finer structure, and takes longer exposure time due to smaller current. To obtain the best form of the focused beam, several further adjustments need to be carried out before exposure, including focusing, stigmation adjustment, aperture alignment, etc.

The sample is placed on an interferometer-controlled laser stage, which defines the positions with respect to the column. During exposure, the beam sweeps across the sample pixel by pixel (step size), with the electron beam being blanked and unblanked by blanker. Usually the whole pattern is divided into small parts (not in FBMS (fixed beam moving stage) mode), referred as writing fields (WFs). Inside each WF, the stage stays still and the electron beam is deflected by the column to cover the whole area. The stage only moves between WF to WF, which will introduce random error causing mismatch between the adjacent WF s, which is called stitching error. Thus, an extra procedure called WF alignment is applied to minimize this stitching error: a unique and easily recognizable feature is first positioned in the center of the screen, then the computer moves the feature to three different places, then the computer will compare the coordinates given by the system and by the operator respectively, and then offer new parameters to redefine the relative frame of axis. After repeated calibrations with increasing magnification ratio (with smaller WF), the scaling and orthogonality of the deflection system may achieve ideal agreement with the stage movement system.

The pattern generator controls the exposure paths by means of operating the beam blanker and scan coil amplifiers in accordance with the data of the patterns from the computer. The scanning speed is determined by two factors: the step size, which means the distance between two adjacent scanned spots, and the dwell time, which is the time span for the electron beam to stay at one spot to provide sufficient dose to expose the resist. Here is the formula of the interdependent relation between the four parameters that determine the dose used for exposing:

\[
\text{Area dose} = \frac{\text{Beam current} \cdot \text{Dwell time}}{(\text{Step size})^2}
\]  
(2.1)

Of course, dose factors in the design are also taken into account during the exposure.
Table 2.1: Vendor-specified parameter range and frequently used parameters in this thesis

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
<th>Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acceleration voltage (keV)</td>
<td>0-30</td>
<td>3-25</td>
</tr>
<tr>
<td>Aperture size (µm)</td>
<td>7.5-120</td>
<td>7.5-30</td>
</tr>
<tr>
<td>Writefield size (µm)</td>
<td>60-1400</td>
<td>100-200</td>
</tr>
<tr>
<td>Current (nA)</td>
<td>0.004-10</td>
<td>0.2-0.3</td>
</tr>
<tr>
<td>Working distance (mm)</td>
<td>2-10</td>
<td>5</td>
</tr>
<tr>
<td>Stepsize (nm)</td>
<td>1-22</td>
<td>6</td>
</tr>
</tbody>
</table>

In Raith 150 EBL system, several important parameters need to be defined to achieve better exposure quality according to specific pattern need to be exposed. Table 2.1 presents the main parameter range from the system and the frequently used parameter by me.

In the following subsections, several important issues about EBL will be addressed.

2.2.1 Proximity effect

When the electron beam is scanning the resist, the real exposed size in the resist is usually larger than the designed size due to the forward scattering of electrons in the resist layer and backscattering of secondary electrons from the substrate. This phenomenon is called proximity effect. Proximity effect introduces random exposure to neighbouring area close to the scanned electron beam, may increase the real exposure dose dramatically and even alters the shape of the structures. Therefore, when exposing a new structure, it is always necessary to take proximity effect into account and adjust the dose. Proximity effect is closely dependent on the pattern density. Due to proximity effect, larger density of patterns requires smaller dose, otherwise overexposure or pattern distortion will occur. To compensate proximity, dose rectification and structure adjustment sometimes are necessary. Decreasing the substrate thickness can also weaken the proximity effect.

2.2.2 Resist

There are two types of resists used in EBL to generate the pattern: positive resist and negative resist. Positive resist usually consists of long-chain organic molecules. After exposure by electron beam, the long-chain molecules break into short-chain ones, becoming more soluble to the developer. Therefore, the unexposed portion of the positive resist is left on the wafer, sharing the same pattern as the desired structure. On the contrary, the exposed negative resist molecules become crosslinked/polymerized, and thus more difficult to dissolve in the de-
2.2. ELECTRON BEAM LITHOGRAPHY

developer. As a result, the unexposed portion of the resist is removed and an in-
verse (photographically “negative”) pattern is left on the substrate. In my work,
positive resist ZEP520A and negative resist Ma-N 2403 are utilized to fabricate the
plasmonic waveguides, metamaterial absorbers and other plasmonic devices.

Usually, resist is spin coated onto the substrate directly. However, noble metals
such as gold, silver exhibit poor wetting and resist adhesion, thus it is difficult
to obtain uniform and stable resist by direct spincoating. Therefore, adhesion
promoter like HMDS (Hexamethyldisilazane) can be used to enhance the adhesion
between the metal and resist.

2.2.3 Dose test

Dose test is to execute a test-exposure process to find the proper dose to be used
in the to-be-realized pattern. This can be done by generating a pattern which then
is replicated throughout the structure with varying dose factor, among which the
right dose will choose. The pattern used in dose test should be the same as or part
of the desired pattern. Dose test should be performed not only for new patterns,
sometimes also for old ones when the resist hasn’t been used for a long time. Dose
test is a very crucial step in doing EBL, because small difference of dose can change
the size and quality of the pattern dramatically, while the dose can be easily changed
due to the variance of the following parameters or conditions, including resist type,
resist thickness, softbake conditions, E-beam acceleration voltage, density, shape
and size of the pattern.

2.2.4 Resist thickness

To have an easier and better lift-off with less peeling off in the edge of the pattern,
it is often recommended that the thickness of the resist is at least 10 times larger
than that of the materials to be deposited onto the resist. There are two ways to
change the resist thickness, which are to change the spinning speed and to dilute the
resist with certain material and ratio. Usually spincoating includes two steps: the
first step is a slow spinning at around 300 rpm (round per minute) for 3 seconds
to cover the whole substrate with resist and avoid resist tear-off due to too fast
acceleration, and the second step is a high speed spinning which can be as large as
6000 rpm for 60 seconds. Higher ramp also results in thinner resist.

2.2.5 Resist thickness calibration

Resist thickness calibration is also a key step, because dose is also sensitive to the
resist thickness. While the resist thickness depends on the following parameters,
including resist material, the ramp, spinning speed, the age of the resist, soft-bake
temperature and time, substrate, substrate size, etc. After the resist is stored in lab
for a long time, resist may have higher viscosity due to vaporization of the solvent,
thus resulting in thicker spincoating even with the same spin parameter. Therefore,
it is indispensable to calibrate the resist thickness in the occurrence of the variation of the previous mentioned parameters such as the resist type, spinning parameters, the substrate and so on. The calibration can be done with the aid of a surface profiler after scratching several lines on the resist.

2.2.6 Anti-charging

When the E-beam is scanning the sample, the sample tends to be negatively charged unless the electrons can drift away to the ground totally. For a silicon substrate, due to its good conductivity, electrons can gain their access to the ground quickly and hardly charge the substrate. However, when it comes to a glass or quartz wafer, the electrons from a high-energy beam can stay on the wafer for some time. When the electrons from the beam cannot be conducted away fast enough, an extra electric field appears and exerts a repulsive force to the incoming electron beam. As a result, it will be difficult to do basic SEM adjustment procedures such as focusing and aperture alignment. Even worse, the pattern will be distorted or damaged by the charging. Then it is necessary to deposit some conductive material like ITO (Indium tin oxide) or a thin layer (5-10 nm may be enough) of aluminium on top of or below the resist to eliminate the charging problem. ITO is a more common choice due to its transparency property to the visible light, therefore it doesn’t need to be removed after exposure. If aluminium is used as an anti-charging layer, it has to be removed by wet etching after exposure.

Sometimes if the density of the pattern is not large, the charging may be not strong enough to influence the pattern during exposure. Then we just need to solve the SEM adjustment problem before exposure. Therefore, we can deposit a conductive layer of material onto the empty area of the sample where no pattern will be written. Then after carrying out the focus adjustment, aperture alignment and astigmatism adjustment, the beam can directly move onto the silica area to write the pattern.

2.3 Film deposition

There are mainly three kinds of physical vapor deposition (PVD) methods: electron beam (E-beam) evaporation, filament evaporation and sputtering. In our fabrication process, electron beam evaporation deposition method is utilized to deposit gold, silver, titanium, germanium, aluminium and alumina film onto our samples. In the machine, a filament source (usually tungsten) is heated by injecting current to emit electrons to heat samples to high temperatures. The electron beam is steered 270° into material source by magnetic fields and rastering, so that the tungsten filament can be sheltered to avoid contamination from the emitted materials.

To achieve high-precision control of deposition thickness, a quartz crystal is utilized to measure the deposition rates in real time. A quartz crystal is a piezoelectric material. When a high-frequency voltage is applied onto certain faces of
the crystal, the crystal surface moves due to the volume change with a resonant frequency, which is proportional to the mass and thickness of the film deposited onto the crystal. The deposition rate can thus be measured in situ by monitoring the resonant frequency change of the crystal. Every material has a unique recipe of the relationship between the resonance frequency shifting and the film thickness. Quartz crystal monitor can achieve the precision of detecting the thickness change of less than one single atomic layer [40].

It is necessary to calibrate the real thickness by means of ellipsometer, surface profiler or thin film interferometry. Because even deposited in the same time, substrates put in different positions of the chamber actually have different deposition rates, with a maximum difference of about 10%.

The deposition is usually operated at a pressure lower than \(5 \times 10^{-7}\) mbar. The standard deposition rate is between 0.5 - 1 Å/s. With lower deposition rate, the deposited film shows better quality of smaller grain size and greater uniformity. If it is to deposit gold or silver onto silica to alumina, usually 2~4 nm thick of Ti or Ge is deposited first to enhance the adhesion between the noble metals and the dielectrics.

Compared to other two kinds of PVD methods, sputtering and filament evaporation, the biggest advantage for E-beam evaporation is that it has the highest purity due to its high vacuum deposition condition and pollution-free heating source of electrons.

2.4 Lift-off

After EBL exposure and development, a reverse pattern is created in the resist layer. Then the function layer is deposited onto the sample. By removing the resist and the materials on top of it with chemical bath, the remaining of the function layer will finally have the desired pattern. This pattern transfer process is called lift-off.

2.5 Fabricated nanostructures

2.5.1 Process of fabricating metamaterial absorber

Our plasmonic metamaterial absorbers have an MIM (usually gold-alumina-gold) structure, with the top layer covered with a periodic array of gold nanoparticles. Due to the electromagnetic resonances between the gold particles and gold film based on localize surface plasmon resonance, the absorber may possess strong absorption in visible or near-IR frequency regime. The absorption characteristics of the absorbers can be tuned by tailoring the thicknesses of the three layers or the shape, size and period of the gold particles. Even though we have fabricated absorbers with different designs (with different shapes, sizes, thicknesses and lattices) (Fig. 2.3), actually those absorbers are produced with almost same fabrica-
CHAPTER 2. FABRICATION OF PLASMONIC DEVICES

Figure 2.3: SEM images of MIM structure based metamaterial absorbers with different particle shapes, sizes and lattices

tion process, all with the pattern generated by EBL using the positive photoresist Zep520A. Here we present the fabrication process of the honeycomb-lattice absorber, and the process flow is illustrated in Fig. 2.4.

Here is the detailed procedure:

1. Gold and alumina deposition.

After cleaning in acetone and IPA (isopropyl alcohol) with ultrasonic bath, the silica substrate is deposited with 4 nm thick titanium, 80 nm thick gold and 28 nm thick alumina. The titanium is used as an adhesion layer and also to improve the particle quality of the gold film.

2. Spin coating resist Zep520A.

Here, Zep520A is diluted by anisole with the volume ratio of one to two, so that thinner resist can be obtained. With the spin speed of 6000 rpm, the substrate is coated with a ∼200 nm thick Zep520A. The uniformity of resist deposition depends on three parameters: high spin speed, the viscosity of the coated resist, and clean substrate [41]. Usually, spin speed larger than 2000 rpm is recommended. To clean the substrate, ultrasonic baths with acetone and IPA should be performed, and prebake at 180 °C for 5 minutes can remove residual moisture or IPA.

3. Softbake.
Bake the substrate at 180 °C for 10 minutes in a contact hotplate. Softbake reduces the solvent concentration in the resist, stabilizes the resist improve the adhesion to the substrate, and avoiding bubbling in following thermal treatment (etching, deposition). After softbake, the resist thickness can be measured by means of surface profiler.

4. Exposure.

The exposure is performed with the following parameters: 10 μm aperture, 25keV acceleration voltage, 5 mm working distance and 100 × 100 μm writing.
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...field. Dot scan is used to generate nanodisk patterns instead of area scan, because it costs less time than the latter one.

5. Development.
Soak the exposed sample in developer P-xylene for 90 seconds and rinse it with IPA for 30 seconds. Dry the sample by a nitride gas gun. After development, the pattern is revealed and can be examined under an optical microscope. Besides, the resist thickness can be rechecked by surface profiler.

6. Metal deposition and lift-off.
4 nm Ti and 30 nm gold is deposited onto the sample by e-beam evaporation with the deposition rate of 0.5 and 1 Å/s respectively.

After soaked in acetone for 2 minutes and remover 1165 for 3 minutes with ultrasonic bath, the sample is rinsed by IPA for 10 seconds. Finally, the resist and residue of metals are removed, leaving the metamaterial absorber structure.

2.5.2 Process of fabricating plasmonic quarter-wave plate

The plasmonic quarter-wave plate consists of periodic sub-wavelength cross-apertures in an 60 nm thick gold film on a silicon substrate. Due to the length difference between two arms of the cross, a phase delay is introduced between the two orthogonal polarizations of the transmitted light. By tuning the arm-width and arm lengths of the cross, a quarter-wave plate at particular wavelength can be designed. In our case, with the arm width 100 nm, arm length 511 nm and 680 nm, the sample can work as a quarter-wave plate at 1550 nm. Fig. 2.5 shows the top-view SEM image of the cross apertures fabricated by EBL.

To generate the cross aperture, negative resist Ma-N 2403 is firstly employed by us. However, due to the weak adhesion between Ma-N 2403 and the silicon substrate, the crosses are easily blown away by the nitride gas gun or drift away due to the surface tension of the liquid during development. Even though we tried to promote the adhesion by adding one layer of adhesion promoter HMDS or increasing the soft-bake temperature and time, the result is still not good enough. Finally, we turn to positive resist Zep520A again, by exposing the other part of the unit instead of cross, which will take more exposure time and have a more complex pattern to draw in the software. In the following part, the exposure procedure of negative resist will be introduced, with the process flow shown in Fig. 2.6.

1. Substrate pretreatment.
The substrate should be free of any organic and inorganic contaminations and physical absorbed humidity. For our plasmonic quarter-wave plate, a double-side polished 300 µm thick silicon wafer is used. Before spincoating, the substrate should be firstly soaked in acetone and IPA with ultrasonic bath...
2.5. **FABRICATED NANOSTRUCTURES**

![SEM Image](image)

Figure 2.5: Top-view SEM image of the plasmonic quarter-wave plate. Inset is the enlarged version.

for 5 minutes respectively. The acetone is used to clean the contaminations of particles and organic impurities, while IPA is used to remove the acetone residuals. After the ultrasonic bath, a 5 minutes hot bake at 120 °C follows to dry the substrate, which also helps increase the adhesion between the substrate and resist. Furthermore, a plasma etching process can also be done to clean silica on top due to oxidation and remaining contaminations.

2. Spincoating.

With the spin speed of 6000 rpm, the thickness of Ma-N 2403 will be around 480 nm.

3. Bake the substrate at 90 °C for 60 seconds.

4. Exposure.

We choose 25 keV acceleration voltage, 10 μm aperture, 5 mm working distance and 100 μm writing field. The area dose is around 80 μAs/cm² and step size is 6 nm.

5. Development.

First soak the sample in developer Ma-D 525 for 120 seconds and rinse it in deionized (DI) water for 2 minutes. The development time for Ma-D 525 should be longer if the developer has been stored for a long time. For instance, the development time can increase up to three and half minutes when the developer is 2 years old. Then dry the sample by blowing it with nitride
gas gun. The resist in the unexposed area will be removed and the cross-shapped resist will be left. Afterwards, the sample will be checked by optical microscope, and the resist thickness examined by surface profiler.

6. Metal deposition and lift-off.

By E-beam evaporation, 4 nm thick titanium and 60 nm thick gold will be deposited on top of the resist with the deposition rate of 0.5 and 1 Å/s respectively.

Both mr-Rem 660 and acetone can work as remover. In our case, acetone is used. First soak the sample in acetone with ultrasonic bath for 10 minutes, then rinse it in IPA for 5 minutes. Then the resist and residual of metals will be removed, and the periodic cross apertures appear in the gold film.
Chapter 3

Optical characterization

Characterization is usually last but not least part of the whole experiment process. It shows the quality, performance and efficiency of the sample fabricated. In my work, two basic optical characterization setups are built and used frequently, including angle-resolved transmission/reflection experiment setup and nanowire propagation loss measurement setup. In addition, the following characterization techniques are also employed during my research but will not be elaborated in the thesis: scanning electron microscope (SEM), atomic force microscope (AFM), leakage radiation microscope, ellipsometer, surface profiler, thin film interferometry and so on.

3.1 Angle-resolved transmission/reflection experiment setup

Our home-made angle-resolved transmission/reflection measurement setup is capable of obtaining the absorption spectra of samples with a fingerprint as smaller as 50 × 50 µm, in the wavelength range from 300 to 1700 nm, from normal incidence to 60° oblique incidence for both orthogonal polarizations. With the incident beam having an angular divergence smaller than 2°, it can be considered as plane wave.

Firstly, let’s take a look at the oblique incident case. As shown in Fig. 3.1(a) [42], a broad-band light (500–2400 nm) from a super-continuum light source is focused onto the sample by an achromatic lens after passing through a collimator, diaphragm, attenuator and polarizer, with the spot-size smaller than 50 µm. If the lens is replaced by an 10× or 20× objective, the spot-size can be even shrunk to smaller than 5 µm. However, the divergence angle will become much larger. Behind the sample there is an objective and a CCD, which are used to locate the sample and beam and make sure that the sample overlaps with the beam position with smallest spot–size (on the focus). The reflected light can be focused onto a single mode fiber and collected in an optical signal analyzer (OSA) through the fiber. By adding a beamsplitter between the objective and CCD, the transmission can also be measured.
With one beamsplitter introduced between the lens and the sample (Fig. 3.1(b)), the reflection from normal incident beam can then be measured. This setup can also be used to photothermal reshape metamaterial absorber particles by means of increasing the intensity by rotating the attenuator. Furthermore, this simple setup can upgrade to a more complicated fusion device with small modification and introducing a pulse generator between the attenuator and the lens. The pulse generator is able to control the rise/fall time, frequency and duty cycle of the incident beam, thereby much more complicated fusion experiment can be realized.

3.2 Nanowire propagation loss measurement setup

This home-made setup is designed to measure the propagation loss of some sub-wavelength waveguide, including nanowires, MIM or hybrid waveguides. Fig. 3.2(a)
illustrates part of the experiment setup. A white light source is utilized to illuminate the sample though two beam splitters, and an objective. Meanwhile, the light from the field will also be imaged into a CCD on top, from which both dark- and bright-field images can be taken. On the bottom, the sample is located on an XYZ transitional stage. On one side of the stage is a fiber taper connected to a light source, which is used to couple excitation light into the waveguide. The fiber taper is made from a normal single-mode fiber by pulling the fiber core under a lamp flame after the jacket and buffer of the fiber are removed. The diameter of the tip of fiber taper can be as smaller as around 1 $\mu$m.

Figure 3.2: (a) Schematic of the home-made propagation loss measurement setup. (b) Optical microscope images of the fiber taper and nanowire at bright- (top one) and dark-field from an visible light CCD with the incident light at 980 nm.

The method used to measure the propagation loss is similar as the cut-back method in the fiber-optics community. Firstly, the fiber taper is moved close to the waveguide until contacts received, and then from one end of the waveguide, scattered light will be captured by the CCD camera in dark field. By carefully changing excitation position (the contact point of the taper and waveguide), the intensity of the scattering light will be found changing accordingly. The propagation loss coefficient will be then obtained by analyzing dark-field images according to the relationship between the scattering light intensity and the propagation distance (from the excitation point to the output end of the waveguide). Fig. 3.2(b) illustrates the optical microscope images of the fiber taper and nanowire at both bright and dark fields.

If we put another fiber taper in the other end of waveguide, the output light
can be directly coupled to this taper from waveguide and thus propagates to OSA. However, to use this method, there are two conditions needed to be fulfilled: firstly, the waveguide must be long enough, even longer than 1 mm, so that the direct illumination from input taper to the output taper can be small enough to be ignored; secondly, the propagation loss must be small, otherwise, due to the long propagation distance and the coupling loss between the taper and the waveguide, the loss will too large and the output signal will be too weak to be detected.
Chapter 4

Metamaterial absorbers and photothermal reshaping

In this chapter, we will present our MIM-architecture based metamaterial absorbers with different shapes of metallic particles, such as square [42], rectangular [43] and circular [44] gold nanoparticles, and different lattices, including square, triangular and honeycomb lattices. Firstly, we will discuss about the honeycomb-lattice absorber with an anomalous high-order mode. Then we will compare the honeycomb-lattice absorber with absorbers consisting of square- and triangular-lattice gold nanodisk arrays. Furthermore, we also present the photothermal reshaping experiment of the absorber particles.

4.1 Plasmonic honeycomb-lattice absorber

Our absorber is fabricated with the standard process by EBL and liftoff as illustrated in chapter 2. Fig. 4.1 illustrates the geometric structure of the metamaterial absorber. The absorber has an MIM structure, consisting of 30 nm thick gold nanodisks, 28 nm thick Al$_2$O$_3$ film and 80 nm thick gold film from top to bottom. Under both gold layers is 4 nm thick Ti, used as an adhesion layer to enhance the binding between gold and dielectric layers. The radius of the gold nanodisks is 90 nm and the distance between two close-by nanodisks is 310 nm. In Fig. 4.1(b), the SEM images demonstrate that the gold nanoparticles of the fabricated absorber have very uniform round profile and well distributed honeycomb lattice.
CHAPTER 4. METAMATERIAL ABSORBERS AND PHOTOTHERMAL RESHAPING

Figure 4.1: (a) Geometric schematic of the honeycomb lattice absorber. Both the top layer nanodisks and bottom film are gold, and are separated by a layer of alumina film. The distance between two adjacent nanodisks is 310 nm, and the diameter of the nanodisks is 180 nm. (b) Top-view SEM image of the sample and inset is the enlarged view.

By means of the home-made transmission/reflection experiment setup, we measure the absorption spectra of the metamaterial absorber for both polarizations and orientations (Fig. 4.2). In the measurement, the transmission is neglected due to the 80 nm thick gold film in bottom, which reflects most of the light. Therefore, after we measure the reflection (R), the absorption is obtained (A = 1 - R). Fig. 4.2 [44] manifests that our absorber sample has almost perfect absorption ability with the fundamental resonance at around 1140 nm: the absorber achieves more than 98% absorption at normal incidence for both polarizations and incident planes; even when the incident angle increases up to 50°, the absorption for all four cases sustains above 90% or even more. Interestingly, besides the angle-insensitive fundamental mode, angle-sensitive high-order resonances are clearly observed in TM modes for both incident planes. In Fig. 4.2(b), for the case of H⊥Sxz, when the incident angle increases to 20°, a high-order absorption peak appears at 671 nm, and the peak shifts to 787 nm as the incident angle increases to 60°, i.e., about 2 nm per degree, together with almost doubled absorption from 35% to 69.9%. Besides the characteristic of angle-sensitivity, the other special property of this high-order mode resonance is its narrow bandwidth compared to the fundamental mode. For example, at 60° incident angle, the full width at half maximum (FWHM) of the high-order absorption peak is about 30 nm, while the counterpart of the fundamental mode is about 220 nm. We believe the high-order mode stems from the coupling between different gold nanodisks with the involvement of propagating surface plasmon polaritons(SPP). In contrast, the fundamental mode originates from localized surface plasmon oscillation between the gold particles and bottom gold film. About the mechanism of the two different kinds of resonances, we will elaborate it later.
4.1. PLASMONIC HONEYCOMB-LATTICE ABSORBER

Figure 4.2: Measured absorption spectra of the honeycomb lattice absorber for both incident planes and polarizations: (a) E \perp S_{xz}, (b) H \perp S_{xz}, (c) E \perp S_{yz}, (d) H \perp S_{yz}. Numbers 0° - 60° denote the incident angle. The absorbances of the fundamental and high-order resonances are also indicated.
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For the case of $H \perp S_{yz}$ (TM polarization, Fig. 4.2(d)), two obvious high-order modes are unveiled with large incident angles. At $20^\circ$, two peaks come to exist at 675 nm and 757 nm, with the absorption of 40\% and 26\% respectively. As the incident angle increases, the first high-order mode stays in around 675 nm and the latter encounters noticeable red-shift, with enhanced absorption for both peaks. At $60^\circ$, the red-shifting peak has moved to 881 nm with the absorption of 75\% and FWHM of 19 nm, while the other high order mode with the absorption of 67\% and FWHM of 88 nm. As for the TE modes, for both cases of $E \perp S_{xz}$ and $E \perp S_{yz}$ (Fig. 4.2(a) and 4.2(c)), no strong red-shifting high order resonance is found.

![Image](image_url)

Figure 4.3: Numerical simulation of absorption spectra map for both incident planes and polarizations: (a) $E \perp S_{xz}$, (b) $H \perp S_{xz}$, (c) $E \perp S_{yz}$, (d) $H \perp S_{yz}$. The black dashed lines indicate the angle-dependent high-order absorption peaks.

In summary, the sample has a fundamental mode at 1140 nm, with almost total absorption over a broad incident angle range regardless of the polarization and the incident plane. While with large incident angles, red-shifting high-order resonance is revealed, with a much narrower bandwidth compared to the fundamental mode.
This high-order mode is not only sensitive to the incident angle, but also depends on the polarizations: only for the TM modes, this red-shifting high-order peak is detected clearly. While for the TE modes, there is no strong red-shifting absorption peak found.

To investigate the nature of these resonances and compare with the experimental result, we also performed computational simulation by means of a commercial software COMSOL MULTIPHYSICS. Fig. 4.3 [44] and Fig. 4.4 [44] illustrate the calculation results about the absorption spectra map and field distributions respectively. In the simulation, we used the data of permittivity of alumina from Paliks’ book [45], and that of gold from the data measured by Johnson and Christy [46]. About the absorption, the simulation results agree well with the experimental results. Firstly, for the fundamental mode, strong absorption at 1140 nm is sustained over a broad range of incident angles and for all four polarizations. Secondly, noticeable angle-sensitive high-order mode is found in both TM modes, also with narrow bandwidth. For example, for the case of \( H \perp S_{yz} \), the calculated red-shifting high-order mode at 60° appears at 880 nm, possessing the absorption of 82% and bandwidth of 26 nm, very close to the experimental counterpart (74.5% and 19 nm at 881 nm).

To better understand the intrinsic properties of the fundamental mode and red-shifting high-order mode, we calculated the field distributions of both modes in the \( yz \) plane at \( x=0 \) nm (Fig. 4.4(a)). Fig. 4.4(b) illustrates the field distribution of the fundamental resonance at 1140 nm at normal incidence for the case of \( H \perp S_{yz} \). We can see that two anti-parallel currents run on bottom surface of the gold particle and top surface of the gold film respectively, driven by the magnetic response to the incident light [26]. And the electromagnetic energy is strongly localized in the dielectric layer between the gold particles and the gold film. This is a localized surface plasmon mode, with coupling between gold disks and the image-part in the bottom gold film [47], without obvious coupling between neighbouring particles. Since this fundamental mode is mainly determined by independent particles, the characteristics of the resonance should not change much even though the lattice is different. As for the high-order red-shifting mode, whose field distribution is shown in Fig. 4.4(c), is a propagating Bragg mode (delocalized surface plasmons) [48, 49]. Apart from the coupling between the gold disk and the gold film, there is also strong coupling between neighbour disks. This propagating Bragg mode is related to the coupling of propagating wave with a reciprocal vector added to the in-plane momenton \( k_{\parallel} \) (\( k_{\parallel} = k_0 \sin \theta \)) of the wave, which has the phase-matching condition based on Bragg scattering theory [49]:

\[
\beta = |k_0 \cdot \sin \theta + q_{mn}|
\]  

(4.1)

where \( \beta \) is the momenton of the Bragg mode (SPPs), \( q_{mn} \) the reciprocal lattice vectors of the honeycomb lattice, and \( k_0 \) is the momenton of the incident light. For the normal incidence case, the \( k_{\parallel} \) is zero, thus the Bragg mode cannot be excited. To maintain the phase-matching condition, as the incident angle increases, \( k_{\parallel} \) should
decrease to compensate the increasing \( \sin \theta \), which explains why the SPP mode has a red-shift. Besides, in this case, the related reciprocal vector should has opposite direction compared to the in-plane component of the incident wave, resulting in that this is a reverse propagating SPP mode. To further confirm this explanation, we calculate the dispersion curve of the SPP wave at an air/alumina/gold interface, shown in Fig. 4.5, combined with the absorption spectrum map of TM mode in \( yz \) incident plane in frequency-\( k_y \) space. In the color map, the downward moving band is the red-shift high-order mode, and the flat broad band beneath is the fundamental mode. The red curves denote the dispersion relation of the SPP modes, while the left one represents a backward propagating mode. We can see that the shape of the red-shift mode resembles the left red solid curve in the frequency domain of 1–1.2. If we move the left red curve to the right by 2, which is contributed by the reciprocal lattice vector, it will overlap with the high-order mode. Therefore, it also proves that this high-order mode is a reverse propagating SPP mode.

![Figure 4.4](https://example.com/figure4.png)

**Figure 4.4:** (a) Illustration of the structure in \( xy \) plane used in simulation. (b, c) Calculated field distribution in the \( yz \) plane at \( x=268.5 \) nm at resonances (b) at 1140 nm at normal incidence and (c) at 880 nm at 60° incident angle. The color map represents the magnetic field of \( x \) component and arrow surface the electric field.

If we define the quality factor \( (Q_f) \) of the plasmonic resonance as the ratio of
the resonance wavelength ($\lambda_r$) and the FWHM of the absorption peak:

$$Q_f = \frac{\lambda_r}{\text{FWHM}}$$  \hspace{1cm} (4.2)

We will see that the high-order mode has a much larger $Q_f$ than the fundamental mode. For example, according to the simulation results, for the TM mode in the $S_{yz}$ incident plane (Fig. 4.3(d)) at 60° incident angle, $Q_f$ of the high-order mode at 880 nm is 34 (the experimental counterpart is even higher, reaching 46), while that of the fundamental mode at 1113 nm is 5. This bandwidth difference can be explained by the different resonance mechanisms they have: the fundamental resonance mainly comes from the localized surface plasmon of the gold nanodisks, while the high-order mode stems from Bragg scattering of the honeycomb lattice.

![Figure 4.5: Absorption spectrum map shown in frequency-$k_y$ for the case of H⊥ S_{yz}. The light line of air is drawn in solid black line. Two red curves denote the dispersion relation of SPPs at an air/alumina/gold interface, with the left curve for the backward SPPs wave and the right curve for forward SPPs wave. The frequency is normalized by $c/a_y$, where $c$ is the speed of light, and $k_y$ is normalized by $2\pi/a_y$. $a_y$ is 930 nm, the lattice constant along y-axis.](image)

In conclusion, we fabricated an MIM metamaterial absorber, with a gold nanodisk array in honeycomb lattice on top layer, operating in the near-infrared regime. We also measured the absorption of the absorber with a broad range of incident angle for both polarizations and incident planes. In addition to a perfect absorption peak at 1140 nm from the fundamental mode, an angle-sensitive narrow-band high-order absorption peak is also observed at the short wavelength range. This high-order mode has a remarkable red-shift with increasing incident angle, and its bandwidth can be as narrow as 19 nm, 10 times narrower than that of the fundamental mode. This high-order mode is proved to be a reverse propagating Bragg scattering mode, and the fundamental mode a localized surface plamon mode. Due to the narrow bandwidth and incident angle sensitive properties of the high-order
CHAPTER 4. METAMATERIAL ABSORBERS AND PHOTOTHERMAL RESHAPING

mode, this absorber has the potential to be utilized as high performance optical sensors and thermal emitters.

4.2 Metamaterial absorbers with different lattices

As we all know, materials consisting of the same kind of atoms in different lattices may have totally different physical properties. For example, graphite, diamond and carbon coke are all comprised of carbon atoms, but have very different properties, such as different conductivity, shapes, hardness, melting points and so on due to different lattices they possess. While metamaterial consists of “artificial atoms”, we are interested in raising the question: how does lattice affect the electromagnetic properties of metamaterial absorbers?

In recent years, many different kinds of MIM-structure based metamaterial has been designed and realized [34, 27, 28, 29], since the first metamaterial absorber operating at microwave wavelength range is experimentally demonstrated in 2008 [26]. Most of the metamaterial absorbers consist of MIM structure, including one layer of noble metal and dielectric material on bottom, and periodic metal sub-wavelength structures on top. Even though the bottom layers of metal and dielectric are indispensable for the structure, people usually tunes the top layer metallic pattern to

![Figure 4.6: (a) Geometric diagram of one unit of the metamaterial absorber with gold nanodisk on top layer. The diameter of the nanodisk is 180 nm. (b) SEM image of the metamaterial absorber with square-, triangular- and honeycomb-lattice gold nanodisks. For all the three kinds of absorbers, the distances between adjacent nanodisks are all the same as 310 nm.](image)
4.2. METAMATERIAL ABSORBERS WITH DIFFERENT LATTICES

change the absorption characteristics of the absorbers, and their focus is mainly on the shape, size of the structures, and little on the effects of different lattices of the sub-wavelength units on the absorption properties of the absorbers. We believe the inter-particle coupling and interaction may introduce different absorption characteristics due to different particle distributions. Hereby, we presents the simulation and experimental results of the absorption characteristics of our MIM-structure based absorbers, whose top layers are composed of gold nanodisks of same size but in different lattice distributions, including square, rectangular and honeycomb lattice distributions.

Figure 4.7: Measured absorption spectra of the absorbers in different lattices and polarizations in the yz incident plane: TE mode for square (a), triangular (b) and honeycomb (c) lattice absorber. TM mode for square (d), triangular (e) and honeycomb (f) lattice absorber.
CHAPTER 4. METAMATERIAL ABSORBERS AND PHOTOTHERMAL RESHAPING

Fig. 4.6(a) presents the unit structure of the three kinds of absorbers: the bottom two films are 80 nm thick gold and 28 nm thick alumina respectively, and the top layer is an array of gold nanodisks with the thickness of 30 nm and diameter of 180 nm. Fig. 4.6(b) demonstrates the SEM images of the absorbers with square, triangular and honeycomb lattices respectively, which all have the same distance of 310 nm between two adjacent nanodisks. To keep the uniformity of the gold particles, all three absorbers are fabricated on the same substrate. Here is the brief introduction of the fabrication process. First the gold and alumina films are deposited onto a silica substrate, then the nanodisk arrays of different lattices are patterned by EBL at the same time. After development, the top layer gold is deposited, followed by lift-off process, which finally reveal the absorber structures.

Fig. 4.6 illustrates the measured absorption spectra of the absorbers over a wide incident angle range for both polarizations in the yz plane. First, for the TE mode (Fig. 4.6(a), (b) and (c)), all three kinds of absorbers share strong angle-insensitive absorption at around 1140 nm, and the average bandwidths of this absorption peak for square-, triangle-, honeycomb-lattice absorbers are 220, 260 and 180 nm respectively. Besides, we don’t see very strong high-order mode in the spectra for the TE mode, which we will have further examination in the simulation part. As for the TM mode (Fig. 4.6(d), (e) and (f)), the absorbers also exhibit almost perfect light harvesting ability at around 1140 nm attributed to the fundamental resonance, with the average bandwidths of 230 nm, 250 nm and 180 nm for the square, triangular and honeycomb lattice absorbers, and the main difference appears in the high-order modes between honeycomb lattice absorber and the other two kinds of absorbers. Only the honeycomb lattice absorber possesses a strong angle sensitive, narrow-band high-order mode, which exhibits noticeable red-shift with increasing incident angle. Details about this high-order mode can be found in last section. Besides, all three kinds of absorbers have a similar high-order mode near 666 nm for the TM mode in large oblique incident angles.

Fig. 4.8 illustrates the simulation results of the absorption spectra of all three kinds of absorbers for both polarizations in the yz incident plane. The simulation is carried out in a commercial software COMSOL MULTIPHYSICS with the same structure parameter as the experimental realized absorbers shown above. The electricity permittivity data of alumina and gold are acquired from Paliks book [45], Johnson and Christy’s paper [46] respectively. Good agreement is found between the simulation and experimental results. Firstly, the fundamental mode achieves almost perfect absorption at around 1140 nm, regardless of the polarizations or incident angles. For TE mode, the bandwidths of the fundamental mode at normal incidence for square-, triangular- and honeycomb-lattice absorbers are 280, 285 and 228 nm respectively, while those for the TM mode are 267, 285 and 227 nm respectively. Thereby, for one thing, the simulation results is in accordance with the experimental results that triangular-lattice absorber has largest bandwidth for the fundamental mode, while honeycomb-lattice absorber has smallest one; for another, the measured bandwidths are smaller than the simulation counterparts, which we assume the reason is that the resonances between fabricated particles are not so
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Figure 4.8: Simulated absorption spectra of the absorbers with the particle size of 180 nm in different lattices and polarizations at the $yz$ incident plane: TE mode for square (a), triangular (b) and honeycomb (c) lattice absorber. TM mode for square (d), triangular (e) and honeycomb (f) lattice absorber.

strong as those in simulation conditions due to larger damping of the fabricated materials. While for the high-order mode, only honeycomb-lattice absorber has a red-shifting narrow-band mode in the short-wavelength range, but all three kinds of absorbers has a high-order mode near 640 nm in large incident angles.

According to both experimental and simulation results, the difference of gold nanodisk distribution has little influence on the positions of both the fundamental mode and the high-order mode at 640 nm (at about 666 nm for the experimental results) for TM mode. The stability of the fundamental and high-order mode can be attributed to their resonance nature, which mainly depends on localized surface plasmon resonance between singular gold nano-paritice on top and its image counterpart in the bottom gold film, little on inter-particle coupling. Even though the absorbers have different densities of gold nanodisks in $xy$ plane, they all have almost perfect absorption at 1140 nm, and this shows that every gold particle unit has a large absorption cross section.

As for the bandwidth difference of fundamental resonance between the absorbers, it can be explained by the difference of inter-particle coupling for different gold nanodisk distributions. As illustrated by the yellow arrows in Fig. 4.6 (b), every nanodisk in square lattice distribution has 4 nanodisks beside it with the same distance of 310 nm, while for triangular and honeycomb lattice, the number are 6 and 3 respectively, which means that the nanodisks in triangular lattice have strongest inter-particle coupling, and those in honeycomb lattice have the weakest.
Therefore, the fundamental mode in triangular-lattice absorber has largest bandwidth, and the honeycomb-lattice absorber has the smallest one. Actually, this phenomenon has been depicted and explained in many papers [50, 51] about metallic nano-particle clusters that aggregating of nanoparticles leads to more damping due to the enlarging volume of all particles and broadening of the bandwidth.

![Figure 4.9: Absorption spectra of the TM mode for the square lattice absorber with different periods at 60° incident angle from the \(xz\) plane with the same particle size of \(\phi = 180\) nm.](image)

In the meantime, the red-shifting narrow-band high-order mode is a reverse propagating SPP mode, which is excited by the incident wave with its in-plane wave vector added by a reciprocal lattice vector as explained in last section. Since the characteristics of this red-shifting mode is mainly determined by the lattice constant or the period of the particles in particular directions, it should be possible to introduce such an SPP mode to a square-lattice absorber by changing its period to produce a proper reciprocal lattice vector to meet the phase matching condition (equation 4.1). Thereby, we calculated the absorption spectra of the square-lattice absorbers with increasing periods of same gold nanodisks (\(\phi = 180\) nm) from 310 to 620 nm for the TM mode at 60° incident angle, and the result is shown in Fig. 4.9. When the period is 310 nm, no obvious narrow-band mode is observed. As the period increases to 485 and 510 nm, a narrow-band appears at 883 and 960 nm respectively, with the bandwidth of about 20 nm. Interestingly, with the period going up to 570 and 620 nm, instead of narrow-band peak, a asymmet-
ric steep hole appears in the fundamental mode, which can be interpreted as Fano resonance [52], an interference between the narrow-band high-order mode based on collective particle resonance and the fundamental mode based on singular particle resonance. Therefore, by means of increasing the period in square-lattice absorber, the narrow-band high-order mode is unveiled. Meanwhile, it should also be noted that the absorption of fundamental mode drops dramatically with respect to increasing period and thus sparser nanodisk distribution. Thereby, honeycomb-lattice absorber exhibits its advantage that it not only shows rich absorption characteristics with different incident angles and polarizations due to its different lattice symmetry, but also exhibits strong absorption from the fundamental resonance.

In conclusion, we compared the absorption characteristics of MIM metamaterial absorbers with top layer gold nanodisks distributed in different lattices, including square, triangular and honeycomb lattice. Stemming from the localized surface plasmon resonance in independent gold particles, the fundamental mode and the high-order mode at around 640 nm for TM mode experience no obvious change in their position and absorption efficiency. The lattice difference mainly brings in the variance of the bandwidth of the fundamental resonance and different high-order mode distribution. The bandwidth variance is resulted from the bandwidth broadening due to enhanced inter-particle coupling with aggregating of nano-particles. A narrow-band red-shifting high-order mode is unfolded in the honeycomb-lattice absorber, due to the interaction between the incident wave and specific reciprocal lattice vector momentum. However, by tailoring the period of the gold-disk distributions, square-lattice absorber can also exhibit such kind of high order mode with the introduction of proper reciprocal lattice vector. Interestingly, a Fano Resonance is observed with particular periods in square-lattice absorber, and this resonance originates from the interaction of the high-order resonance and the fundamental resonance.

4.3 Photothermal reshaping of metamaterial absorbers

Due to the optical response of surface plasmon polaritons, metallic nanoparticles possess enhanced absorption of light in visible and infrared wavelength range. Meanwhile, abundant ohmic heat is generated by the absorption in the nanoparticles, resulting in huge temperature increase or even nuclear rearrangements and permanent shape transformation of the nanoparticles. This thermal effects of plasmonic metallic nanoparticles, such as nanospheres [53], nanoshells [54] and nanorods [55, 56], have been widely investigated in recent decades, with potential applications in tumour treatment [55, 57], nano-welding [58, 59],in vivo imaging [60, 61], biological sensing [62, 63] and optical recording [56], and so on. However, most of the previous studies are focused on the photothermal effects of nanoparticles suspended in an aqueous environment. Here we experimentally demonstrate a photothermal effect of metamaterial nanoparticles in all-dry environment with an absorption wavelength around 1.6 \(\mu m\). After being illuminated by a pulsed
broadband supercontinuum source, the top-layer gold particles of the sample are reshaped from cuboids to spherical domes, with an improvement of the surface smoothness of the particle and blue-shift of the absorption peak of the absorber. Our demonstration may bring a leap for the potential application of photothermal effects in nano-science and nanotechnology.

![Figure 4.10](image.png)

Figure 4.10: Schematic diagram of the photothermal reshaping experiment. (a) Metamaterial absorber before reshaping. (b) The sample is irradiated with a broadband light source. (c) Metamaterial absorber after reshaping.

Fig. 4.10 illustrates the experiment flow. First a metamaterial absorber is fabricated by our standard procedure with electron beam lithography (EBL), which has been presented in previous chapter. The sample is fabricated on a 500 µm thick silica substrate, which is covered by a 60 nm thick gold film and 10 nm thick alumina film, and on top is 40 nm thick gold particles. The gold particles have the size of 170 × 230 nm, and distribute in a square lattice with a period of 310 nm. The fingerprint size of the absorber is 100 × 100 µm. With electromagnetic resonance between the gold particles and bottom gold film, the absorber has a strong absorption at around 1.6 µm. In our photothermal reshaping experiment, after the light beam from a super-continuum light source (ranging from 0.5–2.4 µm) is collimated by a collimator, and passes through a diaphragm and attenuator to control the intensity, the light beam is focused onto the sample by an achromatic lens with a beam size of 20 µm. Behind the sample there are a 20× objective and a CCD, working together to locate the sample and the beam. Fig. 4.10(c) is the schematic demonstration of the absorber after irradiation. The top layer gold particles are
4.3. PHOTOTHERMAL RESHAPING OF METAMATERIAL ABSORBERS

reshaped to spherical domes owing to surface tension in liquid phase with laser pulse irradiation.

Figure 4.11: (A) Top-view SEM image of the sample with both melted and unmelted particles. (B,C) Enlarged tilted view

Fig. 4.11 [43] shows the transformation of the gold nanoparticles from rectangular shape to spherical dome, after the sample is irradiated by a beam with a time-averaged power of 2.3 mW for about 0.2 second, and with the beam size of about 20 μm. In Fig. 4.10 (A), a region of the sample including both melted and original particles is presented, and there is a clear half circular boundary between the melted and unmelted part, which is caused the Gaussian beam profile. If incident power is further increased, damage can be caused to the sample, such as fragmentation of gold particles or even burning the sample in beam center, which is of less interest for us. In Fig. 4.10 (B) and (C), more details can be found with enlarged tilted view of the particles. Before irradiation, the particles have a grainy
surface, rough side edges and even lots of bumps on top of it, which is originated from the e-beam evaporation method of gold deposition and lift-off process during fabrication (Fig. 4.10 (B)). In contrast, the melted particles possess much smoother surface. By means of 3D atomic force microscope (AFM), the size of the particles is measured: the reshaped spherical particles have an average diameter of 160 nm and a height of 90 nm, with the contacting surface to alumina layer having a radius of 70 nm.

![Absorption Spectra](image)

**Figure 4.12:** Measured absorption spectra of the sample for both TM and TE polarizations at 10° incident angle before (A) and after (B) irradiation respectively. Simulated absorption spectra for the original (C) and melted (D) sample respectively. Insets are the top-view SEM images for a single particle.

The resonant wavelength is sensitive to the size and shape of the top layer gold nano-particles. Since there is a huge shape transformation of the gold particles as mentioned above, the absorption characteristics of the metamaterial absorber is examined. By means of our home-made transmission/reflection measurement setup, we obtain the absorption spectra of both the original and melted regions.
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at 10° incident angle, which are shown in Fig. 4.12 (A) and (B) [43] respectively, including both TM and TE polarizations. According to our previous study [42], the absorbance of our absorber is not sensitive to the incident angle. The incident plane intersects with the metamaterial in the direction along which the particle has a smaller edge. For the unmelted region, absorption peaks for the two polarizations are different, at 1.58 µm for TM mode and 2 µm for TE mode [28]. As for the melted area, the dome-shaped particles almost have same absorption peak for both polarizations at 1.1 µm, due to the high symmetry of the particles. The simulated absorption spectra are presented in Fig. 4.12 (C) and (D), which agree well with the measured results. Even for the high-order absorption peak at ~0.6 µm for the melted particles, the simulated and measured results have good conformity. The main difference is that the measured absorption peak has a larger bandwidth due to the fabrication tolerances of the side length of the particles.

In conclusion, we experimentally demonstrate a photothermal effect of our metamaterial absorber due to plasmonic resonance at near-infrared frequency, with the gold nano-particles reshaped from cuboids to spherical domes. As a result, the shape transformation also influences the absorption characteristics of the absorber, which is confirmed by both experiment and simulation. Our observation of gold particle reshaping promises a new potential method of fabricating dome-shape nano-particles or high quality metallic nano-particles for plasmonic and metamaterial structures [64, 65], such as chain waveguide (Fig. 4.13).

Figure 4.13: SEM image of chain waveguide obtained by photothermal reshaping.
Chapter 5

Plasmonic waveguides

5.1 Overview

Waveguide with sub-wavelength confinement of the optical mode is very crucial to realize high-density and compact photonic devices. While the minimum confinement of the optical mode in a dielectric waveguide is restricted by diffraction limit and is in the order half wavelength. There is always a great interest in designing and realizing waveguides with smaller sizes. Different from dielectric waveguide, a plasmonic waveguide can couple the incident light into a propagating free-electron collective oscillation wave, namely surface plasmon polariton (SPP), at a metal-dielectric interface, offering a promising approach to squeeze light into nanoscale regions smaller than the wavelength of light [66].

As an efficient way to manipulate light at nanometer scale, various kinds of plasmonic waveguides have been designed and fabricated, such as gold nanoparticle chain waveguide [67], metal wedges [68, 69], metal strips and slots [70], V-grooves in metal surfaces [71], metal nanowire [72, 73] and so on. And these waveguides can be utilized in a lot of potential applications, such as plasmonic routers and multiplexers [74], Bragg mirrors [75], interferometers [71], and electro-optic devices [76].

Although, plasmonic waveguides exhibit the advantage of sub-wavelength confinement of the field, they also have an Achilles’ heel: short propagation length compared to traditional dielectric waveguide, usually with µm order of propagation length. In order to overcome this weakness, scientists turn to various methods to improve the propagation length, such as by changing supporting substrate [77], utilizing gain dielectrics [78], working at a low temperature [79] or improving crystallinity [80] and so on. In our work, we also demonstrate a SPP-supported MIM stripe waveguide with sub-wavelength mode confinement. Meanwhile, by annealing the waveguide at 300 °C for 18 hours, the propagation length for 980 nm is improved significantly.
5.2 Plasmonic waveguides

As shown in Fig. 5.1(a), our waveguide is fabricated with a three-layer (MIM) structure on a silica substrate, with two layers of gold separated by a layer of alumina spacer, the thicknesses of which from top to bottom are 80 nm, 120 nm and 100 nm respectively. The width of the top layer gold stripe is 400 nm. Fig. 5.1(b) gives the normalized electric field distribution and transverse electric field flow of the fundamental mode at 980 nm, where we can see the electromagnetic field is confined in the dielectric layer in the sub-wavelength dimension. Fig. 5.1(c) is the SEM image of our 30 µm long stripe waveguide, fabricated by EBL.

Figure 5.1: (a) Schematic geometry of the waveguide. (b) Fundamental guided mode at 980 nm in the waveguide. The arrow surface shows the transverse electric field, and the color map represents the normalized electric field. (c) Top-view SEM image of the waveguides.
5.3 Annealing of waveguide

By means of our home-made propagation loss measurement setup, which has been shown in Fig. 3.2, we measured the propagation losses of the waveguide at 980 and 1550 nm after the sample is fabricated. Then the sample is annealed in an oven at atmospheric pressure at 300 °C for 18 hours. During the treatment, the sample is heated from and cooled back to room temperature (25 °C) slowly, which are about 1.5 and 0.5 °C/min respectively, to prevent dramatic thermal shocks to the sample. Then we remeasure the propagation losses of the waveguide at 980 and 1550 nm with the same method as above. The result shows in Fig. 5.2(a) and 5.2(b).

![Graph](image)

Figure 5.2: Measured propagation losses at 980 nm (a) and 1550 nm (b) before (brown circle) and after (cyan square) annealing at 300 °C for 18 hours. The solid line is the fitting line of the propagation distance versus intensity of the scattering light in logarithmic scale. (c) Optical microscope images of the taper and waveguide at bright- (top one) and dark-field from an infrared CCD with the excitation light at 980 nm.

As shown in Fig. 5.2(c), as the fiber taper touches the waveguide, the SPP wave
is excited in the waveguide and light is emitted in one end of the waveguide. As the taper moves backwards, the SPP wave propagates for a longer distance and the emission intensity decreases. By extracting the intensity decay from the optical image, the propagation loss can be calculated. Fig. 5.2(a) presents the measured propagation losses at 980 nm before and after annealing at 300°C for 18 hours. We can see that the waveguide experiences a more than 50% drop in propagation loss, from 0.45 to 0.20 dB/μm. As for 1550 nm, only slight improvement is detected, from 0.31 to 0.28 dB/μm (Fig. 5.2(b)).

We believe that the increase of the propagation length is resulted from the improvement of the gold quality after annealing, including the flattening of the gold film surface and the enlargement of the gold particle size, which agrees well with existing experimental evidences of X-ray diffraction pattern [81], TEM photos [82] and SEM photos [83, 84]. Meanwhile, The gold film morphology change in our waveguide is also examined by the SEM images (Fig. 5.3), where we see that after annealing at 300°C for 18 hours (Fig. 5.3(b)), the surface of the top layer gold film of the waveguide becomes much flatter and the gold particle size also increases significantly. Besides, the inside part of the gold layers could also undergo potential quality improvement which also impart some contributions to the increase of propagation length.

As mentioned above, we use e-beam evaporation to deposit the gold layers onto the substrate, and the deposition process can break into several phases[82, 85]. In the first beginning, evaporated gold particles hit the substrate and aggregate into individual gold nucleus. With deposition going on, nuclei grow into larger grains by way of impingement or surface diffusion of single gold atoms. In the next phase for the discontinuous film, individual grains mainly undergo liquid-like coalescence. Finally, as gold film thickness increases, the islands grow large enough to connect to each other and a continuous film is formed. As the sample is annealed at 300°C in the oven, surface mobility of the gold atoms increases, and gold grains undergo further “coalescence” [86, 87], with gold atoms moving between grains to minimize
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the surface energy of the system. Thereby, smaller grains grow or merge into bigger grains via surface diffusion. As a result, the quality of gold layers is improved by means of the enlargement of the grain size and reduction of film surface roughness during coalescence [83, 84]. It has good agreement with the results shown in our SEM images.

Encouraged by the propagation loss improvement, further annealing experiment is carried out by pushing the annealing temperature to 400 °C for 18 hours, with slow heating and cooling rate maintained. None clear change is noticed from both the SEM photo of the strip waveguide and the measured propagation loss at both wavelengths, compared to the results after annealing at 300 °C for 18 hours. However, when the annealing temperature is pushed to 500 °C (for 18 hours), severe deformation of the strip waveguide is noticed: different parts of the gold strip experience different levels of width shrinking (Fig. 5.3(c)). The measured propagation loss suffers significant increase due to the distortion of the waveguide.

In conclusion, we present an MIM plasmonic waveguide with sub-wavelength confinement of the fundamental mode in telecommunication wavelengths. By annealing the waveguide with appropriate temperature conditions, the propagation loss is reduced significantly due to the improvement of the gold quality in the waveguide. This thermal annealing method may promise an approach to improve the quality of other fabricated plasmonic devices.
Chapter 6

Summary, and future work

In summary, this thesis presents nanofabrication and characterization of plasmonic devices, such as absorbers and waveguides. Here are the main results we have achieved:

1. Utilizing the EBL technique, we are capable of fabricating different kinds of MIM-structure based plasmonic absorbers, which not only exhibit almost perfect absorption at fundamental resonance regardless of polarizations and incident angles at near-infrared frequency domain, but also show rich high-order mode resonance characteristics related to different lattice distributions of top layer nano-particles. It shows versatile application potentials such as thermal emitters, microbolometers, sensors and so on.

2. Besides plasmonic absorbers, other plasmonic nanostructures such as waveguides, plasmonic quarter-wave plate are also realized by means of our nanofabrication technique.

3. We demonstrate two home-made optical characterization setups: angle-resolved transmission/reflection measurement setup and nanowire propagation length measurement setup. The former setup is capable of measuring the absorption spectra of plasmonic absorbers with a fingerprint size as small as $100 \times 100 \mu m$ or even $20 \times 20 \mu m$, including both polarizations, incident planes and the incident angle range from 0 to 60°. In addition, this setup can be adapted to a complex photothermal reshaping setup with the introduction of a pulse generator. The latter setup is able to measure the propagation loss of different kinds of sub-wavelength waveguides.

4. Photothermal effects are utilized to reshape our gold nanoparticles from nanobricks to nanodomes, which unfolds a promising approach to fabricate dome-shape or spherical metallic nanostructures with controllable size and distribution of the particles, such as chain waveguide, etc. Besides, thermal annealing could
also effectively change the shape or even inner structure of gold nanostructures to alleviate damping or loss of the metallic materials and thus improve the quality of the gold nanostructures.

As for the future work, there are several topics and issues we are interested in:

1. Our plasmonic absorbers based on MIM structure have achieved almost 100% absorption in the fundamental absorption peak based on localized surface plasmon, which means they have the potential to be used in thermal-photovoltaic applications. A thermal-photovoltaic system usually consists of two parts, thermal emitter and photovoltaic diode. I would like to do some research on how to adapt our absorbers into an effective emitter to work together with different kinds of photovoltaic diodes, and realize the thermal-photovoltaic function.

2. By means of our photothermal reshaping experiment setup, we are capable of fabricating spherical nano-particles with controllable particle size and distribution. One of the potential application is to fabricate chain waveguide. It would be interesting to investigate the near-field interaction between the particles and the propagation of SPP waves along the chain waveguide.

3. Most of our experiments are carried out in far field conditions. It would be interesting to do some experiments about the near-field of SPPs in our plasmonic absorbers and waveguides. For example, we could utilize a scanning near-field optical microscope (SNOM) to analyze the field distribution of the absorbers and the wave propagation in plasmonic waveguide.
Chapter 7

Guide to papers

I  Yiting Chen, Jin Dai, Min Yan, and Min Qiu, “Influence of lattice structure on metal-insulator-metal plasmonic absorbers”, manuscript planned for Optics Express.

Author’s contribution: I performed the fabrication, characterization and part of the simulation of the plasmonic absorbers with different lattices. I finished the first draft of the manuscript.


Author’s contribution: I performed the fabrication, characterization and part of the simulation of the honeycomb lattice absorber. I finished the first draft of the manuscript.


Author’s contribution: I performed the fabrication, thermal annealing, characterization and the simulation of the strip waveguide. I finished the first draft of the manuscript.


Author’s contribution: I performed the photothermal reshaping and characterization of the metamaterial absorber.

Author’s contribution: I measured the absorption spectra of both samples with the angle-resolved transmission/reflection experiment setup established by myself.


Author’s contribution: I performed the photothermal reshaping experiment and characterized the sample.
Bibliography


