# NDnano Summer Undergraduate Research
# 2022 Project Summary

1. Student name & home university:

Aldo Celis Cordova, Monterrey Institute of Technology and Higher Education

2. ND faculty name & department:

Dr. Edward Kinzel, Aerospace & Mechanical Engineering

3. Summer project title:

Scalable nanofabrication of metasurfaces using microsphere photolithography

4. Briefly describe new skills you acquired during your summer research:

Throughout my stay, I used the nanofabrication facility and the laser laboratory to create nanoscale patterns. In the cleanroom, I became familiar with photolithography and wafer fabrication. I developed samples using an Electron Beam Evaporator (“FC1800 Evaporator”) and later process it by using lasers. In the laser laboratory, I learned the how to do microsphere photolithography (MPL). Finally, for analyzing the results, I manage to operate an FTIR for getting transmittance and use “Magellan 400 FESEM” for SEM imaging.

5. Briefly share a practical application/end use of your research:

Nanofabrication is widely used for crating sensors and electronical devices, and new opportunities arise in creating metasurfaces. Yet, the process requires of expensive machines to create desired nanoscale patterns, as well as it doesn’t scale up. Luckily, microsphere photolithography grants us a cost-effective process for nanofabrication and shows a promising future to the different applications in metasurfaces, such as sensing and color patterning.

6. 50- to 75-word abstract of your project:

Creating a nanoscale hole array, for IR sensors, can be expensive when conventional E-beam Lithography or Focused Ion-beam Milling are used. Therefore, microspheres are exposed to UV light to act as a microlens and, as a result, achieve nanoscale photolithography. This new technique can significantly reduce the cost for nanofabrication and can scale up the process (wider areas can be exposed with a single exposure).

7. References for papers, posters, or presentations of your research:

[1] C. Qu and E. C. Kinzel, “Polycrystalline metasurface perfect absorbers fabricated using microsphere photolithography,” Optics Letters 41, 3399-3402 (2016).
[2] C. Zhu, C. Qu, and E. Kinzel, "Direct-write microsphere photolithography of hierarchical infrared metasurfaces," Applied Optics 60, 7122-7130 (2021).

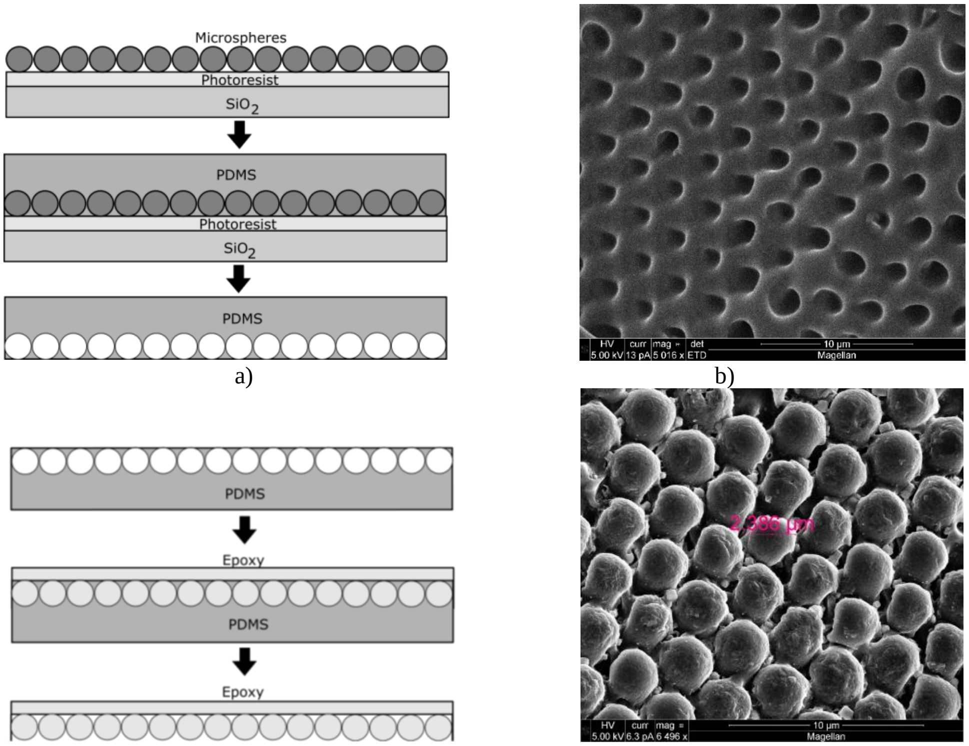
[3] I. Jasim, J. Liu, C. Zhu, M. Roman, J. Huang, E. Kinzel, and M. Almasri, “Microsphere Photolithography Patterned Nanohole Array on an Optical Fiber” (9), 32627–32630 (2021).

[4] R. A. Rahman, T. Uenohara, Y. Mizutani, and Y. Takaya, “First Step Toward Laser Micromachining Realization by Photonic Nanojet in Water Medium,” Nanotechnology, 492-500 (2021).

One-page project summary that describes problem, project goal and your activities / results:

Microsphere lithography can make nanofabrication of metasurfaces cheaper and scalable to pattern wide areas. The process consists in depositing the microspheres on the surface of DI water. Due to capillary forces, the microspheres will rearrange themselves to create a Hexagonal Close-Packed (HCP) array. Afterwards, a substrate can be slowly pulled out of the DI water, and the microspheres will stay on the substrate's surface. When exposed to UV light, the microspheres create a photonic jet that result in a nanohole pattern on the layer below. However, the microspheres must be removed, making them not reusable.

The goal was to create a reusable microsphere HCP pattern for photolithography. The 3 µm microsphere pattern can be copied using PDMS polymer as a casting material. After, the original microspheres can be removed using acetone, leaving the PDMS cast. Finally, to fill the PDMS cast, an Epoxy polymer is used to recreate the microspheres HCP pattern, as shown in *Fig.1 d)*.



a) b)

c) d)

Fig. 1 a) Scheme diagram of the casting of PDMS to copy the microsphere pattern. b) SEM image showing the PDMS cast with the holes once the original microspheres were removed. c) Scheme diagram of Epoxy polymer used to recreate the microspheres. d) SEM image showing the copied HCP pattern form the microspheres.

As seen in the *Fig.1 d)*, this simple process can recreate the microsphere HCP pattern successfully, however improvements can still be sought to make perfect circular microspheres. As the Epoxy polymer cures/hardens, there is shrinkage on the microspheres (compared with the original's size), this causes imperfections that affect lithography.

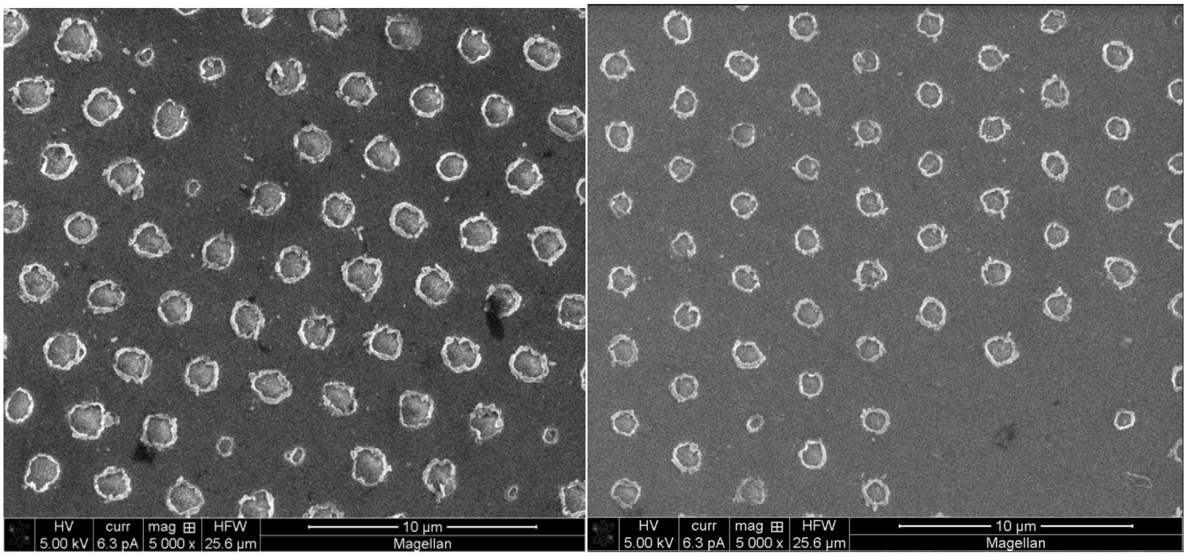
Another promising opportunity rises with the photonic jet caused by microspheres, instead of using photoresist to pattern the nanoholes, it can be used to etch a metal. The experiments consisted of self-assembling the microspheres on top of a 50nm Aluminum sample. Then a femtosecond laser was used to irradiate the microspheres and do micromachining to the Aluminum below them. The same nanohole pattern, as normal microsphere lithography will be created, but as it micromachines a metal, its transmittance can be changed.

In the experiments, the laser was set to have a 1030nm wavelength with a spot size of 100 µm. Multiple shots were made to the sample to verify where micromachining was more optimal (to achieve the smallest nanohole size). I found through multiple testing that Al micromachining can be achieve with low fluences $\left(\frac{Laser's\ Pulse\ Energy}{Samples\ Area}\right)$. Phantos femtosecond laser allowed us to reach a minimal pulse energy of 1.5µJ. However, a consistent micromachining was possible at 2µJ. The number of shots did not change nanohole size greatly, therefore I proceeded to scan through the sample to see if figure drawing is possible.

Successfully, scanning achieves the creation of 2 squares filled with nanoholes. A super low fluence (2.546 mJ/cm$^2$) was tested and no micromachining was achieved. The fluences were chosen after seeing 2 promising micromachining. *Table 1* shows the laser's parameters in the scanned experiment, and Fig. 2 shows the results.

**Table 1. Sample 3 Fluence and Nanohole Diameter**

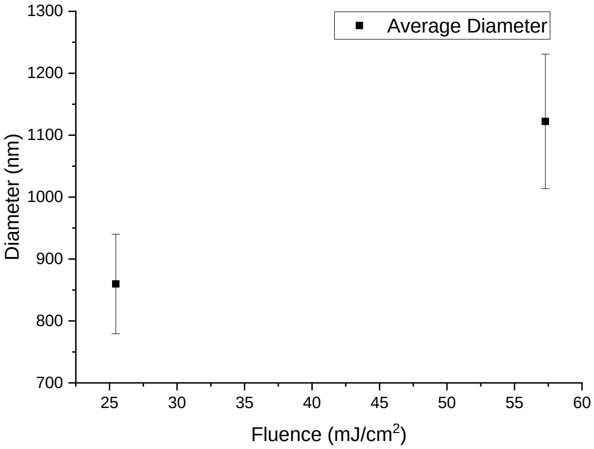
| Square scans | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Scan** | **Laser Parameters** | | | | | **Nanohole** | | |
| | **Spot Size (µm)** | **$F_p$ (kHz)** | **$N_p$** | **$E_p$ (µJ)** | **Wavelength (nm)** | **Average diameter (nm)** | **Standard Deviation** | **Fluence (mJ/cm$^2$)** |
| 1 | 100 | 2 | 2,000 | 4.5 | 1030 | 1122.191953 | 80.2990547 | 25.46 |
| 2 | 100 | 2 | 2,000 | 2 | 1030 | 859.8236842 | 108.614188 | 57.29 |
| 3 | 100 | 2 | 2,000 | 0.2 | 1030 | — | — | 2.5464 |



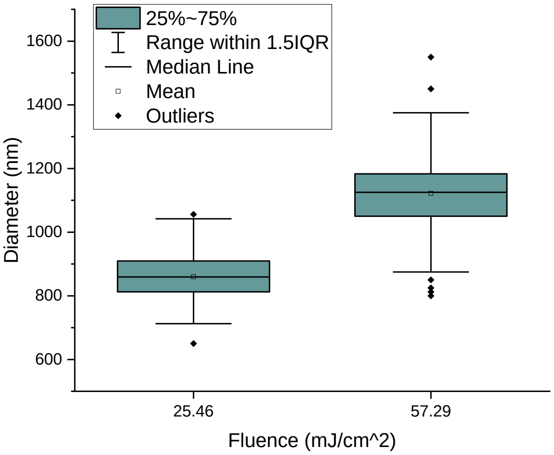
a) b)

Fig. 2 SEM images form a) Scanned sample with 57.29 mJ/cm$^2$, and b) Scanned sample with 25.46 mJ/cm$^2$.

So far, micromachining with this fluence and setup achieve varying nanohole size with debris. As the photonic jet ablates the metal, this heats up and resolidifies on the edges of the nanohole. This causes problems with consistent nanohole size that can be seen on Plot 1 and 2.



Plot. 1 Consistency of nanohole diameter size according to fluence.



Plot. 2 Nanohole diameter size according to fluence.

There seems to be a promising chance to achieve low enough transmittance for visually seeing colors from the Aluminum layer. More work must be done for achieving smaller nanohole diameter size (down to ~150nm), which can be achieved with shorter wavelengths and smaller microspheres.