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14. ABSTRACT Funding for a Nanoscribe Photonic Professional GT 3D printer was requested for the fabrication of nanostructures, especially those designed as metamaterials for applications in the IR. This 3D printer also supports the development of inverse methods for improved metamaterial and scattering target designs. Currently there are 100 Nanoscribe systems installed worldwide. The instrument uses a two-photon absorption process to expose uv resist and thresholding allows ~ 100nm linewidths to be written if used with care. The laser in the system operates with a wavelength of 700nm and 100fs pulses with an 80MHz pulse repetition frequency. Several resist or resins are					
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a. REPORT	b. ABSTRACT	c. THIS PAGE			Michael Fiddy
UU	UU	UU	UU		19b. TELEPHONE NUMBER 704-687-8594

Report Title

Final Report: 3D Nanostructure Design and Fabrication

ABSTRACT

Funding for a Nanoscribe Photonic Professional GT 3D printer was requested for the fabrication of nanostructures, especially those designed as metamaterials for applications in the IR. This 3D printer also supports the development of inverse methods for improved metamaterial and scattering target designs. Currently there are 100 Nanoscribe systems installed worldwide. The instrument uses a two-photon absorption process to expose uv resist and thresholding allows ~ 100nm linewidths to be written if used with care. The laser in the system operates with a wavelength of 790nm and 100fs pulses with an 80MHz pulse repetition frequency. Several resists or resins are available in which this two-photon polymerization process can be exploited for high speed and high resolution 3D printing. Ongoing research at UNCC through the CfM has been the development of new polymer-based resists, incorporating 4 to 5nm sized nanospheres which allows one to change the base refractive index from which the structures are made. This provides another dimension of flexibility for applications of the Nanoscribe.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
01/31/2017	2 Daniel B. Fullager, Glenn D Boreman, Tino Hofmann. Infrared dielectric response of Nanoscribe IP-Dip and IP-L monomers after polymerization from 250 cm ⁻¹ to 6000 cm ⁻¹ , Thin Solid Films, (): . doi:
TOTAL:	1

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
TOTAL:	

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Presented at NSF IUCRC Meeting in October 2016

Number of Presentations: 1.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

08/28/2015 1.00 Morteza Karami, Christopher Rosenbury, Steven Kitchin, Michael A. Fiddy. Negative-index polarization-independent metamaterial, CLEO 2015. 11-MAY-15, . : ,

TOTAL: 1

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

None

Patents Awarded

None

Awards

None

Graduate Students

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

<u>NAME</u>
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>
Total Number:

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See attachment

Technology Transfer

None

W911NF1510381 3D Nanostructure Design and Fabrication

Prepared by M. A. Fiddy January 26th 2017

Introduction

Funding for a Nanoscribe Photonic Professional GT 3D printer was requested for the fabrication of nanostructures, especially those designed as metamaterials for applications in the IR. This 3D printer also assists with the development of inverse methods for improved metamaterial and scattering target designs.

Instrument details

Award total: \$542,662

✓ Laser lithography system 'Photonic Professional' according to the data sheet 'Photonic Professional';	NA	1/EA	317,459.00 USD	1 EA	317,459.00 USD
✓ Photonic Professional Option GT:Ultra-fast galvo-scanner based scan head with scanning control unit and analog control, rapid x-y-scanning for two- photon polymerization, full embedding in NanoWrite 	NA	1/EA	77,916.75 USD	1 EA	77,916.75 USD
✓ fs-fiber laserPulse length < 120 fsRepetition rate: 80 MHz ± 1MHz Average power > 120 mWCenter wavelength: 780 nm ± 10 nm 	NA	1/EA	64,313.00 USD	1 EA	64,313.00 USD
Motorized scanning stage with writing field of up to 100 x 100 mm2 	NA	1/EA	35,910.00 USD	1 EA	35,910.00 USD
High-sensitivity microscope camera; 1388(H) x 1038(V) = 1.4 Mega Pixel; color; 4.65 µm x 4.65 µm; chip size: 1/2" 	NA	1/EA	13,113.00 USD	1 EA	13,113.00 USD

Immersion objective; 63x; NA = NA 1.4;WD = 190 µm; appropriate for fixed laser focus and focus scanning 	1/EA	8,197.00 USD	1 EA	8,197.00 USD
Immersion objective; 25x; NA NA =0.8;WD = 380 µm; appropriate for fixed laser focus and focus scanning 	1/EA	8,616.00 USD	1 EA	8,616.00 USD
✓ Air objective; 20x, NA=0.5, NA WD=2.1 mm; appropriate for fixed laser focus and focus	1/EA	3,807.00 USD	1 EA	3,807.00 USD
✓ Active self-leveling isolation NA frame for installations in clean rooms 	1/EA	7,493.00 USD	1 EA	7,493.00 USD
✓ Sample holder: 10 x 30 mm Ø; NA aluminium; anodized; sample thickness: 170 µm 	1/EA	603.00 USD	1 EA	603.00 USD
Sample holder DiLL; aluminium; NA anodized; for sample dimensions (45-76) x (19-26) mm ² ; rectangular; substrate thicknessST = 1.0 mm; 30 mm Ø; ST = 0.17 mm;25 x 25 mm ² ; rectangular; ST = 0.7 mm; 11- 25.4 mm Ø; ST = 0.3 mm 	1/EA	434.00 USD	1 EA	434.00 USD
NA	1/EA	917.00 USD	1 EA	917.00 USD
Stainless steel working desk for placement of screen, keyboard, mouse etc.; 120 x 80 cm ² 				

Instrument total: \$538,778.75
Delivery charges: \$2121.04
Services: \$1762.20

The Nanoscribe's capabilities

The Nanoscribe can write features down to 120nm. Each tool is custom built and costs 433,000 Euros (~\$542,000). Ours was shipped to the US from Germany in the Spring of 2016 but was held in customs for many months due to an issue over payment of import duty. This was

eventually resolved and the instrument delivered and installed August 4th to 9th 2016. The kick-off and training meeting with the vendor was held at UNCC on August 5th 2016. This late date left very little time for results from the Nanoscribe to be obtained and reported relating to the above project concepts, and is why this final report is being submitted late.

At the kick-off and training meeting, many faculty, students and some local company representatives attended. The acquisition of the Nanoscribe had been conveyed to the members of the NSF I/UCRC Center for Metamaterials (CfM) which is led by UNC Charlotte.

Currently there are 100 Nanoscribe systems installed worldwide. The instrument uses a two-photon absorption process to expose uv resist and thresholding allows $\sim 100\text{nm}$ linewidths to be written if used with care. The laser in the system operates with a wavelength of 790nm and 100fs pulses with an 80MHz pulse repetition frequency. Several resists or resins are available in which this two-photon polymerization process can be exploited for high speed and high resolution 3D printing. Ongoing research at UNCC through the CfM has been the development of new polymer-based resists, incorporating 4 to 5nm sized nanospheres which allows one to change the base refractive index from which the structures are made. This provides another dimension of flexibility for applications of the Nanoscribe.

Many faculty at UNCC who work on nanoscale device fabrication (see <http://opticscenter.uncc.edu>) have arranged to use the Nanoscribe and so its impact on several projects is clear. These include faculty members such as Dr. Glenn Boreman, Dr. Ish Aggawal, Dr. Tom Suleski, Dr. Tsing-hua Her, Dr. Menelaos Poutous, Dr. Ed Stokes and Dr. Marcus Jones and over 20 funded graduate students. A new faculty member, Dr. Tino Hofmann has a particular interest in and need for the Nanoscribe. His expertise is in developing ellipsometer techniques for measuring the permittivity or index tensor associated with highly anisotropic thin films. The Nanoscribe can write form-birefringent structures with great precision which, for IR ellipsometry can be modeled through effective medium approximations to determine the equivalent permittivity tensor components. Controlled writing of such structures allows immediate improvements to be made in both effective medium modeling for structured materials as well as ellipsometer model fitting methods. This approach also serves as the foundation for being able to develop more accurate methods for measuring the effective medium properties of metamaterials in general.

The Nanoscribe will support on-going projects funded by ARO and we note that the NSF I/UCRC Center for Metamaterials (centerformetamaterials.com) has projects supported by ARO, ARL Adelphi, ARL CERDEC and AFRL as well as several defense contractors such as Raytheon.

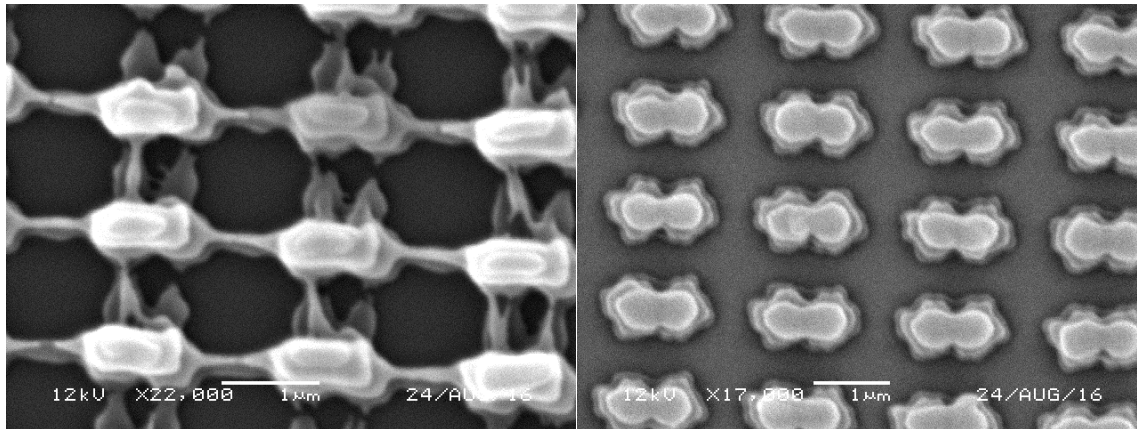
Progress to date

In the first few months of having the instrument, the following have been successfully completed:

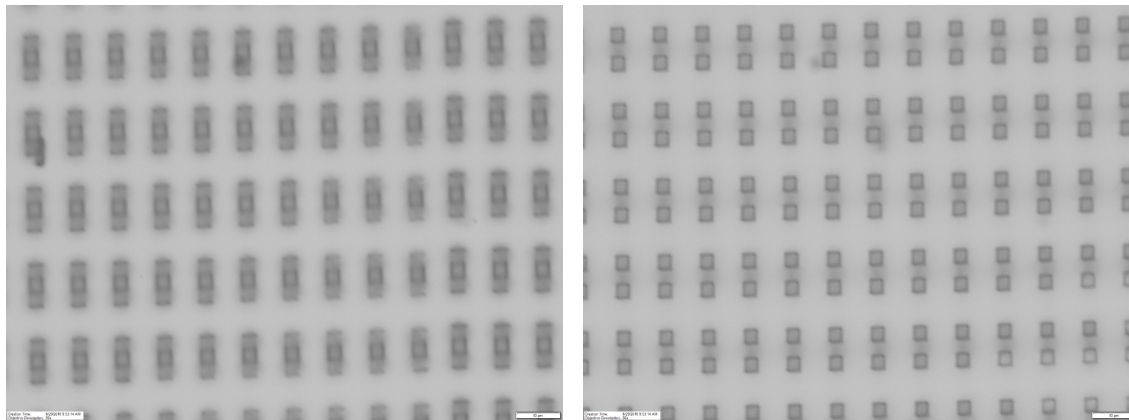
- 1) Installed and verify proper operation
- 2) Demonstrate the ability to write on semiconductor and metal substrates

- 3) Rapid prototyped an alignment device for a SNOM
- 4) Demonstrated the ability to write naked-eye-visible arrays from nanoscale features
- 5) Constantly improved stitching ability for larger structures
- 6) Developed structures for collaboration with other groups working with plasmonic and dielectric resonators, microspheres, free space vortices, infrared metasurfaces etc.

Initial attempts at creating sub-micron features demonstrated that process parameters needed to be improved, for example with these vertical split ring resonator structures:

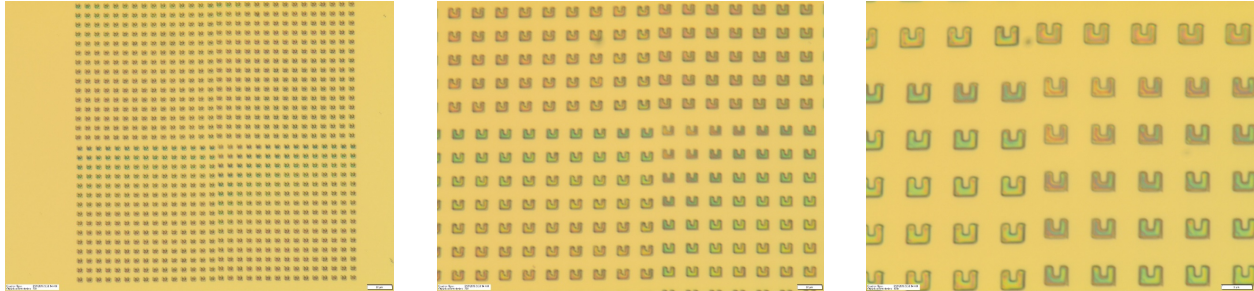


The first iteration is seen on the left and the second on the right after adjusting the write speed.

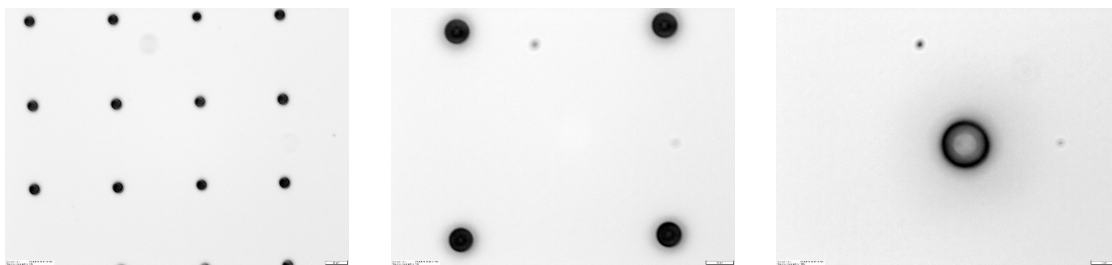


Identical frames at different depth of focus reveal that it is much easier to produce uniform arrays for characteristic features larger than $\sim 700\text{nm}$

Flat split ring resonator arrays are more straightforward to write:

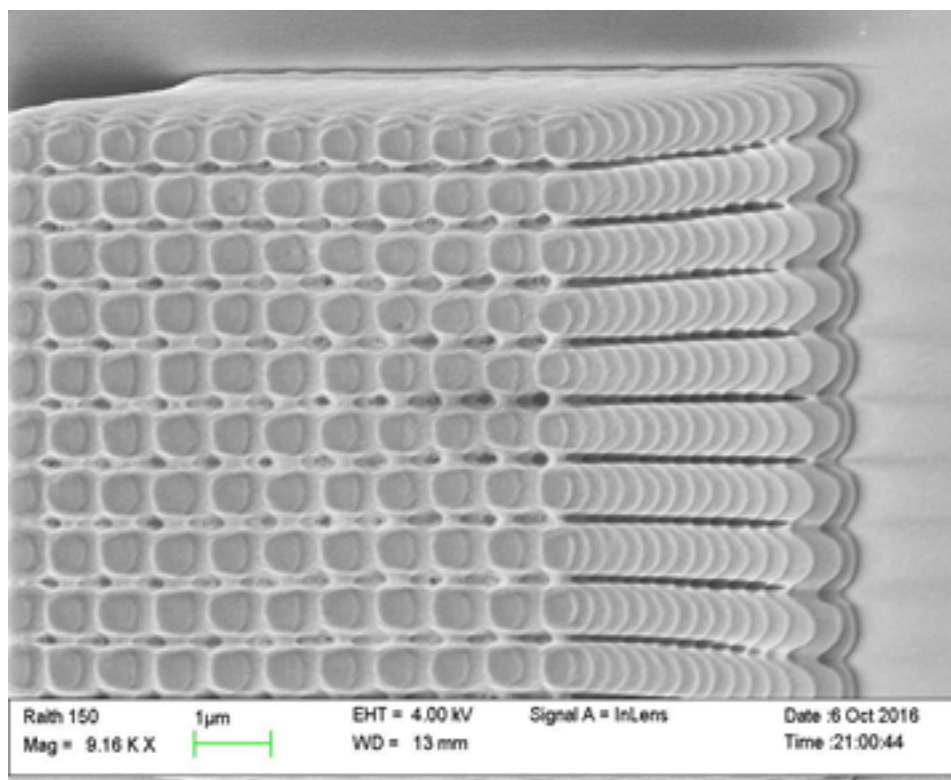


The ability to make large scale arrays of uniform microspheres is considered to be a disruptive technology for enhancing focal plane arrays in IR imaging systems (parametrically modulated type-III strained layer superlattice photodetectors).



Figures show microsphere arrays with 25, 10 and 5 scales respectively.

An example of a 3D structure written using the newly installed Nanoscribe is shown below. It is an image obtained using our Raith 150 e-beam system. A scale of one micron is indicated and the image is of a series of nanospheres written one on top of the another in an array of rows and columns. Writing nanospheres with this precision and being able to return and write more with such excellent positional accuracy was considered to be one of the important and early test structures to assess the overall capability of the instrument. While straightforward to use in principle, the training to become proficient in its use to make complex structures such as this is a matter of weeks rather than days. The Nanoscribe can write structures by translating most CAD design files into its own file format. The piezo stage allows volumes as large as 300 microns cubed to be written with 5nm return positional accuracy. The writing beam is stationary and the resist volume is moved. In one mode liquid resists can be used in a lens immersion configuration, requiring great care in post-use cleaning of the objective lens. In another writing mode solid resists can be used. An important and attractive feature of this instrument is that one can write structures in materials that can then serve as molds for mass replication of additional structures. Highly complex large structures can take several hours to write and removing unexposed resist from the final structure also needs to be done with care.



Further details about the instrument and its use can be obtained from Dr. Hofmann while I am on leave of absence at DARPA

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