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14. ABSTRACT
With this DURIP, we will establish a world-class research facility in advanced optical fiber characterization for high power fiber lasers. This unique capability will open up opportunities for us to assert leadership in the important strategic area of defense fiber laser technology.
The development of ultra large mode area fibers suitable for high power lasers is an outstanding challenge, requiring improvements in structural and material composition characterization during preform and fiber manufacturing. The proposed tabletop SEM-EDS is a vital diagnostic instrument for real time analysis during fiber

15. SUBJECT TERMS
Microstructured optical fibers, fiber characterization, fiber optics amplifiers and photonic crystal fiber

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UU	UU	UU	UU		Rodrigo Amezcua Correa / 407-823-6853

Report Title

Final Report: High-Resolution Imaging and Material Composition Analysis Facility for High Power Fiber Lasers

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The development of ultra large mode area fibers suitable for high power lasers is an outstanding challenge, requiring improvements in structural and material composition characterization during preform and fiber manufacturing. The proposed tabletop SEM-EDS is a vital diagnostic instrument for real time analysis during fiber production. It will allow us to deterministically make improvements to our fiber development by fine-tuning of the many process parameters in real-time during fiber draw. Both the yield of the fiber manufacturing process and fiber performance will be greatly enhanced by this on-site characterization method. Provision of this instrument will put us on a par with European laboratories and will allow us to exploit the full potential of large mode areap photonic crystal fibers (PCFs) for the development of the next-generation of high average power and high peak power fiber lasers and fiber amplifier systems.

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)

Received		Paper
07/06/2017	1	Amy Van Newkirk, J. E. Antonio-Lopez, James Anderson, Roberto Alvarez-Aguirre, Zeinab Sanjabi Eznaveh, Gisela Lopez-Galmiche, Rodrigo Amezcua-Correa, Axel Schülzgen. Modal analysis of antiresonant hollow core fibers using S^2 imaging, Optics Letters, ( ): 3277. doi: 1,049,902.00
TOTAL:	**1**	

Number of Papers published in peer-reviewed journals:

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

Received	Paper
TOTAL:	

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

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

Received	Paper
07/06/2017	2 Amy Van Newkirk, J. E. Antonio -Lopez, James Anderson, Roberto Alvarez -Aguirre, Rodrigo Amezcua -Correa, Axel Schülzgen. Higher Order Modes in Anti-Resonant Hollow Core Fibers, Specialty Optical Fibers. 01-JUL-16, Vancouver. : ,

TOTAL: 1

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

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

Received	Paper

TOTAL:

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

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Names of Under Graduate students supported

NAME PERCENT_SUPPORTED

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

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See attachment

Technology Transfer

1) **Period covered by report**: August 15, 2015 to July 31, 2016
2) **Proposal Title**: DURIP: HIGH-RESOLUTION IMAGING AND MATERIAL COMPOSITION ANALYSIS FACILITY FOR HIGH POWER FIBER LASERS
3) **Agency Grant Number:** W911NF-15-1-0338
4) **Author of Report:** Rodrigo Amezcua-Correa
5) **Performing Organization Name and Address:**
   CREOL –The College of Optics and Photonics, University of Central Florida. 4000 Central Florida Blvd. Orlando, FL 32816-2700.

DURIP: HIGH-RESOLUTION IMAGING AND MATERIAL COMPOSITION ANALYSIS FACILITY FOR HIGH POWER FIBER LASERS

Final Report

by: Rodrigo Amezcua Correa (PI)
Lawrence Shah (co-PI)
Axel Schülzgen (co-PI)
Martin Richardson (co-PI)

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ABSTRACT (from proposal)

With this DURIP, we will establish a world-class research facility in advanced optical fiber characterization for high power fiber lasers. This unique capability will open up opportunities for us to assert leadership in the important strategic area of defense fiber laser technology.

The development of ultra large mode area fibers suitable for high power lasers is an outstanding challenge, requiring improvements in structural and material composition characterization during preform and fiber manufacturing. The proposed tabletop SEM-EDS is a vital diagnostic instrument for real time analysis during fiber production. It will allow us to ***deterministically make improvements to our fiber development*** by fine-tuning of the many process parameters in real-time during fiber draw. Both the yield of the fiber manufacturing process and fiber performance will be greatly enhanced by this on-site characterization method. Provision of this instrument will put us on a par with European laboratories and will ***allow us to exploit the full potential of large mode areap photonic crystal fibers (PCFs)*** for the development of the next-generation of high average power and high peak power fiber lasers and fiber amplifier systems.

Keywords: Microstructured optical fibers, fiber characterization, fiber optics amplifiers and photonic crystal fiber

Contents

The Report Documentation Page

(1) Submissions or publications under ARO sponsorship during this reporting period. List the title of each and give the total number for each of the following categories:

(a) Papers published in peer-reviewed journals (1)

1) **"Modal analysis of antiresonant hollow core fibers using S2 imaging"**, A. Van Newkirk, J. E. Antonio-Lopez, J. Anderson, R. Alvarez-Aguirre, Z. Sanjabi Eznaveh, G. Lopez-Galmiche, R. Amezcua-Correa, and A. Schülzgen Optics Letters Vol. 41, Issue 14, pp. 3277-3280 (2016).

(b) Papers published in non-peer-reviewed journals
N/A

2) **Presentations (1)**

i. Presentations at meetings, but not published in Conference Proceedings

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

iii. Peer-Reviewed Conference Proceeding publications (other than abstracts): 1

1) **"Higher order modes in anti-resonant hollow core fibers"**, A. Van Newkirk, J. E. Antonio-Lopez, J. Anderson, R. Alvarez-Aguirre, R. Amezcua-Correa, and A. Schülzgen, OSA Specialty Optical Fibers, Vancouver Canada, July 2016, paper SoM3F.4

(d) Manuscripts:
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(e) Books
N/A

(f) Honor and Awards
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(g) Title of Patents Disclosed during the reporting period
N/A

(h) Patents Awarded during the reporting period
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(2) Student/Supported Personnel Metrics for this Reporting Period

(a) Graduate Students:
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(b) (b) Post Doctorates:
N/A

(c) Faculty:
N/A

(d) Undergraduate Students
N/A

(e) Graduating Undergraduate Metrics (funded by this agreement and graduating during this reporting period):
N/A

i. Number who graduated during this period:
N/A

ii. Number who graduated during this period with a degree in science, mathematics, engineering, or technology fields:
N/A

iii. Number who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields
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iv. Number who achieved a 3.5 GPA to 4.0 (4.0 max scale)
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v. Number funded by a DoD funded Center of Excellence grant for Education, Research and Engineering
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vi. Number who intend to work for the Department of Defense
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vii. Number who will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields
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(f) Masters Degrees Awarded:
N/A

(g) Ph.D.s Awarded:
N/A

(h) Other Research staff:
N/A

(3) "*Technology* transfer"

N/A

(4) Scientific Progress and Accomplishments

See page

(5) "Copies of technical reports"

N/A

Scientific Progress and Accomplishments

1. Summary

This grant enabled the purchase of critical equipment for fiber optical fiber characterization and development. This grant builds upon DoD support in other programs, such as the ARO/JTO funded MRI on “Fiber Laser Light Engines - A New Platform to Collectively Address Power-limiting Constraints” (grant #: W911NF-12-1-0450), ARO DURIP – “Advanced Splicing Facility for High Power PCF Laser Fabrication” (grant # W911NF-13-1-0283) and AFSOR DURIP grant “MCVD lathe system for fiber preform fabrication” (grant #FA23861313019). In addition to related research to overcome the current limitation in power scaling of fiber lasers due to thermal mode instabilities, this new SEM has enabled us to fabricate a wide range of optical fibers including: antiresonant hollow core fibers, air-cladding fibers, multicore fibers to name a few.

With this DURIP support we have developed processes and protocols for the fabrication of microstructured optical fibers with precisely controlled structural parameters. In the order, to optimize our fiber fabrication process the fiber structure and chemical composition has to be characterized using the SEM/EDS microscope acquired with this grant. This has allowed us to establish an advance fiber characterization facility in CREOL.

The awarded budget and the final purchase details are summarized in Table 1. The ARO DURIP award was $124,200, whereas the final equipment cost was $124,200.

Table 1: Purchasing

Proposed Equipment	Vendor	Award	Final Costs
Phenom ProX SEM with EDS & Pro	**NanoScience Instruments Inc**	$124,200	$124,200

2. Description of Equipment: Phenom ProX SEM with EDS

The Phenom ProX SEM is the **world’s fastest and easiest to use SEM** (it takes less than 30 seconds from loading a sample to obtaining high quality a SEM image). It is unrivaled by other desktop SEMs in the image clarity and crispness. Frame-by-frame averaging and high pixel resolution (up to 2048x2048) provides striking images with little effort. The Phenom ProX SEM is the world’s **highest resolution desktop SEM.** It offers better than **14nm resolution and magnification up to 130,000times.**

All Phenom SEMs are designed to be vibration insensitive. All other SEMs on the market are sensitive to vibration and require a quiet, vibration-free environment. The Phenom is the only desktop SEM with an integrated navigation camera which makes navigation on a sample even at high magnification very easy. The navigation camera has the same point of view as the detector and is part of the never-lost navigation featue. The optical camera also allows sample evaluation before doing SEM. The Phenom SEM comes with a self-centering motorized x/y stage, which allows for easy navigation.

The vibration insensibility and small size of the Phenom ProX SEM allow us to install it and use it in CREOLs fiber fabrication facility and analyze samples in real time while they are fabricated. For application ***load to image time is critical*** – this SEM allows to obtain a SEM image in as little time as if we using a 100x optical microscope, allowing on-line optimization of our optical fiber fabrication process.

For our application, non-conductive silica fibers have to be analyzed in real time while they are fabricated. For this reason, the fibers cannot be coated in order to be analyzed. Therefore, a low vacuum SEM which reduces charging as the Phenom system is required.

The Phenom tabletop SEM/EDS system has allowed us to improve the yield of photonic crystal fiber (PCF) manufacturing process and develop novel active fibers with improved optical performance. The requested Phenom ProX is indispensable to assess and fine-tuning the drawing conditions in real time during fiber fabrication. Besides, we are also using this machine to optimize the active core material composition of ultra large mode area fibers for high power laser systems. This instrument adds to our current fiber processing, fabrication and characterization facilities acquired under several grants.

The Phenom tabletop SEM/EDS has been a fundamental equipment for the development of advanced fibers for high power applications and direct supports all our fiber fabrication projects. The full potential of large mode area PCF for high power fiber laser can only be achieved utilizing ***SEM and EDS to determine the precise structure and material composition of the fiber in real time during fiber draw.*** During drawing, diffusion and loss of some materials can occur; therefore, it is required to monitor the material composition of the preform at every stage prior to and during fiber drawing. The Phenom SEM/EDS provides immediate assessment of the fiber structure for fine-tuning of the drawing conditions in real time during fiber manufacturing. Besides, it allows us to optimize the active core material composition and improve the preform fabrication and fiber drawing conditions to achieve the required optical properties.

The fabrication of rare earth doped PCF for high power fiber lasers involves a complex interplay between material composition, structural properties and fiber drawing conditions. This has made it extremely challenging to determine the optical properties of the final fiber prior fiber drawing. The guidance properties of PCF can differ fundamentally by **nanometer size variations** in the micro or nano structured cross section and by compositional variations in the material forming the active core region. Therefore, in order to achieve a full control of the fiber fabrication process and optical properties, we rely on advanced structural and compositional analysis instruments to gain knowledge of the fibers precise geometry and material composition of the active core region. To illustrate the importance of state-of-the-art imaging and material composition analysis (SEM/EDS) instruments for the development of next-generation active PCFs, Fig 1. shows the design and pictures of the core material for active fibers being developed in my laboratory. The core of this fiber consists of ~500,000 of individual strands of glass – the nano structure cannot be observer from the optical microscope images. Each individual unit has a specific material composition. This ***nano-structured core*** is designed to control the refractive index and the active ion-dopant transvers profile of the optical fiber core. The gain core consists of 1000’s of nano-strands (about 20 nm in diameter) of Yb-ions co-doped glass in a fluorine and boron co-doped background. This fiber fabrication process opens the possibility of complex engineering of the fiber material composition down to the nanometer scale with ultrahigh precision.

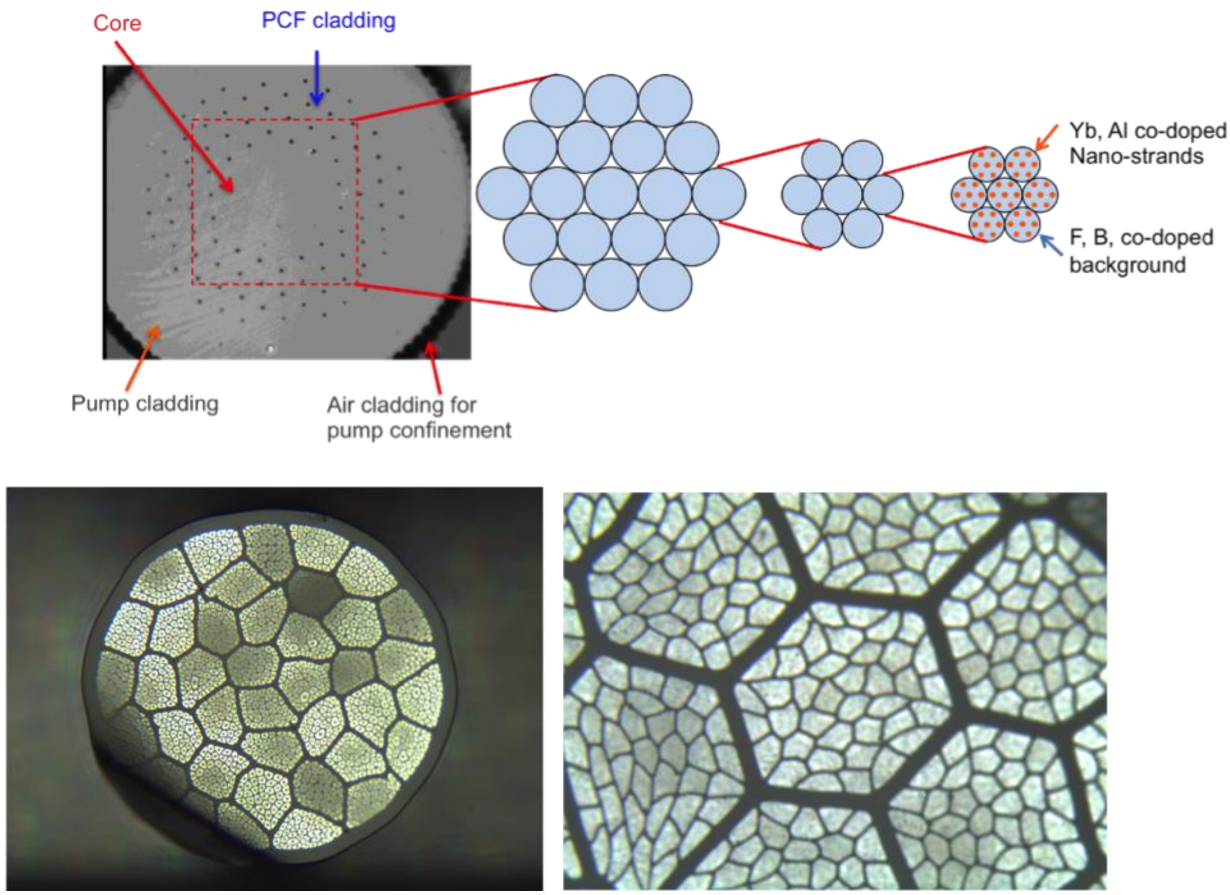

Figure 1. (top left) Microscope image of a rod-type PCF. The large fiber diameter (~2mm) protects the guided light from any bending- induced perturbation. (top right). Core design consisting of 1000's of individual doped filaments assembled to create a nano-structured active core. (bottom) Microscope images of the active core material fabrication.

The Phenom ProX Scanning Electron microscope offers features that are unique and make this product exceptionally suitable for our purposes. The Phenom SEM is the only SEM that is not sensitive to vibrations and therefore can be used in an environment where other SEMs cannot be used. No special lab requirements are required and therefore there is no additional cost involved in setting up the instrument or operating the instrument in our fiber fabrication facility. The Phenom can be run in noisy environments such as our fiber fabrication laboratory.

The Phenom SEM's sample load and unload times are by a factor of **5-10 times faster than any other SEM,** allowing for process optimization during fiber fabrication. The Phenom ProX SEM is the fastest and easiest to use SEM on the market (**it takes less than 30 seconds from loading a sample to getting a SEM image**) and provides best-in class image quality, clarity and crispness. Frame-by-frame averaging and high pixel resolution (up to 2048x2048) provides striking images with little effort. The Phenom ProX provides **better than 14nm resolution and magnification up to 130,000times**.

The Phenom is also the **only desktop SEM with an integrated optical microscope. Two microscopes, Scanning Electron microscope and optical microscope in one small package**. An integrated navigation camera and the self-centering motorized stage provide the unique never-lost navigation feature. **The optical microscope acts as navigation camera which** makes navigation on a sample even at high magnification very easy. The optical microscope provides color information at 20-135 times magnification allows us to evaluate our samples before using the SEM.

In Summary, the Phenom SEM is the only desktop SEM on the market that is suitable for our purposes and fiber fabrication environment because it offers these absolutely needed features:

- **Insensitivity to a noisy environment and building vibrations.**
- **Fast sample load and unload times in less than 30 seconds.**
- **Ease of Use & Never-Lost Navigation:** Interactive Relation Between Optical and Electron Image by taking advantage of the motorized x/y stage.
- **14 nm resolution and**
- **Electron Microscope: 80 - 130,000x magnification**
- **Brilliant images:** Due to the use of the high-brightness electron source, the Phenom SEM produces the best image quality. The Phenom offers the highest resolution, and 130,000times magnification and highest pixel density (2048x2048)
- **The Phenom SEM is the only desktop SEM that provides full EDS elemental analysis integration: one manufacturer, one software, one warranty.**
- **Image mapping (tiling) application software included.**
- **Smallest desktop SEM on the market, fits completely on a typical desktop**
- User maintenance-free operation. The use of the long-life and high brightness electron source allows the Phenom to be operated without any maintenance for 18 months before a preventative maintenance is due. The Phenom SEM is the only SEM that offers this feature.

Electron Source: The Phenom uses a high performance electron source CeB6 (Cerium Hexaboride): Benefits: Lifetime > 1 year, very stable beam, bright image, significantly reduced noise in SEM image. Small spot size. Standard acceleration voltages 5 kV, 10 kV and 15kV. Customizable accelerating voltage settings from 4.8kV to 15kV. Needs only 5keV to acquire magnifications up to 130,000x.

Other SEMs use a tungsten source (W), lifetime 60-100 hours, in low vac mode less than 50 hours. At lifetime end, the filament breaks like a light bulb without any indication. Source exchange has to be done by a trained user. Main disadvantage of Tungsten filaments are that the inner part of the microscope will contaminate faster.) To acquire high magnifications with a tungsten source, the acceleration voltage has to be increased. Disadvantages of higher voltages are: Charging effect makes sputtering of non- conductive samples (polymers, paper, rubber...) necessary.

For our application, non-conductive silica fibers have to be analyzed in real time while they are fabricated. For this reason, the fibers **cannot be coated**. Therefore, a low vacuum SEM which reduces charging as the Phenom system is required.

EDS: The Phenom uses a state of the art silicon drift detector. Combined with the high brightness source, the Phenom offers higher count rates than the competition, improving accuracy and allowing for faster EDS measurements. Sample does not need to be tilted to get good EDS data, which allows you to image and take EDS data without moving the sample. EDS Software and hardware are fully integrated, offering the same look and feel as the imaging user interface. One manufacturer, one warranty.

3. Advanced Optical Fiber Characterization

With this DURIP support we have established a dedicated facility to the characterization of specialty optical fibers. A picture of the Phenom SEM EDS in our laboratory is shown in Fig. 2.

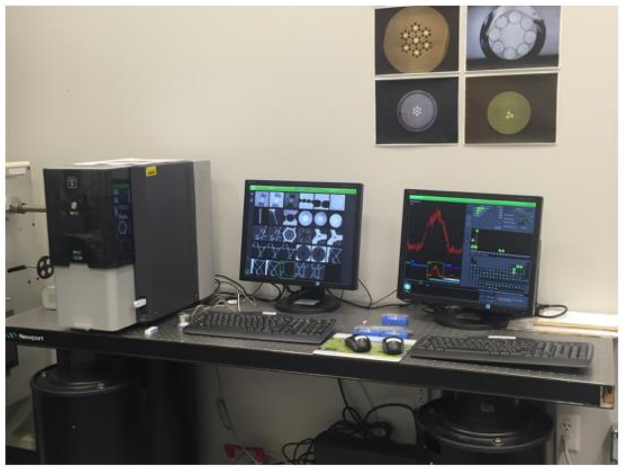

Figure 2. Phenom SEM & EDS system

UCF is one of the few academic institutions in the U.S. able to design and fabricate optical fibers of superior performance. With this DURIP support, we have developed processes and techniques for the fabrication of active and passive microstructured optical fibers. We have successfully demonstrated passive and active fibers structures including: ytterbium-doped fibers, thulium-doped fibers, erbium-doped fibers, antiresonant hollow core fibers, photonic bandgap fibers, photonic crystal fibers and multicore fibers to mention some. SEM images of some fibers fabricated at CREOL are shown in Figure. 2.

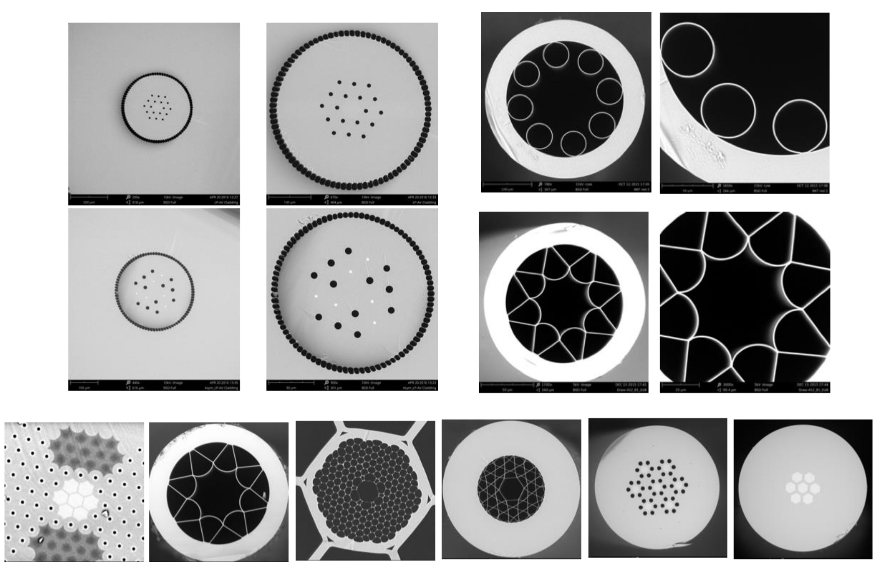

Figure 3. Scanning electron microscope (SEM) image of various microstructured optical fibers fabricated at CREOL. The glass features in these fibers range from ~100 nm to several micrometers.