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14. ABSTRACT
With the DURIP fund, we acquire a glass processing station to enhance our capabilities to achieve ultra-high performance photonic resonators and fiber structures with specially designed nano/micro features for building optical sensors with lower detection limits and detection resolution beyond what can be achieved in our group and other labs in the field. The equipment immensely contributes to our research project funded by PECASE (supported by DoD-ARO under the contract/grant number W911NF-12-1-0026) on high performance optical sensors. The multipurpose glass processing station also allows us to develop and fabricate novel photonic devices for the control

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Report Title

Final Report: A Versatile Glass Processor for High-Performance Photonic Platforms

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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

TOTAL:

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

TOTAL:

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

The PI was elected as a Fellow Member of The Optical Society (OSA).

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

Final Report

(Dates covered: August 1, 2014 to July 31, 2015)

Title: A Versatile Glass Processor for High-Performance Photonic Platforms

- **Program Manager:**

  Dr. William W. Clark (william.w.clark9.civ@mail.mil)

- **ARO Proposal Number: 65207ELRIP**

- **ARO Grant Number: W911NF1410419**

- **PI: Dr. Lan Yang, Edwin H. and Florence G. Skinner Professor, Electrical and Systems Engineering Department, Washington University, St. Louis, MO 63130**

1. Introduction

With the DURIP fund, we acquire a glass processing station to enhance our capabilities to achieve ultra-high performance photonic resonators and fiber structures with specially designed nano/micro features for building optical sensors with lower detection limits and detection resolution beyond what can be achieved in our group and other labs in the field. The equipment immensely contributes to our research project funded by PECASE (supported by DoD-ARO under the contract/grant number W911NF-12-1-0026) on high performance optical sensors. The multipurpose glass processing station also allows us to develop and fabricate novel photonic devices for the control of light and energy flow. Specifically, we fabricate a novel sound wave sensor that can respond to particular characteristic frequency depending on the specific sizes and shapes of a fiber taper optical waveguide. We also design and fabricate a bottle-shaped resonator from an optical fiber. Compared with WGM resonators in other shapes, such as microspheres and microtoroids, bottle resonators are more practical as a sensor for real-time monitoring of dynamic processes in micro/nanoscale in non-fluorescent solution, which might help provide information not available before using conventional measurement.

2. Summary of results

2.1 Fabrication of Bottle WGM microresonators for *in-situ* sensing of dynamic process in non-fluorescent solution

The Vytran automated glass processor allows us to fabricate bottle resonators with finely controlled shapes and sizes. In our experiments, we have used a bottle whispering-gallery-mode (WGM) resonator embedded in nonfluorescent polyacrylamide gel to characterize its dynamic gelation procedure. The bottle resonators are fabricated from standard silica optical fiber by tapering the fiber at two locations to create a bulge in the center. **Figure 1** shows a typical bottle resonator.

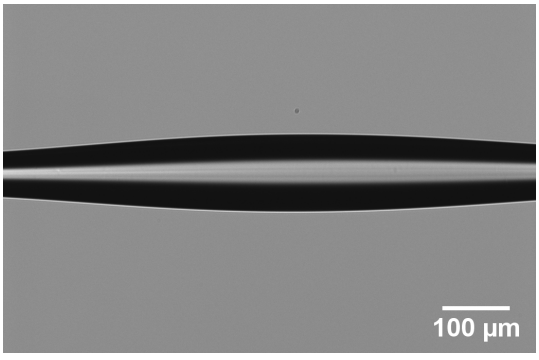

Figure 1.*Optical microscope image of a typical bottle WGM resonator. The optical mode is confined in the vicinity of the bulge in the center.*

Light is coupled into the resonator by a tapered fiber coupler. Evanescent coupling using tapered fiber coupling is extremely sensitive to the relative position of the resonator and the coupler. The use of bottle resonator, as opposed to the commonly used silica microsphere or microtoroid resonator, has an advantage in the ease of coupling.

Hydrogels tend to have significant expansion/contraction during gelation due to hydration/dehydration, and in the case of microsphere and microtoroid resonator, the coupling between the resonator and the tapered fiber coupler tends to be lost due to gel deformation. On the other hand, bottle resonators have a large radius of curvature in the axial direction and the tapered fiber coupler can be gently pressed against the resonator without slipping. This makes the coupling very stable during the dynamic gelation of hydrogels.

In order to measure the gelation of hydrogels using WGM resonance, we place the bottle WGM resonator between two pieces of glass slides, separated by a 1mm spacer. The tapered fiber coupler goes through the gap between the glass slides and is placed in contact with the bottle resonator. A fiber coupled tunable laser (765 nm-781 nm) is continuously scanned across a 0.1nm spectral range to monitor the optical modes within the bottle resonator. Polyacrylamide gels with different cross linker concentrations were prepared and injected into the gap between the glass slides using a pipette. The transmission spectrum through the resonator was to monitor the gelation of the polyacrylamide gel.

Due to the relatively large size of the bottle resonator, the optical modes densely populate the scanned spectral range, with many modes overlapping each other. Traditionally in order to monitor the shift in resonance wavelength a particular mode is chosen and its position is recorded using a peak finding algorithm. However in the case of a bottle resonator with densely populated modes shifting together, it can be difficult to track a particular mode with such algorithm. Instead, we have developed a cross correlation based algorithm, similar to those used for estimating time delay of acoustic signals, in order to obtain the wavelength shift. The wavelength shift computed this way essentially corresponds to the average wavelength shift of the modes within the scanning range

Figure 2 presents the observed wavelength shift of a bottle resonator during the gelation of polyacrylamide gel with two different cross linker concentrations. The curves are normalized and plotted together with normalized elastic modulus change obtained from rheology. Due to slight variation in the reactant concentration, the gelation curves have slight variation for both WGM resonance and rheology measurement. However, qualitatively the shapes of the curves are the same between WGM resonance and rheology for the 12% cross linker gel, with an initial lag phase followed by a fast gelation phase and eventual saturation. This demonstrates that WGM resonance is a feasible way to characterize the gelation of polyacrylamide gel. An interesting observation is that for 37% cross linker concentration, the WGM resonance curve shows an initial dip, whereas in the rheology curve no such dip is observed. We are still investigating the origin of this dip in the WGM resonance curve. Our current speculation is that this dip is related to the change in molecular polarizability due to free radical formation that precedes polymerization; formation of free radicals in the acrylamide monomers decreases their polarizability and leads to a blue-shift in the resonance wavelength, which appears as a dip in the WGM resonance gelation curve. If our speculation is correct, this suggests that WGM resonance allows us to observe additional

features in the gelation process not observable with rheology alone; this would greatly enhance our understanding in the gelation mechanism of different hydrogels.

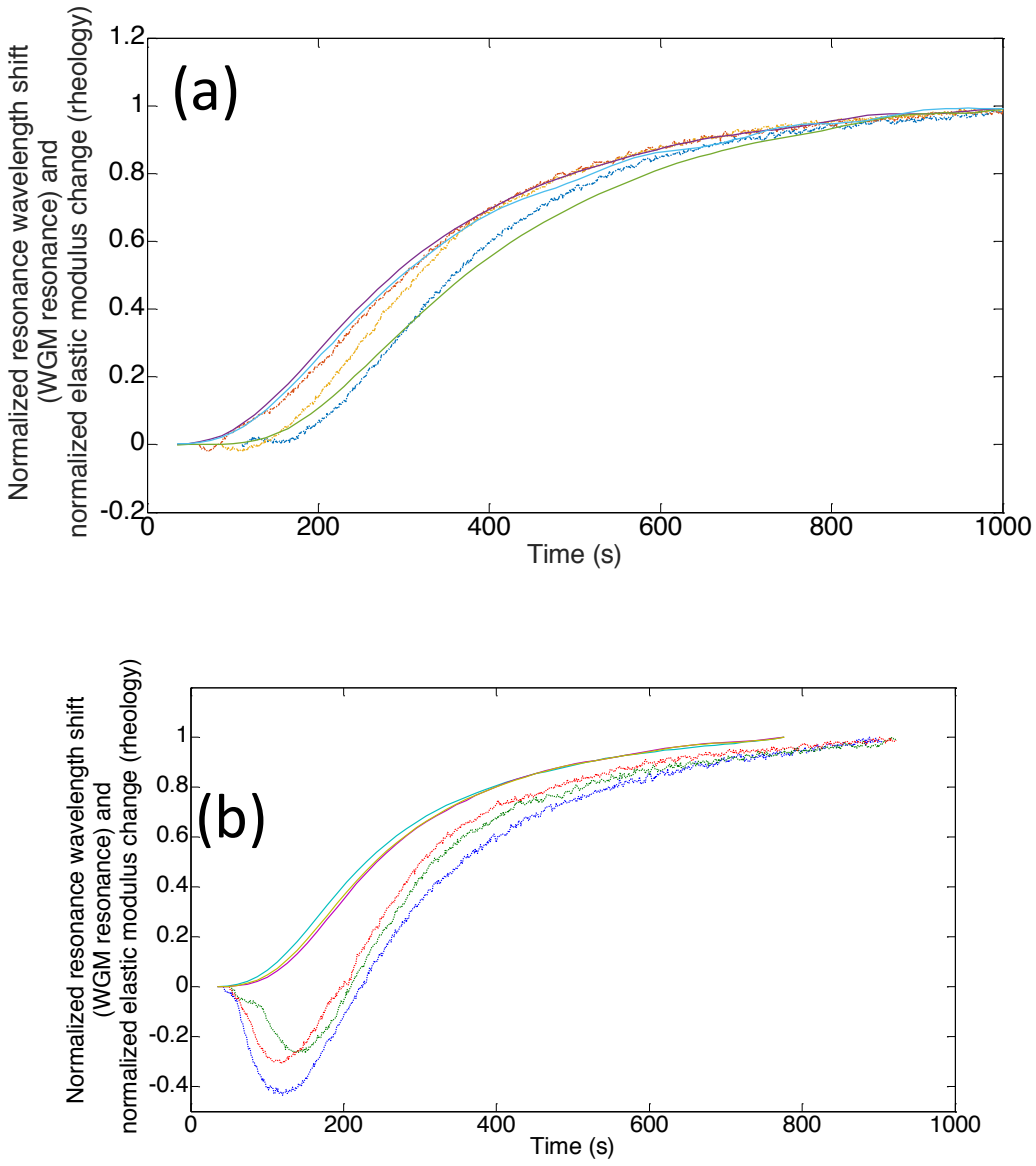

Figure 2. *Normalized resonance wavelength shift from WGM resonance measurement (dotted curves) and normalized elastic modulus change from rheology measurement (solid curves) for a) 12% cross linker and b) 37% cross linker. Cross linker concentration is relative to total monomer concentration. The figure shows the results from three repetitions for each gel. The gelation curve from WGM resonance and that from rheology are qualitatively in good agreement, except for the dip in the WGM resonance gelation curve observed at around 150s for 37% cross linker.*

To demonstrate the influence of cross linker concentration on the rate of gelation, we have measured the gelation time of 4 polyacrylamide gels with different cross linker concentration with both rheology and WGM resonance. The gelation time was defined as the time at which the normalized gelation curve reaches the half point between its

maximum and its minimum. The result is presented in **Figure 3**. Although there is a large variance in the gelation time, we see a clear trend; the gelation time decreases initially with cross linker concentration, but the decrease levels off at concentration around 25% - 37%.

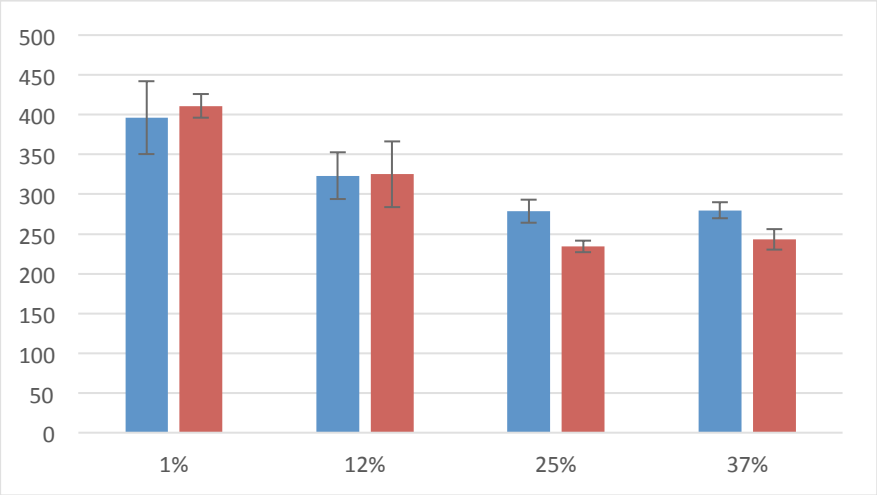

***Figure 3**. Gelation time of polyacrylamide gel measured by WGM resonance and rheology for different cross linker concentration.*

So far, we have collected some preliminary results showing the measurement of polyacrylamide gel with different cross linker concentration by using a WGM bottle resonator. It seems promising in providing a new technique and additional insights in studying the gelation of hydrogels. Our plan for the future is to study the influence of other parameters, such as total monomer concentration and initiator concentration, on the gelation kinetics of polyacrylamide. We also plan to study the gelation of other hydrogels with different gelation mechanisms. Finally it is interesting to study in further details the origin of resonance wavelength shift in WGM resonators during gelation and compare it with the gelation curve observed through rheology.

Currently, there are no techniques that allow high precision, robust, real-time, in situ investigation of the dynamic process of small non-fluorescent solutes (e.g. salts, drugs), such as diffusivity, in hydrogel and hydrogel-like environments. With the Vytran glass processing station, we can design and develop a series of bottle resonators with various geometries and sizes for systematic investigation of their sensing applications in nonfluorescent solution.

1. Acoustic Sensing and Filtering Using WGM Microresonators

WGM resonators are known to respond to mechanical pressures. Although sound wave is a pressure wave applied to media, there has been no quantitative characterization of direct optical response to sound for either optical fibers or WGM resonators. Because the eigen mechanical frequencies of a microresonator with size in the range of $10 - 100$ mm is on the order of megahertz, the resonator itself cannot respond to sound in the audible frequency range ($20 - 20{,}000$ Hz). For a resonator-taper-coupled system, the coupling condition, which affects the light transmission of the system, is highly sensitive

to the mechanical movement of the fiber taper triggered by the pressure wave, *i.e.*, the sound wave. Therefore, we can encode the sound to the fiber taper's mechanical movements, which can be characterized directly from the light passing through the system. The tapered fiber waveguide serves as a sound-wave sensor, while the WGM resonator acts as a filter.

The most important response characteristic is whether and how the resonator-taper-coupled system is tuned to pure tones, which will help us develop a new acoustic sensor using high-quality WGM resonators. **Figure 4A** shows preliminary data obtained from a fiber taper alone (about 12 mm in length and 1.3 μm in diameter) elicited by 20 tones with a frequency range of 80−12,000 Hz. The tone duration was 1 s with an inter-stimulus interval of 2 s. The sound intensity was 70 dB SPL. In the plot, the frame axis was arranged so that the tone frequency increased with the frame number. Here the frequency axis is the Fourier Transform of the light-intensity fluctuation.

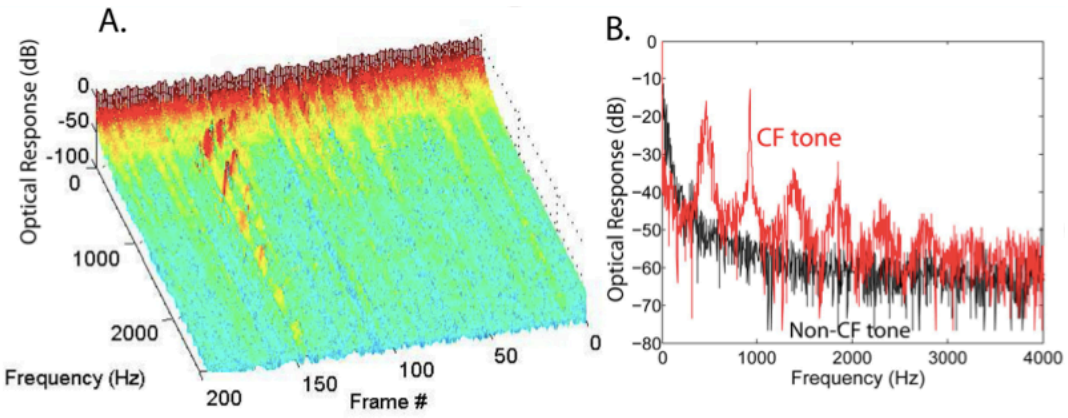

Figure 4. *Frequency responses to tones recorded with an optical fiber taper.*

The tone frequency that elicits the largest response is termed as the characteristic frequency (CF). First, there was always low-frequency components (near DC) whether a sound was presented or not, as indicated by the relatively uniform red color on the upper left side (**Fig. 4A**). This is because the laser input was constantly fed to the fiber taper. When a 9218-Hz tone was presented at Frame #150, the taper showed multiple peaks in the optical frequency spectrum, which was not observed for other tone frequencies barring a smaller response to the tone right below it (7081 Hz). Here, we define the CF of this taper as 9218 Hz. For a better comparison, we superimposed the optical spectral response to a non-CF tone (**Fig. 4B**, black) and the CF tone (red). The non-CF response had more energy close to DC, whereas the CF response had more energy at higher frequencies. It is worth noting that the oscillation "frequency" of the transmitted light in our system, presented as optical response in **Fig. 4**, is different from the frequencies of the tones that were used to elicit the fluctuation in the optical signals. For example, the peak frequencies in the taper's response to a 9218-Hz tone occurred at 467 Hz, 933 Hz, 1400 Hz, 1867 Hz, *etc*. Our preliminary results showed that that the peak frequencies in the optical response were determined by intrinsic mechanical-resonance frequency of the optical fiber taper. With different tones serving as the stimulus to the fiber taper, we could observe distinct optical response indicated by different frequency spectra. That

being said, each unique fiber taper is associated with a specific CF.

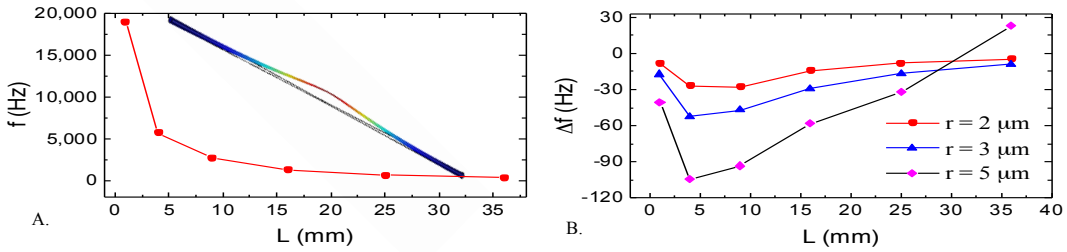

Figure 5. *A) Mechanical resonant frequency of the fundamental mode as a function of the length of a tapered fiber with a waist diameter of 1 μm. B) Frequency differences between tapers with different diameters (2 μm (red), 3 μm (blue) and 5 μm (black)) and a taper with a waist diameter of 1 μm (as shown in (A)) as a function of the taper length.*

Our numerical simulation results show that the intrinsic mechanical resonant frequency of a tapered fiber changes with the length and waist diameter (**Fig. 5**), which might consequently affect the CFs associated with a particular tapered fiber. If each resonator-coupled system only responds to specific CFs, a series of resonator-coupled systems with different sound tunings can act as both sound sensors (like a microphone) and frequency filters. In the future, we will explore the correlation between the characteristic frequencies of sound, to which the resonator respond, and the parameters of the device, such as geometries, materials, major (minor) diameters of the resonators as well as the parameters of the fiber taper waveguide. The follow-up study will help us design a high-resolution sensor with response to a particular sound frequency.