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Development of MEMS Photoacoustic Spectroscopy

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Abstract

After years in the field, many materials suffer degradation, off-gassing, and chemical changes causing build-up of measurable chemical atmospheres. Stand-alone embedded chemical sensors are typically limited in specificity, require electrical lines, and/or calibration drift makes data reliability questionable. Along with size, these "Achilles' heels" have prevented incorporation of gas sensing into sealed, hazardous locations which would highly benefit from in-situ analysis. We report on development of an all-optical, mid-IR, fiber-optic based MEMS Photoacoustic Spectroscopy solution to address these limitations. Concurrent modeling and computational simulation are used to guide hardware design and implementation.

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NOMENCLATURE

amb	ambient, written as subscript with (T)emperature or (P)ressure
AMPL	Advanced Manufacturing Process Laboratory
ATR	Attenuated Total Reflectance
BOX	buried oxide
CAD	Computer Aided Design
cm	centimeter
cm ⁻¹	wavenumber, or 1/wavelength in centimeters
CMP	chemical mechanical polish
dB	decibel
dBu	decibel with respect to RMS voltage
DOE	Design of Experiment
DUT	Device Under Test
FEM	Finite Element Modeling
FO	fiber optic
FFT	Fast Fourier Transform
FTIR	Fourier Transform Infrared
FWHM	Full Width at Half Maximum
GC	Gas Chromatography
Hz	Hertz, 1/sec
IR	infrared
kHz	kilo-Hertz
LFL	lower flammability limit
MeV	mega-electron volts
m	meter
ms	millisecond
MEMS	Micro-Electro-Mechanical Systems
MESA	Microsystems & Engineering Sciences Applications
N/A	not applicable
NIR	near infrared
P	pressure
Pa	Pascal
PAS	Photoacoustic Spectroscopy
PhC	Photonic Crystal
PIR	Polycrystalline Infrared
ppm	parts per million
pptr	parts per trillion
PWT	plane wave tube
Q	Quality factor
QCL	Quantum Cascade Laser
RMS	root mean square
ROC	Radius of Curvature
SEM	Scanning Electron Microscope
SMA	Sub Miniature, type “A”
SOI	Silicon on Insulator

T	temperature
μm; um	micron or micrometer

1. INTRODUCTION

1.1. Motivation

After years in the field, many materials suffer degradation, off-gassing, and chemical changes causing build-up of measurable chemical atmospheres. Chemicals can be corrosive to electronics and other materials, causing accelerated degradation and reduced time-to-failure. Even benign compounds may indicate known or unknown age-related issues that require action. Obtaining reliable chemical information from sealed environments is difficult at best. Stand-alone embedded chemical sensors are typically limited in specificity, require electrical lines, and calibration drift makes data reliability questionable. Along with size, these "Achilles' heels" have prevented incorporation of gas sensing into locations that could highly benefit from incorporation. Standard optical spectroscopy methods offer a partial solution by keeping most of the analytical hardware outside the system. This allows the equipment to be sufficiently complex and sophisticated to perform high quality measurements and analyses. However, standard hardware and techniques are often not directly adaptable to in-situ gas measurements, especially where complex mixtures of gases may be present. Significant barriers include optical access, sufficient interaction pathlengths, and transmission of useful wavelengths through fiber optics to the enclosed areas. This may include safety exclusion regions, where toxic or explosive compounds may be present. To enable such in-situ analysis, we have been developing MEMS-based fiber optic infrared Photoacoustic Spectroscopy (PAS) for safe and information-rich chemical monitoring suitable for sealed environments.

1.2. Approach

The technical issues described above can be overcome using all-optical mid-IR Photoacoustic Spectroscopy (PAS) enabled by Micro-Electro-Mechanical Systems (MEMS). Developing all-optical MEMS PAS presents many challenges and risks. The technological elements required for this work had not previously been combined into a single assembly, as few applications have a need for both rigorous safety requirements and extremely limited access. To address the challenges we combined a cross-disciplinary team. The project work and objectives included modeling and design of microfabricated PAS cells with small sensing volumes, developing two advanced optically-transduced microphones, delivering broadband excitation light over highly attenuating chalcogenide fibers in the near- and mid-IR ($1000\text{-}5000\text{ cm}^{-1}$), deconvolving PAS spectra from complex analyte mixtures, and forming correlations between PAS signatures and gaseous aging by-products. The project leveraged Sandia's MESA facilities (Center 1700), Computation and Simulation (Center 1500), and expertise in stockpile surveillance and chemical analysis (Centers 1700 and 1800).

1.3. Application Areas

Applications that can benefit from fully matured non-invasive monitoring range in complexity. At the smallest scale, micro-volume gas samples can be characterized

without withdrawing a sample. The evolution of gases can be traced during accelerated aging of samples, components, and sub-systems. Over long periods shelf-life units can be monitored. Changes to surveillance units can be observed while being subjected to simulated environments. The effects of assembly steps can be monitored in real-time. The loftiest goal is to enhance reliability of the nuclear weapons stockpile through 100% gas surveillance in the field. Technical and bureaucratic barriers aside, a better understanding of normal gas evolution in the stockpile would enable detection of individual and systemic changes without the need for field returns. Such data would improve quantification of margins and uncertainties and improve logistics when field returns were otherwise necessary. Other applications include intrusion detection, quantification of gases in explosive environments (e.g. methane in a coal mine), and in-situ characterization of solids and liquids.

PAS cells can be fabricated in MEMS with an integrated optical microphone for high sensitivity and optical isolation. Direct integration of specialized IR transmitting ($1000 - 6700 \text{ cm}^{-1}$) fiber optics and low-loss light guiding, enable support equipment (strong light sources, data acquisition equipment, etc.) to be separated by hundreds of meters or more, which is essential when safety considerations are extreme.

Success in this project includes a significant advance toward a small yet robust, all-optical method and hardware enabling in-situ characterization of gas composition in systems important to the National Security Enterprise. Systems include components, sub-systems, and accelerated aging test beds whereby many materials may be heated together for combined effects testing.

1.4. The Photoacoustic Effect/Prior Work

The photoacoustic effect in all phases has been well documented in the literature [1-5]. It was reported as early as 1861 as the “photothermal effect” by Alexander Graham Bell [5]. It occurs when molecules are suddenly illuminated by electromagnetic energy from a light source with energy matching its absorption bands. Vibration-rotation excitation occurs, which is rapidly converted to heat and pressure through collisional relaxation processes, and thereby an acoustic pulse is created. When the source is modulated (e.g. with optical chopper wheel or a pulsed diode), acoustic pulses are synchronized with the modulation frequency. The effect occurs in solid, liquid, and gas phases, in direct proportion to the concentration of the absorbing molecule and the light intensity. Because typical absorption cross-sections are so small, a small fraction of the exposed molecules absorb light. The fraction increases linearly with light intensity over many orders of magnitude, even when powerful lasers are used. When a broadband source is scanned, the resulting spectra are very similar to the analytically powerful technique of IR Spectroscopy for determining composition and concentration. Spectra from both methods result from the molecular absorption of electromagnetic radiation, and contain a great deal of information for identifying and quantifying chemicals, even in complex mixtures. PAS can be used when IR Spectroscopy is not suitable, such as when the optical pathlength is constrained, or when very limited volumes of gas are available.

Figure 1 shows an exemplary method for generating photoacoustic spectra. Light from the source is collimated, wavelength selected, and then re-focused into an IR fiber optic. The light source is modulated by the chopper wheel. A second fiber optic carries information on the mechanical deflection of a membrane microphone that is an element of the PAS cell.

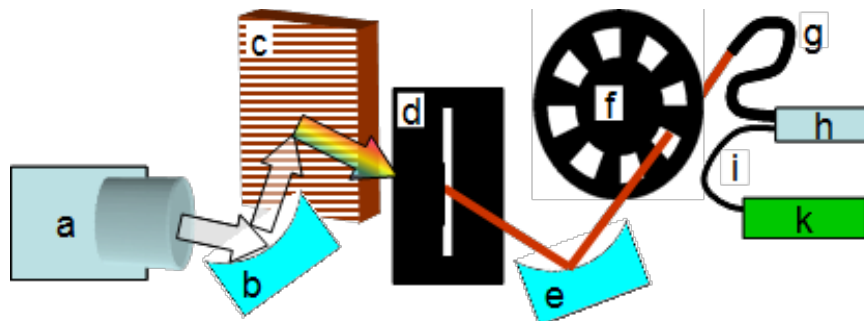


Figure 1. Schematic of exemplary PAS setup. a) IR light source, b) collimating mirror, c) diffraction grating, d) slit, e) focusing mirror, f) chopper wheel, g) IR fiber optic, h) MEMS PAS cell in isolated environment, i) optical microphone fiber, k) optical microphone source and electronics.

In practice, the photoacoustic effect may be obtained in many different ways. The central optics may be eliminated if a pulsed scanning laser is used. Modulation may also be time modulated, as with a Michelson Interferometer. With this setup, each wavelength of light has a beat frequency that is deconvolved using Fourier Transform mathematics. A photoacoustic beat pattern results. Once deconvolved, the result is a spectrum of wavelength vs absorption intensity.

IR techniques are widely used due to the rich information contained in the spectra. PAS has a niche use within the field. Unlike IR Spectroscopy, the PAS signal scales inversely with cross-sectional area, but not with pathlength. This makes microfabrication of PAS cells a logical direction. Despite this advantage, most microfabrication efforts have focused on development of ever more sensitive piezoelectric microphones. With some exceptions [6-7] PAS cells are often fabricated by traditional machining methods for use in the lab with emphasis on improving detection. While the optical excitation is typically through free-space paths, use of fiber optics has been reported [8], but only in the near-IR region ($3000\text{--}12,500\text{ cm}^{-1}$), well outside the highly valuable “fingerprint” region ($600\text{--}1200\text{ cm}^{-1}$). The fingerprint region is important for exact chemical identification for this is where small differences in molecular composition and structure result in significant spectral differences. Yet this region is inaccessible to common fiber optics. In an unrelated LDRD, a custom Attenuated Total Reflectance (ATR) IR Spectroscopy system for the NIR region of $1800\text{--}4000\text{ cm}^{-1}$ was built towards miniature, in-situ analysis of motor oil [9]. A blackbody radiation light source and a pyroelectric detector were insufficient for obtaining useful data in the fingerprint region (but worked well in the near-IR). Block Engineering, LLC has created a pen-sized multi-pass IR MEMS absorption spectrometer, but it requires electrical power within the unit [10]. These efforts are unsuitable in many ways for incorporation within a hazardous or restricted access environment. Fiber optics have also been used for transducing the photoacoustic signal [11-12], however, this was done by wrapping the fiber around a full-size PAS cell. Other

microfabrication efforts involve just the microphone sensor, such as a piezoelectric quartz MEMS tuning fork [13].

1.5. Mid-IR Fiber Optics

Mid-IR transmitting fiber optics are an essential hardware component to the conceptual design. Over recent years, these fibers have improved significantly in transparency and ruggedness, though they fall far short on both counts compared to telecommunications fibers [14]. Each type of IR fiber has advantages and disadvantages. Various options include chalcogenide, polycrystalline, hollow core waveguides, and more exotic photonic lattices and supercontinuum fibers [15-16].

Chalcogenide fibers, which include sulfur, selenium, and/or tellurium, have good short distance transparency [17]* in the near- and mid-IR ($1000\text{-}6700\text{ cm}^{-1}$). The high cost (recently \$1400/meter for 135 micron core) and transmission losses of these fibers has prevented most research efforts from employing them when free-space optical paths suffice. CorActive High-Tech, Inc. (Quebec, Canada) fabricates these fibers under an exclusive license from the Navy Research Laboratory.

High Tech Photonics, Inc. (Delray Beach, FL) offers polycrystalline IR (PIR) fibers made of pure AgCl:AgBr. With somewhat higher attenuation [18]*, these fibers have the advantage of a much broader transparency window ($550\text{--}2500\text{ cm}^{-1}$) and no absorption bands, compared to chalcogenide fibers. This covers the fingerprint region, but C-H and -OH fundamental absorption bands ($2900\text{--}3500\text{ cm}^{-1}$) are largely attenuated (but still present due to their strength). Recently, the cost was \$650/meter, including titanium SMA connectors. These fibers have other common attributes. Both are extremely radiation tolerant, operate up to 200°C , and can withstand high optical power densities ($\sim 10\text{ kW/cm}^2$). They are also sturdy and moderately flexible. Applications include carrying powerful CO_2 laser pulses.

For this work, PIR fibers from High Tech Photonics were used. The core and cladding dimensions were 900 and 1000 microns, respectively. Two sets of fibers were obtained. The first had SMA 905 connections at both ends. The second set had an SMA 905 connector at one end and bare fiber at the other.

1.6. Comparison of Competitive Techniques

Table 1 compares mid-IR fiber optic (FO) MEMS Photoacoustic Spectroscopy as researched here alongside other relevant analytical techniques that the scientific community could use in less restrictive situations. MEMS PAS does not provide leading edge trace-level sensitivity or simplicity. It does, however, provide a niche where a reasonable combination of sensitivity and specificity combine with a unique stand-off capability.

* Due to copyright protection, fiber transmission plots are not shown here. Refer to the product brochures.

Table 1. The subject all-optical, FO MEMS PAS compared to similar techniques and high-end standard technologies. Entries are typical and systems can vary greatly depending on actual setup.

Quality	F.O. MEMS PAS	Standard PAS	IR Spectroscopy	ATR/IR Spectroscopy	Stand-alone sensors	Gas Chromatography	Mass Spectroscopy
Size/Weight	very small, with larger benchtop equipment	medium, with larger benchtop equipment	large benchtop equipment	medium, with larger benchtop equipment	very small	large benchtop equipment	large benchtop equipment
Complexity	moderate (custom)	moderate	moderate	moderate	low	moderate-high	moderate-high
Optical Pathlength	short (< 1 cm)	~15 cm	1 meter	~40 microns in liquid	N/A	N/A. Requires gas sample	N/A. Requires gas sample
Open Air Region	N/A	600 – 12,500 cm^{-1}	600 – 12,500 cm^{-1}	N/A	N/A	N/A	N/A
Waveguide Region	550 – 2500 cm^{-1}	3000 – 12,500 cm^{-1}	N/A	1000 – 12,500 cm^{-1}	N/A	N/A	N/A
Limits of Detection in gas phase	ppm	ppb with lasers	sub-ppm	poor gas phase sensitivity	low percent	ppb – ppt	sub-ppt
Specificity	moderate	moderate	moderate	moderate	low	very high	very high - the gold standard when combined with GC
Electrical paths	no	yes	no	no	no	no	no
Extreme environment insertion	yes	no	no	no	no	no	no

2. THE MEMS PHOTOACOUSTIC CELL

2.1. Theory

A survey of the literature revealed few microfabricated photoacoustic cell designs. They tended to mimic miniaturized traditional cells – e.g. 1:20 scale footprint with a flat instead of round cross-section [7]. This design concept can be further miniaturized by considering the theoretical equation [3] for generating the photoacoustic signal S (Equation 1).

$$SQ \propto \frac{I_0(\lambda)G(\gamma-1)L}{2\pi\nu V}; \text{ for cylindrical cells} \quad (\text{Equation 1})$$

where Q and G are the quality factor and geometrical factor of the cell, I_0 is light intensity at an absorbed wavelength λ , γ is heat capacity, ν is the modulation frequency of the light source, L is pathlength, and V is cell volume ($\pi r^2 L$). The equation is commonly expressed with the cell's cross-sectional area when the pathlength is canceled in the numerator and denominator. Unlike traditional absorption-based IR Spectroscopy, a longer photoacoustic pathlength does not help. While a longer pathlength excites more molecules, the resulting pressure pulse is also spread in direct proportion to the pathlength, resulting in unity gain (no gain). This only causes the gas sample to be larger – equal to the cell volume. Taken to its extreme, no pathlength is required. In fact, PAS can be quite successfully applied to analyzing thin films, opaque solids, and liquids. The “pathlength” is not meaningful when considering the absorption of a photon by a molecule. However, the absorbing molecule is part of its surrounding environment, which inelastically transduces the electro-magnetic radiation into thermal energy, resulting in a mechanical acoustic wave subject to the properties of the surrounding media (respective speeds of sound, reflections, attenuation, re-thermalization, etc.). Photoacoustic excitation can be used as a microscopy technique, including in tissues, a technique that is currently undergoing much research in the bio and medical communities [19-22]. As gases are by nature less dense than solids, a non-zero pathlength is required to contain the vapor phase molecules.

Microfabrication appears to be ideally suited for increasing the photoacoustic signal with no loss due to length. Highly defined and reproducible shapes can be achieved to increase the resonant quality factor and thus signal. Certain MEMS substrates (silicon, germanium) enable other unique advantages, such as IR illumination directly through the transparent bandgap of the cell's material. A gold coating can alternately be used to multiply the probability of a photon's absorption without increasing cell size. Access ports can be micron-sized. Another advantage is that an optical microphone can be co-fabrication directly on an acoustically compliant membrane comprising the PAS cell. In the end, the microphone dominates the size constraints of the conceptual integrated designs.

In using micro-PAS cells one potential disadvantage arises. Macroscopic PAS cells are designed to resonate. This can enhance the signal up to 100-fold as small, repeated photoacoustic forcing of the cell walls amplifies their displacement and thus the microphone's signal. A requirement for resonant operation is that the excited molecules must have time to thermalize into a pressure pulse, which in turn must dissipate before the next

excitation pulse. Depending upon the excited molecules and surrounding environment, repetition rates above $\sim 10,000$ Hz may cause saturation of excited states that no longer cause an acoustic pulse. Thus, macroscopic PAS cells are typically designed for 1-5000 Hz with an active resonant length ~ 4 inches. Micro-PAS cells have much higher resonances and thus can't take advantage of resonance operation. The other advantages must overcome this deficit to obtain equal sensitivity.

Non-resonant operation also has advantages. First, it is no longer necessary to tune the repetition rate to cell's resonance. The cell's resonance is susceptible to drift by multiple factors, including temperature and the changing gas composition. A second advantage is that it enables a richer study into the nuances of molecular relaxation processes. Rather than being limited to highly filtered resonant frequency of the PAS cell, the microphone's response to individual pulses can be traced. The rise and fall of a single pressure pulse can thus be observed, with thousands of repetitions signal-averaged to reduce noise. This enhances the ability to study energy transfer and relaxation kinetics. These processes start with a photoacoustically excited molecule's first intermolecular collision. The molecules may scatter elastically, with conservation of existing momentum; the excited molecule may elastically transfer its excited state to an identical, unexcited molecule; or the energy may be converted into translational energy partitioned between the molecules, thus increasing the system's kinetic energy, temperature, and pressure. Such effects have been studied previously with resonant cells [23]. The work reported here uses the broadband FTIR method, in which cell resonances are irrelevant.

2.2. Cell Resonance Modeling

During the PAS cell design process, modeling and dynamic computational simulations were used to understand and optimize cell design, its resonances, fluid-solid coupling, fluid flow, and viscous and thermal losses. COMSOL Multiphysics[®] software was used.

Figure 2 shows modeling of the acoustic response for the PAS cell. The cavity domain is defined by two short cylinders of different diameters, one atop the other. The larger (top) cylinder of the assembly has a diameter of 4 mm and height equal to 1.1 mm. The lower chamber is a smaller cylinder with a diameter of 2 mm and a height of 0.5 mm; they are positioned such that their axes are co-linear. The location of the microphone is at the bottom center of the lower chamber. The upper chamber has the triangular intrusion of length 0.75 mm. There two gas inlet/outlet ports to the upper chamber have little effect on the acoustic response of the cavity.

Isosurfaces of acoustic pressure, $\text{abs}(p)$, corresponding to the first five resonant frequencies are shown in the figure. Red contours correspond to the maximum pressure and blue contours represent zero pressure levels. Each row in the figure layout displays two views, top and bottom, of the pressure field for the corresponding frequency.

The eigenmodes (waveforms), and their corresponding eigenvalues (frequencies) are directly related to the cavity dimensions. For example, the first eigenpair correspond to the largest dimension of the micro-cavity, *i.e.* the diameter of the upper chamber. Here, one observes a single half-wave spanning the upper chamber with maximum pressures on opposing side

walls and a fundamental frequency of 49,577 Hz. The next higher eigenvalue is 55,282 Hz. This 2nd natural frequency corresponds to an eigenmode that is transverse to the 1st eigenmode. This slightly higher frequency is due to the presence of the triangular intrusion, which effectively reduces the cavity dimension in this direction. Similar observations can be made to explain the remaining eigenpairs.

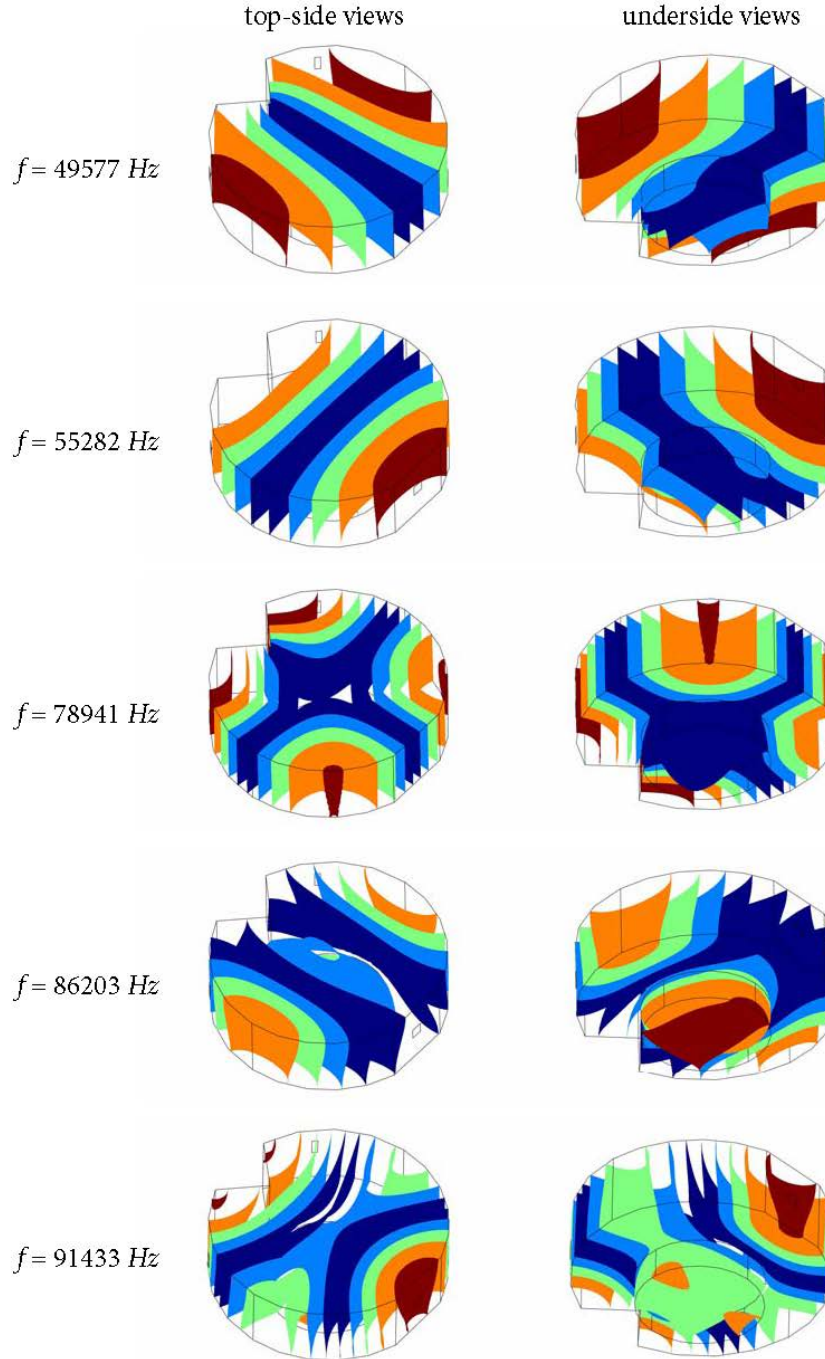


Figure 2. Resonant frequencies and modes for the PAS cell design.

A final observation is noted regarding device design. Maximum sensitivity of the microphone will be realized if the microphone is positioned at locations of peak pressures. Sometimes this is not possible. If the position of the microphone is determined by competing design criteria, then the cavity should be excited/driven at those frequencies where the pressure at the microphone is maximum. For a microphone positioned bottom center of the lower chamber it would appear that excitation frequencies near the 4th eigenvalue would be optimum. Despite the above analysis, it is known that the photoacoustic excitation frequency must be lower than the cell's resonant frequencies, as described in the Introduction. In addition, the microphones were designed to have a cut-off frequency of 20-30,000 Hz. Regardless, the rise time of a photoacoustic impulse is quite fast, containing a broad range of frequency content. It is this pressure forcing that drives the microphone membrane in the broad, flat broadband response region.

Dynamic simulation was also performed to understand the dynamic response of the hardware to the photoacoustic pulse. As the cavity was not yet fully designed the geometries are not the same. At sub-resonance, the general principles apply to both designs. As a starting point, a model system of system of 1 ppm carbon dioxide in nitrogen was assumed, illuminated by 0.1 mW pulses at the 2350 cm⁻¹ absorption band.

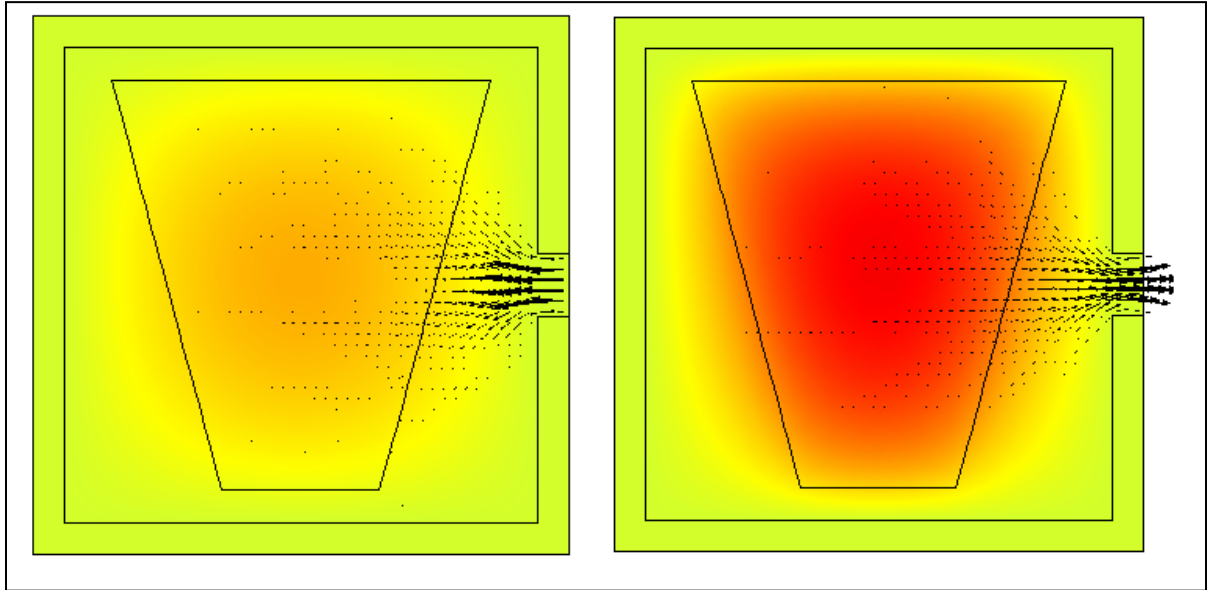


Figure 3. Two screen shots of a dynamic pressure pulse simulating a photoacoustic pulse in a confined cavity.

Figure 3 illustrates a 2D domain for evaluation of a simple, pulse-heated, breathing micro-cavity. The cavity is square with a side length of 6 mm and is surrounded by a rigid wall of thickness 0.4 mm. Air within the cavity is heated (within the trapezoidal outline) with a sequence of rectangular thermal pulses, assumed to come light absorption during photoacoustic excitation. Pulse separation is 110 ms with a dwell time of 90 ms (this is much slower than a typical PAS repetition rate). Peak heating during the pulse is 0.1 mW/m². The consequence of this intermittent heating is to temporarily raise the pressure of the internal air thereby causing an outflow through the side vent (gas port) to ambient conditions, *i.e.*

$T_{\text{amb}}=20^{\circ}\text{C}$ and $P_{\text{amb}}=1$ atmosphere. The vent port is 0.8 mm wide with a length of 0.4 mm (this latter dimension is the thickness of the wall surrounding the cavity). Hence, in this case there is little flow resistance through the outflow ‘ducting’. The model includes conjugate heat transfer from the heated gas to the cavity walls and convective heat loss from the walls to the surrounding environment. Thus, there is some cooling of the air within the cavity via heat lost to the surroundings.

As air is expelled from the cavity during heating the remaining air in the cavity becomes less dense (from loss of mass) resulting in a lower pressure inside the cavity. At this time air flow through the vent reverses as external air re-enters the cavity to equalize the pressure differential. This behavior is concurrent with the non-heating portion of the pulse sequence. Overall this simple model does a credible job of predicting the pressure response on the walls of the cavity. It follows that if one of the cavity walls were replaced with a flexible membrane that could function as a microphone the acoustic response of the cavity to rapid heating could be predicted. Amplitude of the pressure on the cavity walls can be increased if the flow impedance in the exhausting ductwork is increased. In the final cavity design, the ducts were filled with capillary tubing with 200 micron IDs and length upwards of 0.5 meter each. Subsequent modeling showed much higher flow impedance during a pressure pulse, such as to make their contribution negligible compared to membrane deflection.

2.3. IR Reflectivity Modeling

Optical thin-film modeling studies were undertaken to understand the impact of gold coating the MEMS-PAS cavity. Based on optical scattering matrix theory, calculations were performed for reflectivity of a system where light originating in free-space is incident upon a surface coated with Au and Ti on an Al_2O_3 substrate [24]. Complex indices of refraction as a function of wavelength from 2 to 20 μm (wavenumbers of 500 to 5000 cm^{-1}) were acquired from the literature for all of the materials. A MathCAD script was written to calculate the parallel and perpendicular reflectivity components as a function of wavelength (Figure 4), incident angle (Figure 5), and film thickness [25]. This data was then used to compute the quality (Q) factor for the cavity for the single-reflection case. All calculations were performed assuming a specular reflection case (optically smooth surface).

The scattering matrix (S) is assembled from the geometry of Figure 6 where the subscripts correspond to the layer numbers, assuming materials of air or vacuum on the far left (n_1), Au (n_2), Ti (n_3), and Al_2O_3 (n_4) respectively.

$$S = H_{12}L_2H_{23}L_3H_{34} = \begin{bmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{bmatrix} \quad \text{Eq. 1}$$

From Equation 2, the layer transition matrices (L_i) and boundary transition matrices (H_i) were constructed for each layer and then cascaded as shown in Equation 1.

$$L_m = \begin{bmatrix} e^{-i\beta_m} & 0 \\ 0 & e^{i\beta_m} \end{bmatrix}$$

$$H_{jk} = \frac{1}{\tau_{jk}} \begin{bmatrix} 1 & \rho_{jk} \\ \rho_{jk} & 1 \end{bmatrix}$$
Eq. 2

In Equation 2, the phase shift is calculated for each transition matrix as follows:

$$\beta_m = \frac{2\pi}{\lambda} n_m d_m \cos \theta_m$$
Eq. 3

where the values for amplitude transmission coefficient (τ) and amplitude reflection coefficient (ρ) are calculated from the well-known Fresnel theory [24, 26-27]. It is noted that the index of refraction for all the materials is a function of wavelength and thus, the scattering matrix becomes a function of wavelength. The measured index of refraction values as a function of wavelength for Au, Ti, and Al₂O₃ were found from the literature [28-29]. Thus, the value of the reflectivity R of the layered film as a function of wavelength and input angle is as follows:

$$R = \left(\frac{S_{12}}{S_{22}} \right)^2$$
Eq. 4

and is a function of the material optical properties, various thicknesses and the incident angle of the light onto the surface.

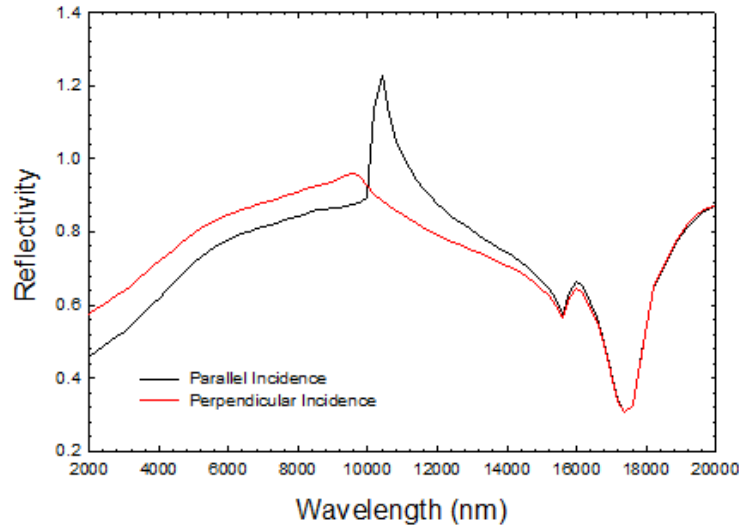


Figure 4. Simulated reflectivity for both parallel and perpendicular polarized incident light. The peak with greater than unity reflectivity is due to a constructive resonance in the material stack-up.

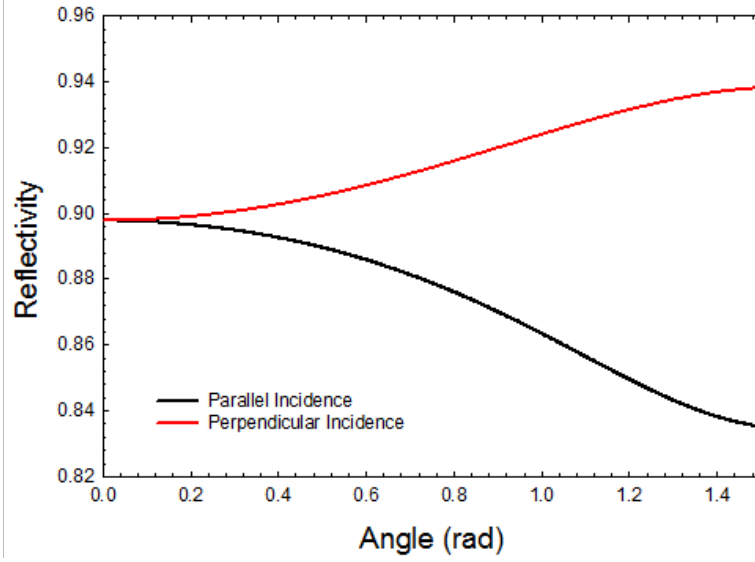


Figure 5. The effect of incident angle versus reflectivity for wavenumber equal to 1000 cm^{-1} (10 micron wavelength).

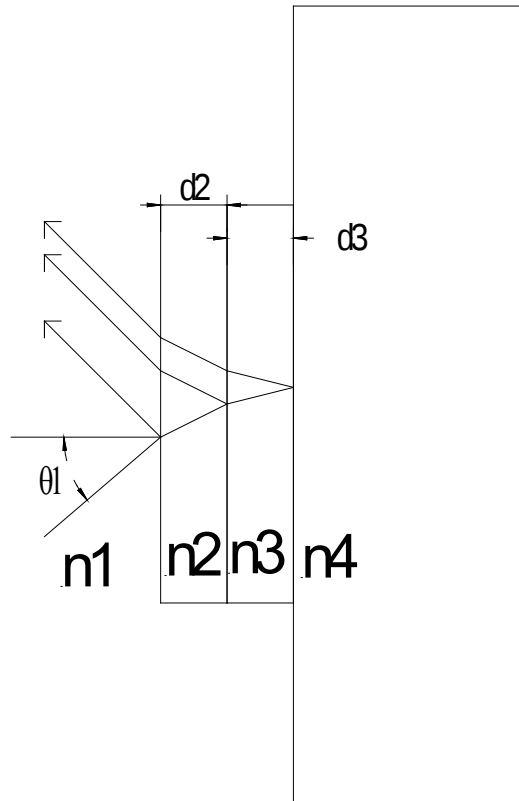


Figure 6. Diagram of the optical geometry used to construct the scattering matrix. The indices are for free space (n_1), Au (n_2), Ti (n_3), and Al_2O_3 (n_4) respectively. Light is incident from the left as shown and the film thicknesses corresponds to the layer numbers as shown.

2.4. Design

Modeling and computational simulation led to a conceptual PAS cell design concurrent with designs for optically transduced microphones (next section). This ensured mutual compatibility within the physical constraints of combining these two elements. While the ultimate goal was co-fabrication with the microphone, it was more practical and lower risk to start with a reusable PAS cell into which many iterations of the microfabricated microphones could be individually fitting for testing. The CAD design is shown in Figure 7. The PAS cavity has an internal diameter of 4 mm and a height of 1.5 mm. The microphone, lid, and IR fiber ferrule are not shown. All parts, except the fiber optic, are coated with 1000 Angstroms of gold on a titanium adhesion layer. A triangular feature (a) opposite the IR fiber port (b) reflects incoming light, homogenizing illumination of the cavity to ensure maximum photoacoustic effect. The microphones fit into the bottom cavity (c). Two gas ports (d) at adjacent corners fit GC capillary tubing for test gas sample introduction and venting. Also not shown are 4 electrical traces. These are for fine tuning various iterations of the optical microphones during the development phase, and are not intended to be part of the final design.

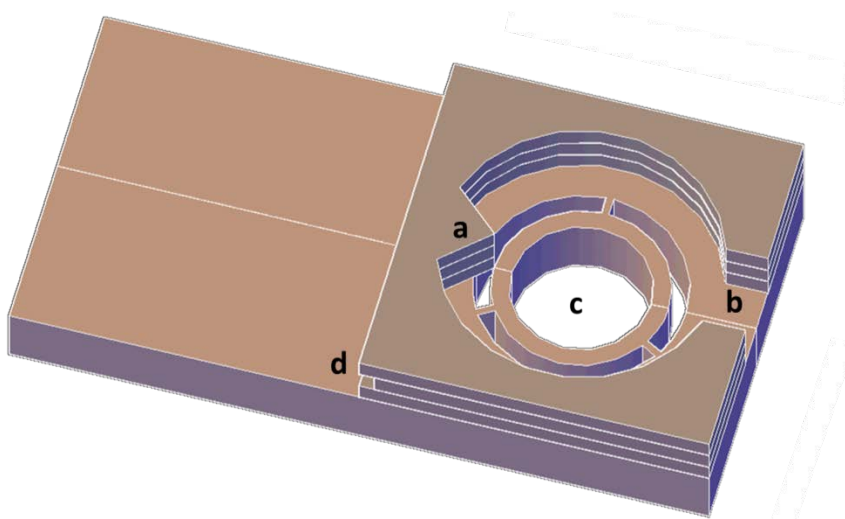


Figure 7. Main body of photoacoustic cell. IR fiber illumination is through port at right. The microphone seats into the bottom feature, and a lid covers and seals the assembly.

2.5. Fabrication (Construction)

A set of photoacoustic cells was constructed in the AMPL from the pieces shown in Figure 8. The cell was built up from layers of AD-96 amorphous silica from CoorsTek, Inc. (Golden, CO). Each piece was cut from wafers ranging from 10 to 20 mils thick using a CO₂ laser following a CAD design. Pieces were aligned using alumina rods and bound together with HF-74 epoxy from Epoxy Technology, Inc. (Billerica, MA). The parts were then coated with 1000 Angstroms gold over a 50 Angstrom titanium adhesion layer. Connections between the optional electrical pads and microphones were made with electrically conductive, silver filled epoxy. The fiber optic was not potted, but press-fit into the ferrule, which was help in place with Krazy Glue for solvent-based removal. Figure 9 shows a picture of a nearly assembled cell.

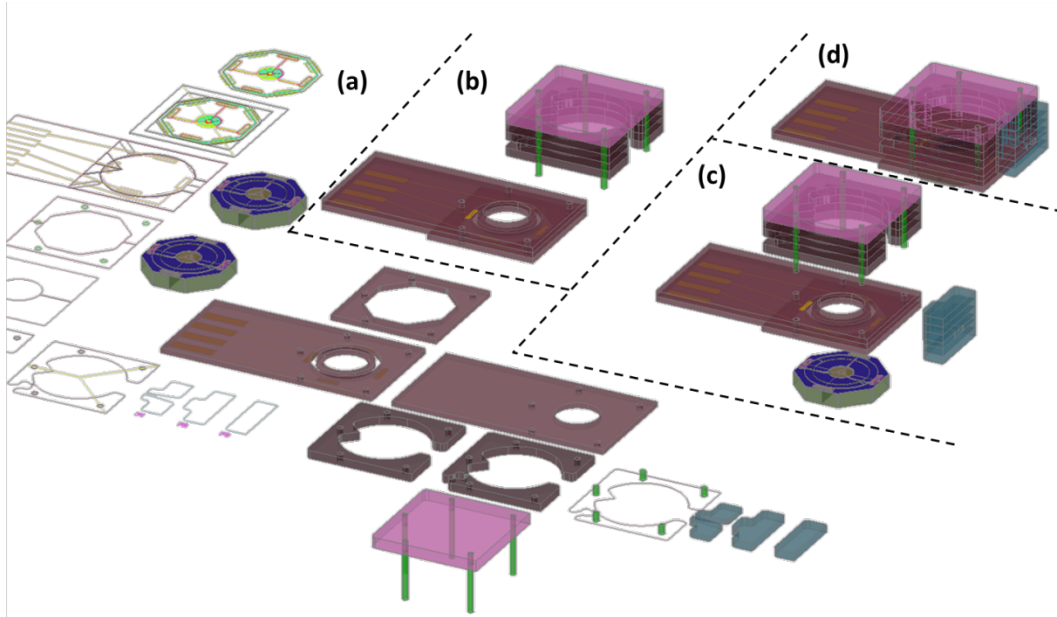


Figure 8. Photoacoustic cell assembly in various stages. (a) Piece parts, including the MEMS microphone (blue octagons); (b and c) a cell partially assembled; (d) fully assembled PAS cell.

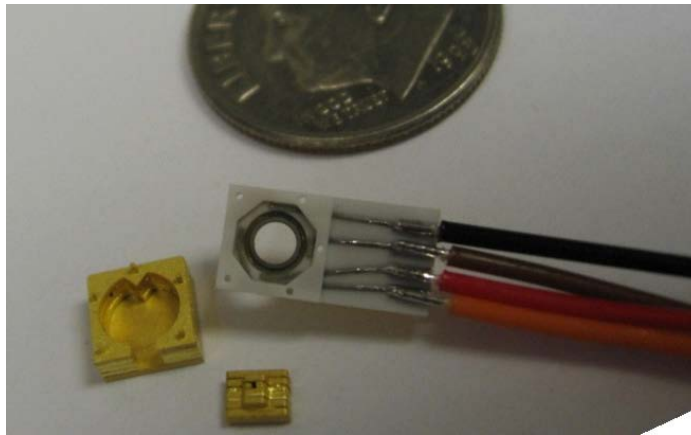


Figure 9. Nearly assembled PAS cell. (Bottom) fiber optic ferrule; (middle left) main PAS cavity with lid attached and alignment pins protruding; (right middle) handling layer with electrical lines for tuning prototype microphones (microphone not shown); (top) dime for scale.

3. INTERFEROMETRIC MEMS MICROPHONE

Microphones with optical transduction are required for electrical line-free remote readout of the acoustic signals. Common telecommunications grade fiber optic are suitable. Not only does this electrically isolate the sensor, but optical based sensing has very high sensitivity relative to electrical methods. Radiation darkening is insignificant with high-OH fibers. Two optical microphone concepts were selected for maturation and integration into the PAS setup. The first is described here, and the second in next section.

3.1. Design

The interferometric displacement concept described here has previously been used in both an optical microphone and an accelerometer [30-32]. During this project, further refinement of the design and methods of fabrication were made. It was selected for its known performance rather than its novelty. The design utilizes an interferometric technique whereby a gold-coated 50/50 transmission diffraction grating is integrated in the middle of the acoustic back-cavity. Together with the gold-coated acoustic diaphragm, a Michelson interferometer is formed. This proven approach provides sub-picometer displacement resolution and pressure sensitivity in the low $\mu\text{Pa}/\sqrt{\text{Hz}}$ range. The acoustic resonant frequency is adjustable from 10 kHz to 100 kHz, depending on membrane stiffness (thickness) and diameter. In our experiments, a He:Ne laser was used to illuminate the grating and diaphragm structure. The zero- and first-order reflections were compared to provide a self-referenced output for common mode noise cancellation using a balanced photoreceiver. This is critical for achieving the required sensitivity levels. The zero order beam must first be attenuated with a linear variable filter to balance the optical power.

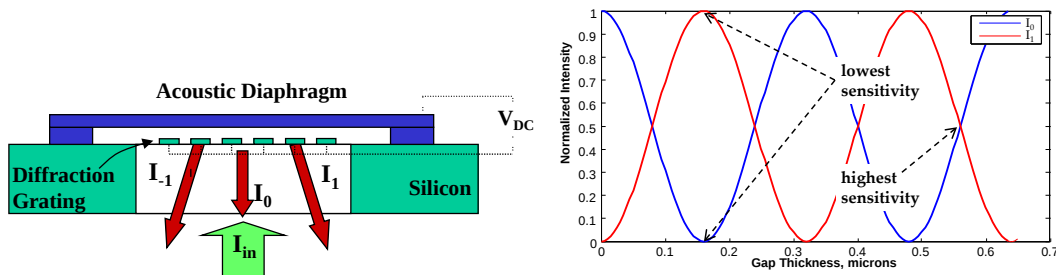


Figure 10. Schematic of the interferometric detection technique.

Another element of the microphone design was electrostatic actuators. Due to the extreme sensitivity of interferometry, there was a high probability that the as-fabricated membrane-grating distance would not be near optimal separation for the measurement laser wavelength. In fact, many functional microphones were at the least sensitive part of the curve. By starting with the electrostatic tuning capability the project was able to proceed without risky and expensive fabrication re-work. Such tuning is not incompatible with the objectives of the project. The required voltage and current can be obtained with on-board optical conversion using photo-cells.

3.2. Fabrication

Microphones were fabricated using the SUMMiT process at Sandia's MESA Fab starting with SOI (silicon on insulator) wafers. Figure 11 shows the flow of wafers through the entire process. Figure 12 shows completed microphones with diaphragms ranging from 0.5 to 2.0 mm diameter. A larger size is more sensitive but also has higher noise characteristics. A more complete description of fabrication details of this microphone and of the full Photoacoustic Spectroscopy system are given in a patent application by Okandan et al [33].

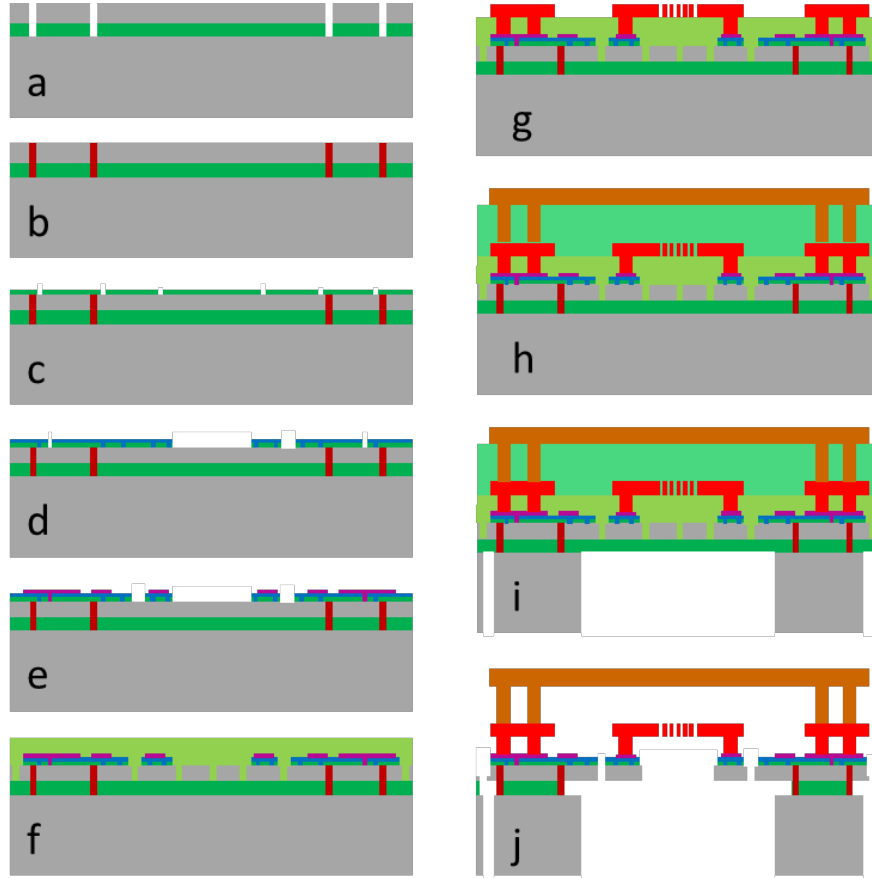


Figure 11. Flow diagram of SUMMiT fabrication process. (a) etch device layer (L1); etch buried oxide; (b) backfill with doped poly; CMP to remove divots; (c) deposit/grow oxide (6kA); etch oxide (L2); (d) deposit nitride; etch nitride/oxide (L3); (e) deposit poly0; etch poly0 (L4); (f) etch oxide/nitride/device layer (L5); fill back with oxide; (release seams) + sacox1; 2um oxide thickness over poly0; (g) etch oxide (L6); deposit 2.25um poly2; etch poly2 (L7); (h) deposit oxide (sacox3); CMP; oxide height 6um above p2; etch oxide (L8); deposit 2.25um poly3; etch poly3 (L9); (i) Bosch etch (L10); (j) release.



Figure 12. Interferometer microphones.

3.3. Static Bias Voltage Characterization

As mentioned above, electrostatic electrodes enable tuning the average separation of the diaphragm-grating separation to optimal sensitivity. A treatment of the theory is presented here.

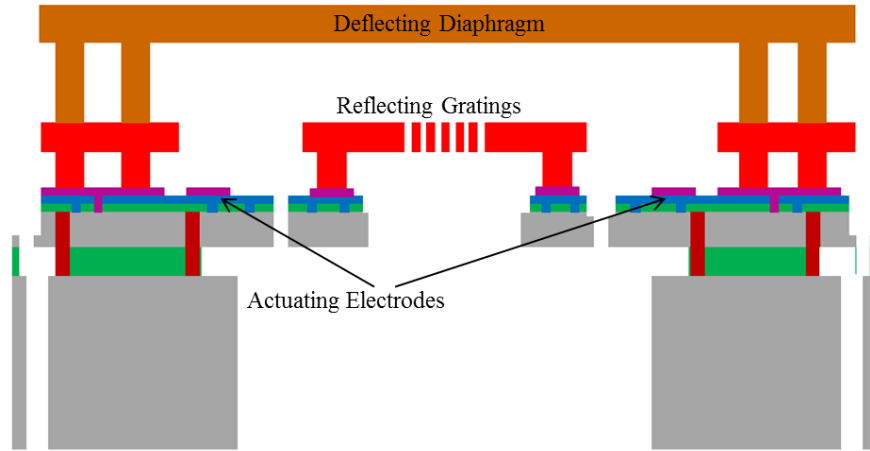


Figure 13. Device cross-section.

The zero and first diffracted orders can be derived using a far field approximation [32]:

$$I_0 = I_{in} \cos^2 \left(\frac{2\pi d}{\lambda} \right) \quad \text{Eq. 1}$$

$$I_{\pm 1} = \frac{4I_{in}}{\pi^2} \sin^2 \left(\frac{2\pi d}{\lambda} \right), \quad \text{Eq. 2}$$

and

where d is the gap distance between the diaphragm and optical gratings, I_{in} is the incident light intensity, and λ is the wavelength. Using trigonometric identities, the diffracted orders can be written as

$$I_0 = \frac{I_{in}}{2} \left(1 + \cos \left(\frac{4\pi d}{\lambda} \right) \right) \quad \text{Eq. 3}$$

and

$$I_{\pm 1} = \frac{2I_{in}}{\pi^2} \left(1 + \cos \left(\frac{4\pi d}{\lambda} - \pi \right) \right) \quad \text{Eq. 4}$$

As shown in Equation 3 and Equation 4, the diffracted orders form a complimentary pair, i.e. they are 180° out of phase when considering the intensity versus the gap distance. Thus, after applying the appropriate compensation to account for the differences in amplitude, a differential scheme can be implemented to enhance the sensitivity and reject laser intensity noise.

A bias voltage between the diaphragm and one of the underlying electrodes results in an electrostatic force on the diaphragm. The diaphragm deflects in response, leading to a decrease in the gap. The bias voltage is used to tune the gap to the point of maximum linearity between the intensity and the displacement of the diaphragm. Theoretically, this occurs at the inflection points of Equation 3 and Equation 4.

The electrostatic force is given as

$$F_e = \frac{\varepsilon AV^2}{d^2} \quad \text{Eq. 5}$$

where ε is the permittivity of air, A is the shared area between the diaphragm and the bottom electrode, and V is the voltage applied. The gap d is defined as the nominal gap, d_0 , minus the deflection due to the electrostatic forcing, d' ,

$$d = d_0 - d' \quad \text{Eq. 6}$$

Thus, the electrostatic force equation becomes

$$F_e = \frac{\varepsilon AV^2}{(d_0 - d')^2} \quad \text{Eq. 7}$$

The electrostatic force is balanced out by the spring force of the diaphragm,

$$F_m = kd' \quad \text{Eq. 8}$$

Equating the two forces results in the following equation relating the diaphragm displacement to the voltage:

$$d'(d_0 - d')^2 = \frac{\varepsilon A}{k} V^2 \quad \text{Eq. 9}$$

Normalizing the diaphragm displacement by the nominal gaps, the equation becomes

$$\frac{d'}{d_0} - 2 \left(\frac{d'}{d_0} \right)^2 + \left(\frac{d'}{d_0} \right)^3 = \frac{\varepsilon A}{kd_0^3} V^2 \quad \text{Eq. 10}$$

The gap is on the order of microns whereas the gap shift induced due to the electrostatic force is on the order of a quarter wavelength of the laser, or 158 nm. Thus, the term d'/d_0 is

small so the squared and cubic term in Equation 10 can be assumed to negligible resulting in the following relationship:

$$d' = \frac{\varepsilon A}{k d_0^2} V^2 \quad \text{Eq. 11}$$

The important result here is that $d' \propto V^2$ for small displacements. The diffracted order intensities are cosine functions of d' and thus V^2 for small displacements. As a result, when determining the point of maximum linearity, the intensity measurements are plotted versus the square of the bias voltage.

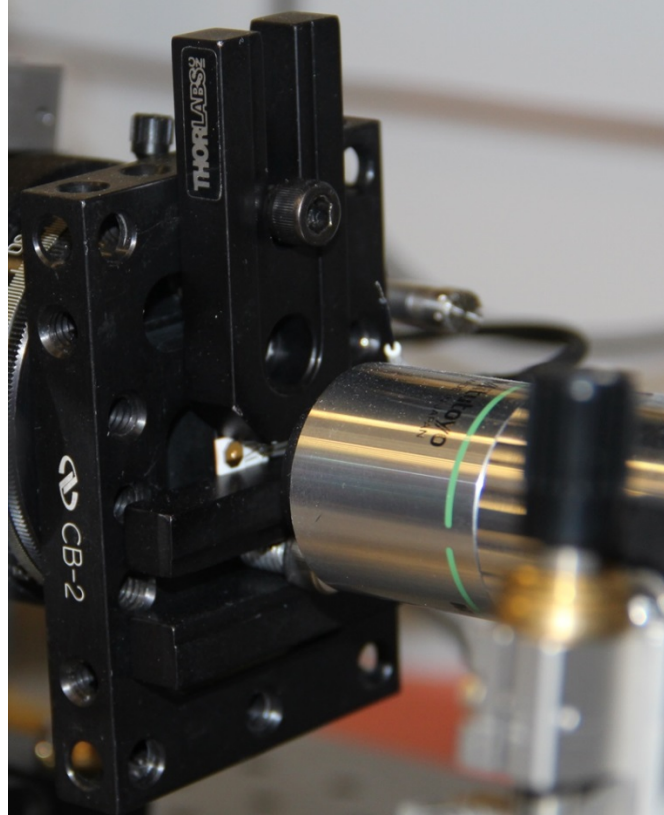


Figure 14. Interferometric MEMS microphone mounted to bottom of PAS cell with laser light focused on diffraction grating through microscope lens.

The following describes the experiment to tune the bias voltage to the optimal operating point of the microphone using the setup depicted in Figure 15. A He:Ne laser (633 nm) was used to interrogate the microphone (Figure 14). The zero and first diffracted order intensities were measured separately with a Newport Model 2107 differential photodetector. The relative intensities of the two signals were balanced using an optical attenuator to eliminate signal bias offset. The bias voltage was applied between the diaphragm and the actuating electrode in Figure 13. The output voltage was then recorded for the two diffracted orders for a series of bias voltage settings.

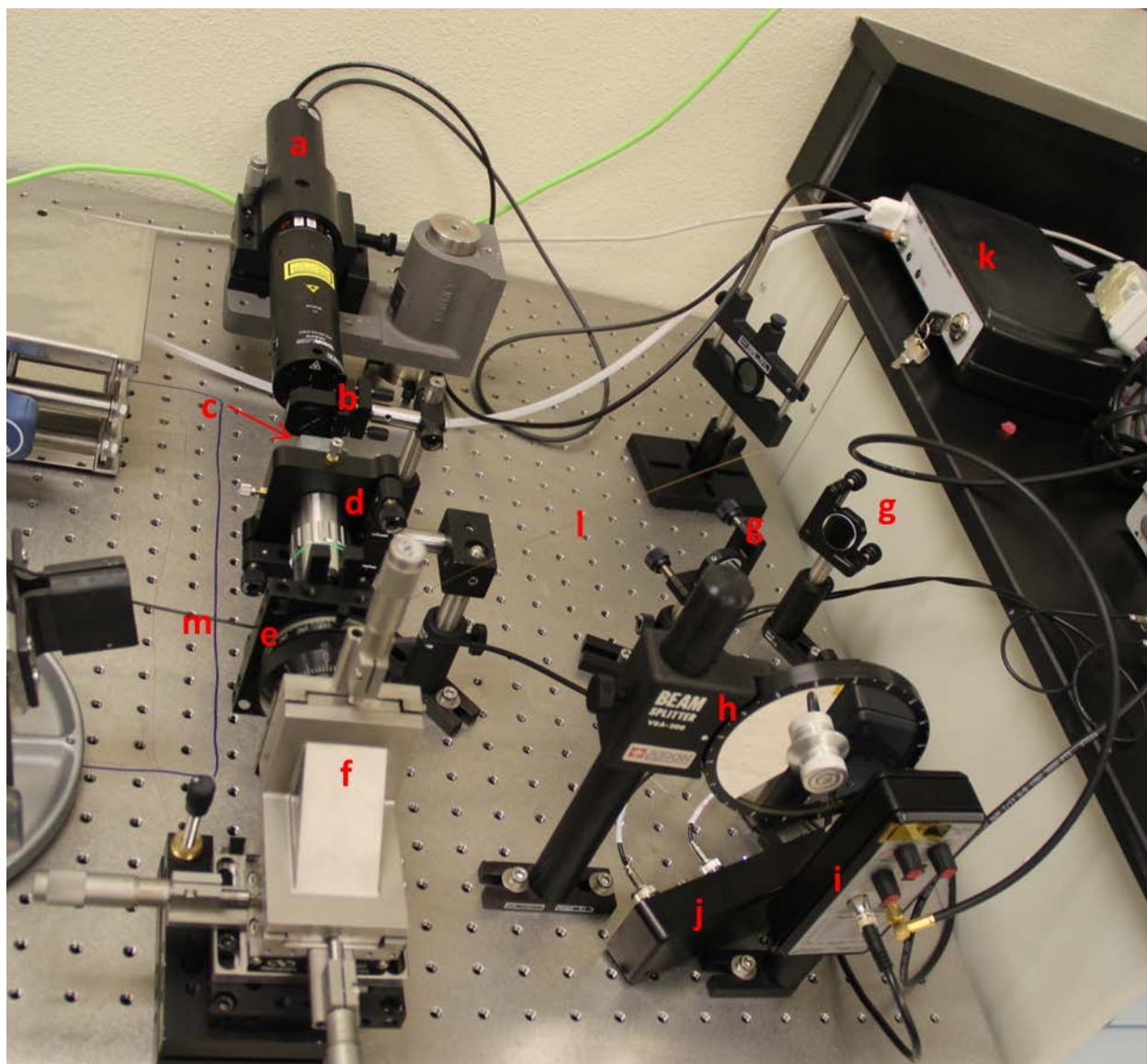


Figure 15. Experimental setup. (a) stabilized He:Ne laser; (b) iris; (c) 50:50 beam splitter; (d) microscope objective; (e) rotational stage (optional) and clamp holding microphone or fully integrated PAS cell; (f) 3-axis micrometer stages holding (e); (g) mirrors (2x); (h) neutral density filter wheel; (i) differential photodetector; (j) electrostatic voltage interface box; (k) laser power supply/controller; (l)* capillary tubing; (m)* PIR fiber optic interfacing PAS cell to FTIR (off screen). *Only for FTIR testing.

The experimental results of bias voltage versus the photodiode output are shown in Figure 16. The approximate point of maximum linearity and sensitivity occurred at a bias voltage of 11 V, as shown in by the black line. All of the subsequent acoustic measurements were done using 11 V as the bias voltage.

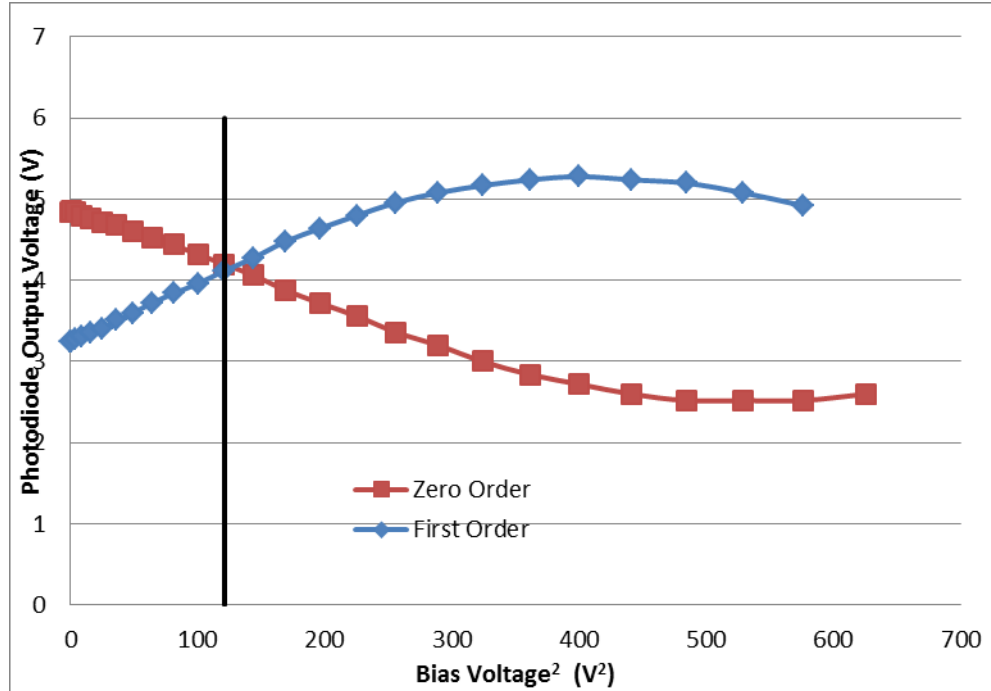


Figure 16. Optical output versus bias voltage to tune gap to point of maximum linearity.

3.4. Acoustic Testing

The following describes an acoustic experimental setup and the results used to estimate the sensitivity of the optical microphone. The test setup consisted of an acoustic plane wave tube (PWT), an instrumentation grade 4938 Bruel and Kjaer ¼ inch pressure-field microphone (Reference), and the device under test (DUT), i.e. the optical microphone. A PWT is a rigid waveguide designed such that only planar waves propagate below its cutoff frequency, which is dependent on cross-sectional geometry and the isentropic speed of sound of the associated gas, in this case air. The plane wave tube is excited via an acoustic driver, in this case a BMS 4593ND 1.4" coaxial neodymium compression driver. During normal operation, the plane wave tube is terminated by a sound hard boundary condition. Therefore, excitation at frequencies below the cutoff will result in standing waves. The Reference and DUT were mounted in the sound hard termination and were simultaneously exposed to approximately the same oscillatory pressure for drive frequencies less than the cutoff frequency.

The DUT in this experiment was the optical microphone. The optical microphone was interrogated using free space optics. This prevented mounting of the optical microphone in the sound hard termination for multiple reasons. Mainly:

1. free space optical interrogation required backside access to the microphone diaphragm, which exposed the acoustic back cavity, changing the dynamics of the microphone and exposing it to external acoustic signals; and
2. the optical interrogation would be susceptible to vibrations from the PWT relative to the rest of the elements in the optical path, and would produce a signal coherent with the acoustic signal.

For these reasons, the sensitivity was investigated via a free space acoustic test. Free space acoustic tests are known to be highly inaccurate, but sufficed for our purposes. An acoustic calibration requires that the two microphones receive the same magnitude pressure signal to get an accurate sensitivity estimate and the signal should also be simultaneous so that the phase response can be estimated. The requirements of the free field calibration are 1) known microphone locations, 2) fully characterized sound field, and 3) elimination of acoustic reflections. This can be achieved with great effort by testing in an anechoic facility and performing a full far-field characterization of the acoustic source. This was determined to be beyond the scope of this project.

We used a non-terminated PWT tube as our acoustic source. Due to the free-space optics and required breadboards, it was not possible to eliminate, but only to minimize, acoustic reflections. The acoustic test was able to determine that the optical microphones indeed responded to acoustic waves, which allowed an estimated sensitivity within an order of magnitude. The microphones were down-selected for placement in the photoacoustic setup based on the relative response of the DUTs with respect to each other.

A sample of the characterization results is given in Figure 17. For this test, the differential photodetector was set to a gain factor of 30,000, the bias voltage was 11 V, and 100 averages were taken. The amplitude of the frequency response function and the coherence between the DUT and Reference are plotted. A coherence near 1 indicates good signal to noise ratio and a low random uncertainty in the frequency response function estimation. The microphone sensitivity was predicted to be flat over the frequencies tested. The response was not flat since the two microphones were in different acoustic fields due to the standing wave patterns formed by reflections off the walls, breadboards, etc. in the lab. Also, the backside of the microphone was exposed such that low frequency acoustic waves could equilibrate across the diaphragm before the diaphragm reacted.. This explains the low frequency roll-off in the frequency response function response.

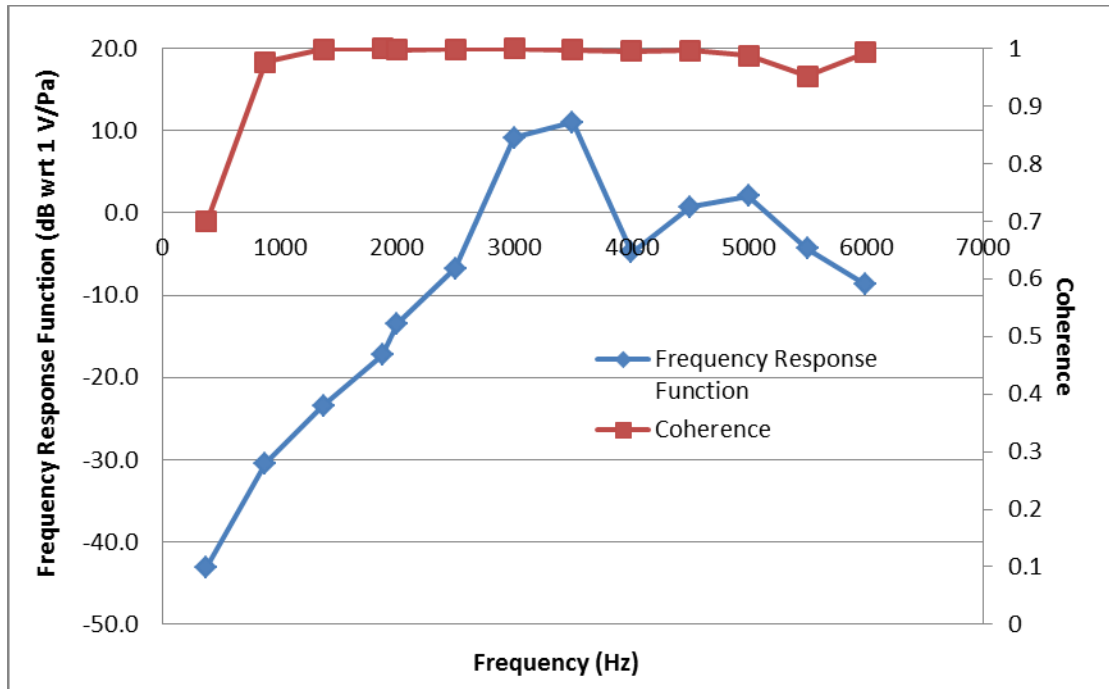


Figure 17. Acoustic measurement of the frequency response function and corresponding coherence.

4. OPTICAL RING RESONATOR MICROPHONE

In a completely novel approach, microphones were fabricated with optical waveguide micro-ring resonators integrated into the acoustic diaphragms. Each optical resonant cavity was accessed through an optical waveguide “bus” running in close proximity such that evanescent coupling (transfer) of light could occur. Wavelengths of light resonant with the optical properties of the resonance cavity were strongly coupled and transferred from the waveguide to the cavity while other wavelengths remained in the bus. The light would subsequently transfer to a second, identical waveguide that started with no light. Light in both the “Through” bus and the “Drop” bus could be monitored at their output ports. The pair of waveguide’s provided a “positive” and “negative” energy spectrum of resonant wavelengths of light when a tunable diode laser focused into the Through bus was scanned.

4.1. Theory

Theory showed that strain from deflection of the microphone membrane would cause a shift in the wavelength of light resonant within the optical cavity [34]. This is similar to the response of a Fabry-Perot cavity. These concepts are depicted in Figure 18 and Figure 19. To monitor a microphone, the diode laser would not be scanned, but rather tuned to a wavelength located at the half-maximum of a resonance. As the microphone membrane oscillated, the intensity of the signal would modulate with amplitude equal to the resonance’s slope times the resonant frequency shift. This effect would be linear over a limited range, determined by the microphone’s stiffness and the optical resonator’s quality factor (Q). Both can be tuned during microphone fabrication.

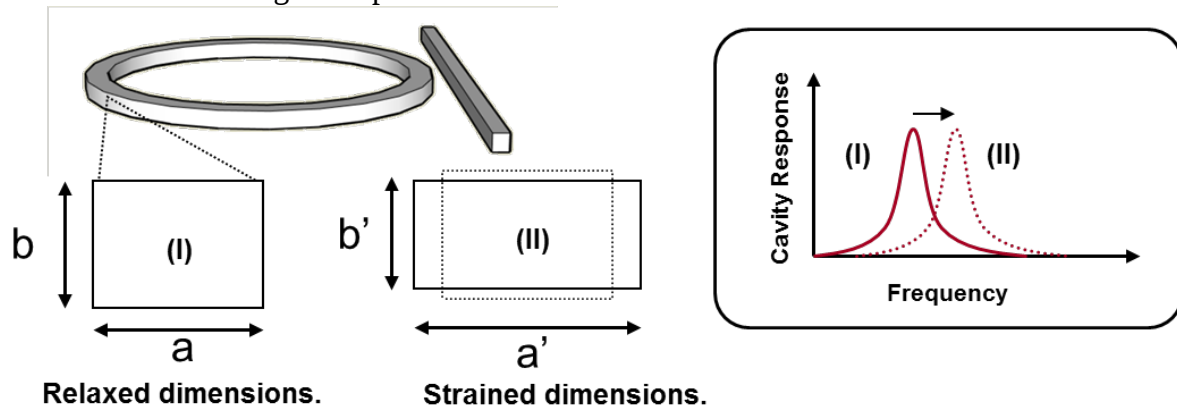


Figure 18. Schematic of a micro-ring cavity (top left) next to a segment of a waveguide bus. When subjected to strain the dimensions of the ring change (lower left and middle), which causes the resonant wavelength(s) to shift (right).

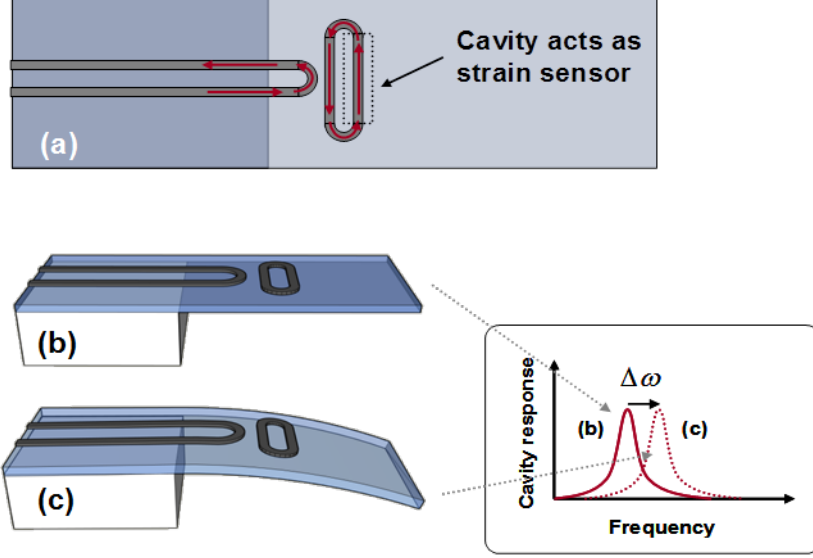


Figure 19. Conceptual layout of a strain micro-ring strain sensor. The location could be a microphone membrane, cantilever, or other location that may flex.

Theory predicts a micro-ring cavity with a $Q \sim 10^6$ should be possible using our theoretical materials approach. With such a resonance and Pound-Drever-Hall frequency locking [35], the cavity optical frequency could be measured to about 1kHz precision. Thus, strain would be measurable to approximately 1 part in 10^{11} .

4.2. Design

This microphone design started with gaining an understanding of the effects of strain on the ring resonator waveguides. Modeling through the equation below showed that a small range of cross-sectional dimensions would result in strong coupling of strain transduction via wavelength shifting (Figure 20). The waveguide construction of both the micro-rings and buses would be identical, as required by the 2-D fabrication design on top of a buried oxide layer, which also served as the etch stop when the membrane was Bosch etched. The membranes would be complicated structures of BOX (buried oxide), silicon, nitride, and addition oxide layers. These were required to work together as a compliant microphone membrane with minimal compressive stress. That aspects of microphone deflection and strain are addressed below.

$$\frac{\delta\omega}{\delta S_{xx}} = -\frac{\omega}{n_g} \left[\left(\frac{\partial n_{eff}}{\partial n_c} \frac{\partial n_c}{\partial a} \cdot a + \frac{\partial n_{eff}}{\partial a} \cdot a - \sigma \frac{\partial n_{eff}}{\partial b} \cdot b \right) - \sigma n_{eff} \right]$$

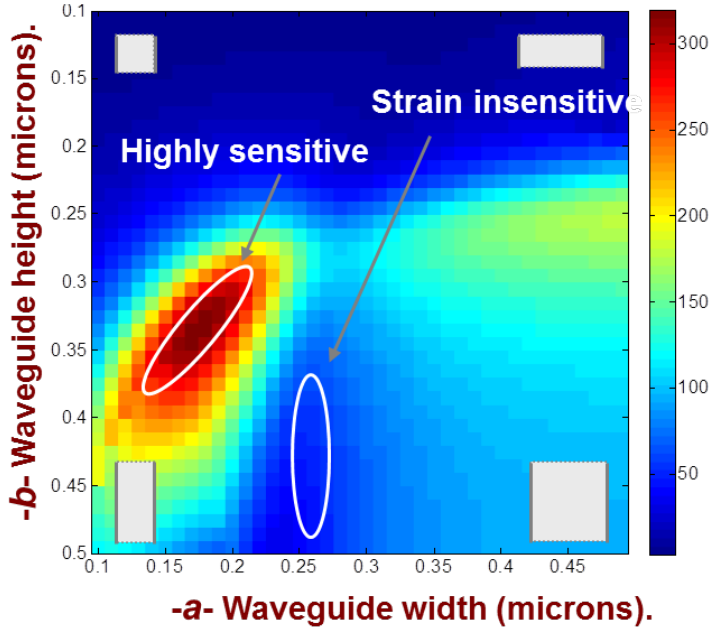


Figure 20. Modeling of frequency sensitivity of TE cavity mode (THz per unit strain, S_{xx}) to strain for a range of waveguide aspect ratios.

The layout of the a single set of waveguides and the section of microphone membrane into which they are integrated are shown in Figure 21. The theory, modeling, and experimentation used to arrive at this design point are described in the sections that follow.

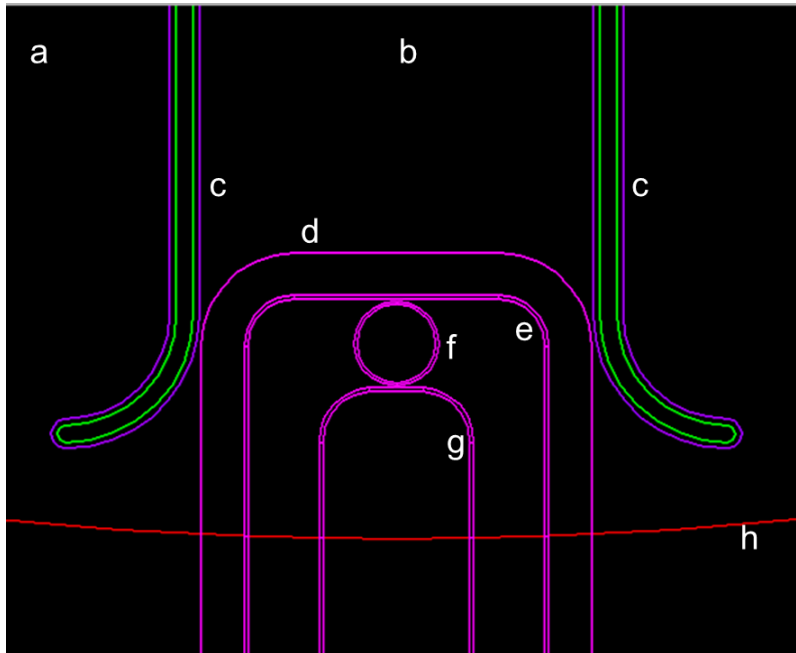


Figure 21. Layout of waveguides on microphone. (a) microphone membrane; (b) bridge connecting (a) to the device substrate; (c) trenches separating the membrane from the bridges supporting it; (d) edge of waveguide oxide layer; (e) Through waveguide that carries light from the laser; (f) micro-ring optical resonator; (g) Drop port that carries light evanescently

coupled from the Through port via the micro-ring; (h) edge of the Bosch etch, which defines the boundary of the microphone back-cavity.

4.3. Acoustic Modeling

The novel aspect of this microphone lies in its ability to convert an acoustic signal into a measurable optical signal. This is accomplished via integrated waveguide-based strain sensors located at the anchored ends of the microphone tethers that detect strain as a function of sound pressure induced membrane deflection. However, this is just one component of an acousto-mechanical-optical system that comprises the overall microphone. Before delving into the operating principles surrounding the strain sensors which will be covered in another section, an explanation of the lumped element modeling created to help guide our mechanical design is in order. This modeling technique is commonly used to predict frequency response in MEMS microphones and other transducers. There are several advantages to using this type of modeling. For one, it provides a visual representation of a system from which differential equations may be directly formed [36]. Secondly, these circuit models are intrinsically correct from an energy point of view [37] and are useful for combining electrical, mechanical, and acoustic systems into one model. In our case, we are treating the microphone as a classical plane circular piston mounted in an infinite surface or baffle.

In this model, acoustic elements are defined as follows. The diaphragm is modeled as a lumped mass, M_{ad} , and compliance, C_{ad} , and damping is included as a resistance, R_{ad} , that accounts for loss mechanisms such as thermoelastic dissipation and anchor/support loss. In our model, the acoustic mass is equivalent to the mechanical mass of the diaphragm divided by the square of the diaphragm surface area, S^2 , and is represented by an inductor symbol with units of kg/m^4 . Since our microphone diaphragm is a tethered piston, its acoustic compliance is equal to the compliance of a single tether, C_{teth} , divided by the number of tethers, n , and multiplied by S^2 . Besides damping losses associated with the diaphragm, other forms of acoustic energy loss must be taken into account such as energy radiated into the air by the diaphragm known as radiation resistance, $R_{ad,rad}$. One of the more dominant forms of acoustic loss is the loss of energy around the perimeter of the diaphragm or vent acoustic resistance, R_{av} . Normally, this quantity is limited by the presence of a capacitive back plate in traditional condenser microphones. However, since our microphone does not have a back plate, it becomes even more important to control this quantity by minimizing the cross sectional area of the diaphragm gap that the acoustic wave sees. Otherwise, the microphone's flatband frequency response could be severely degraded. Our model determined that a gap of $2\text{ }\mu\text{m}$ around the diaphragm perimeter was as large as could be tolerated to avoid this. As can be imagined, this made process integration a challenge especially with an $11\text{ }\mu\text{m}$ thick film stack that required a 5:1 aspect ratio RIE dry etch to pattern.

Finally, the acoustic compliance of the air filling the silicon back cavity must be accounted for. In case our vent resistance was not big enough, our mitigation strategy was to increase the back cavity compliance by extending the length of the back cavity hole using packaging structures. Fortunately, the required gap spacing of $2\text{ }\mu\text{m}$ did not go beyond the capability of our etching tool making this option unnecessary. A circuit diagram depicting the acousto-mechanical features of our optical microphone is shown in Figure 22.

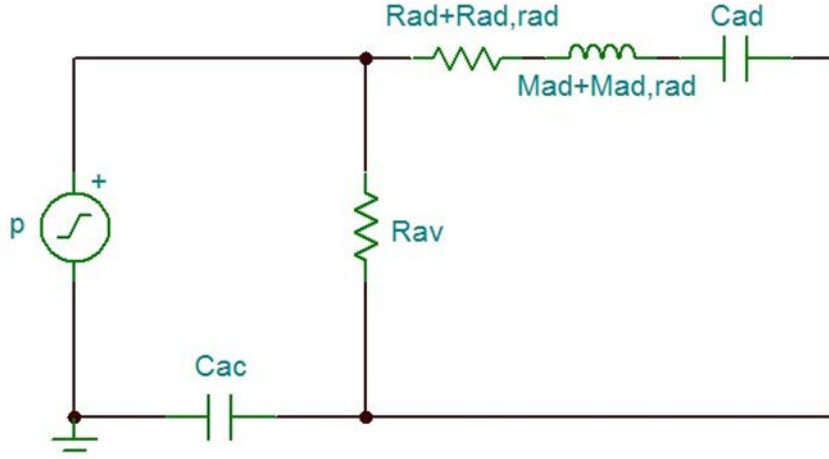


Figure 22. Circuit diagram depicting lumped element microphone acousto-mechanical model. (M. Williams and E. Langlois, Sandia National Labs, 04/19/12)

The variables used by the model, their definitions, governing equations and values used in the model are listed in **Table 2**.

Table 2: Inputs for microphone lumped element model.

Variable	Description and Units	Equation	Values
p	effective sound pressure (Pa)		1V sine wave
M_{ad}	diaphragm acoustic mass (kg/m ⁴)	$M_{ad} = M_m/S^2 = (\sum_i^n \rho_i t_i)/(\pi a^2)^2$ ρ = layer density, t = layer thickness, a =diaphragm radius	3.24E4 kg/m ⁴
$M_{ad,rad}$	radiation acoustic mass (kg/m ⁴)	$0.270 \rho_0/a$	8.16E2 kg/m ⁴
C_{ad}	diaphragm acoustic compliance (m ⁵ /N)	$C_{ad} = (C_{tether}/n) \times S^2$ C_{tether} = tether compliance; n = # of tethers	1.46E-16 m ⁵ /N
C_{ac}	back cavity acoustic compliance (m ⁵ /N)	$C_{ac} = V/\rho_0 c^2$ V = back cavity volume; ρ_0 = density of air; c = speed of sound in air	3.58E-15 m ⁵ /N
R_{ad}	loss mechanisms (thermoelastic dissipation, anchor/support loss, etc.) (N-s/m ⁵)	$R_{ad} = 2\zeta\sqrt{M_{ad}/C_{ad}}$ ζ = 0.01, order of magnitude approximation for damping	3.01E4 N-s/m ⁵
$R_{ad,rad}$	radiation resistance (N-s/m ⁵)	$Rad,rad = 0.159\omega^2 \rho_0/a$	variable
R_{av}	vent acoustic resistance (N-s/m ⁵)		variable

One of the more difficult variables to quantify is the vent acoustic resistance. Obtaining reliable values is crucial as this parameter greatly affects the frequency range of operation, particularly the microphone cut-on frequency. Since this is a rather complicated fluid mechanics problem, we resorted to finite element modeling (FEM) using COMSOL v4.1. Two types of fluid models were built to determine acoustic resistance. One model was a full

3D simulation of the volumetric flow rate of air through the gap between the microphone tether/membrane and the substrate edge at a differential pressure of 0.1 Pa. Angular symmetry was employed in all of our designs such that the flow around one tether could be multiplied by the number of tethers in the design to arrive at the total volumetric flow. This type of 3D modeling was performed on two different tether designs. A second model was built for air flow through a 2D slit of width, w , and height, h . For a constant differential pressure, dP , of 0.1 Pa, the areal flow rate, Q_0 , for slit dimensions (w/h) of $9\mu\text{m}/7\mu\text{m}$ and $7\mu\text{m}/1\mu\text{m}$, respectively, were modeled and plotted in Figure 23.

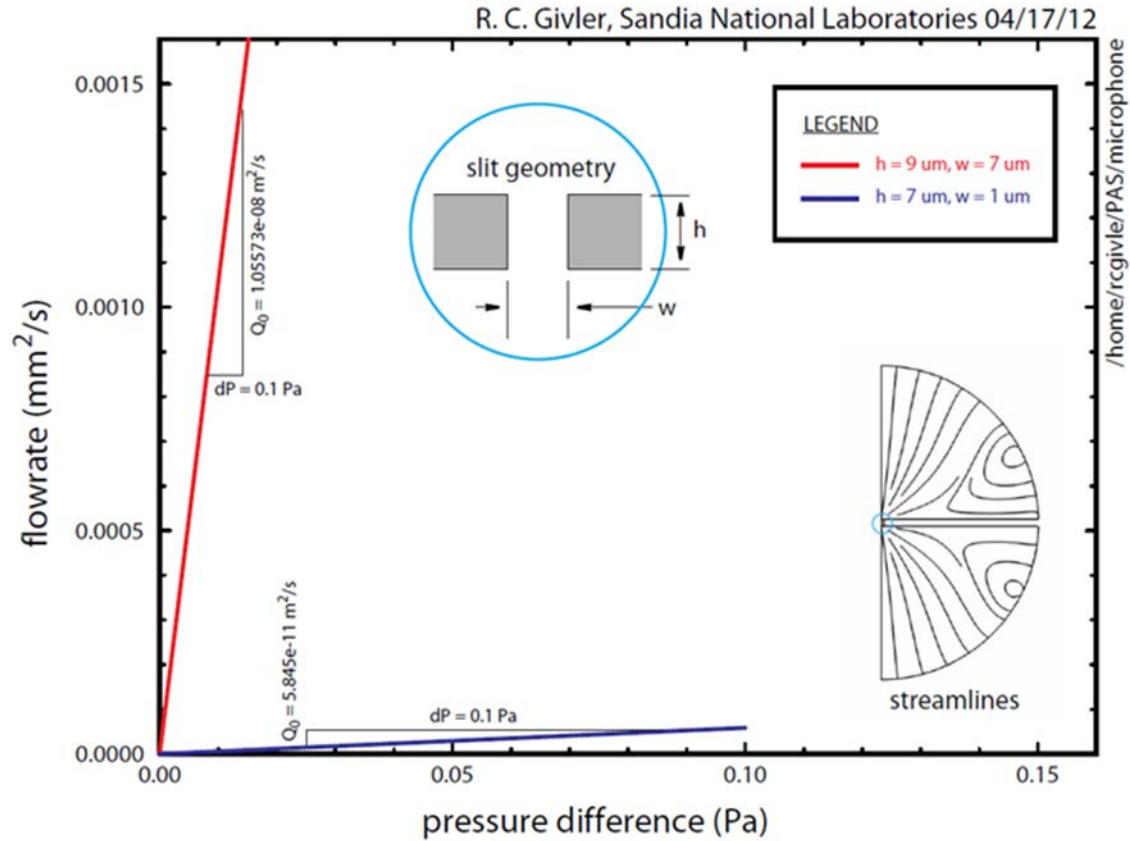


Figure 23. Flowrate (per unit depth) vs. pressure difference for steady, creeping air flow through a 2D slit (dimensions shown above).

The inverse slope of the lines provide the vent acoustic resistance with units of $\text{N}\cdot\text{s}/\text{m}^5$. Upon careful examination of the two models, it was observed that a simple linear relation could be used to estimate the flow rate through 3D slit geometries by multiplying the 2D areal flow rate, Q_0 , by the total arc length, s_{tot} , of the gap along the membrane perimeter. This comparison is shown in Figure 24 with an estimated error between 2% and 4%.

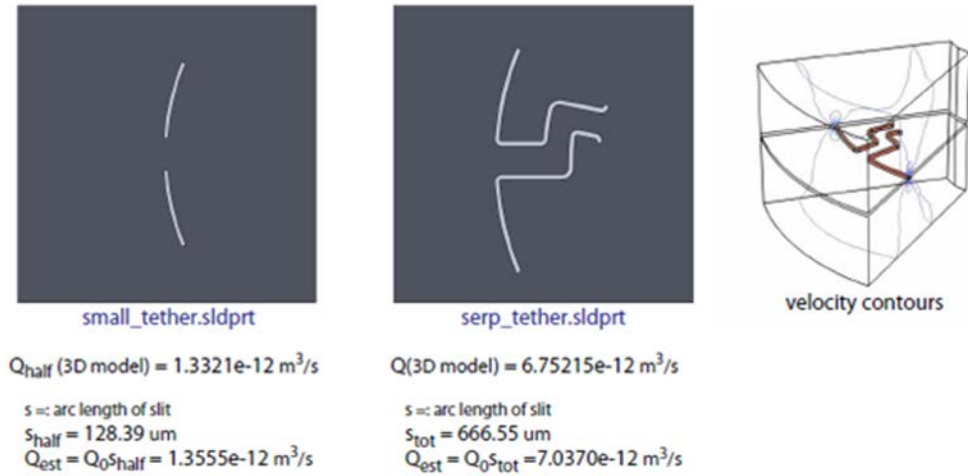


Figure 24. Comparison of volumetric flow rates obtained directly by 3D FEM and indirectly by multiplication of the 2D FEM areal flow rate, Q_0 , by the total arc length, s_{tot} , of the slits.

Again, dividing dP by the calculated volumetric flow rate Q_0 will give the vent acoustic resistance.

Once this parameter has been determined, a simple analytical model of the microphone frequency response can be built. For this purpose, MATLAB v. R2010b commercial software was used to solve for the normalized diaphragm deflection per unit pressure in dB as a function of frequency in Hz. Plots for both 8 tether and 3 tether microphone designs are shown in Figure 25.

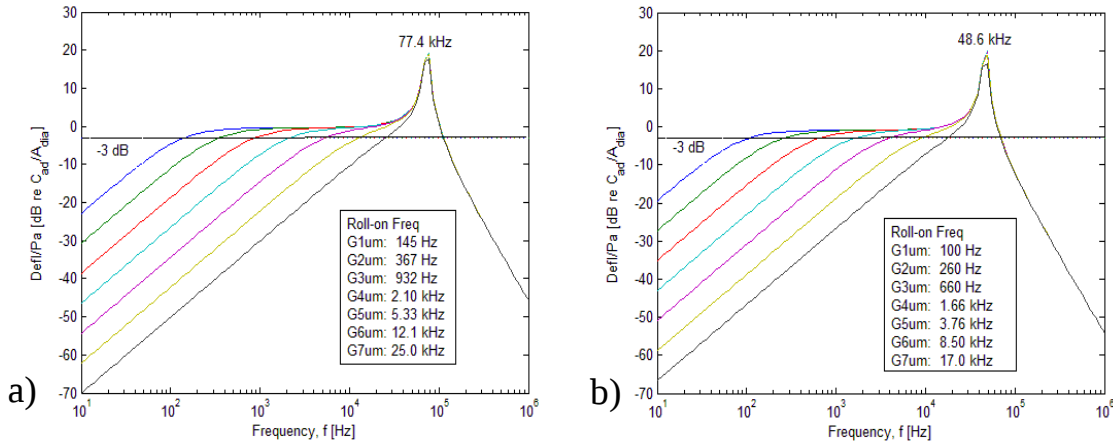


Figure 25. Frequency response plots for a) 8 tether microphones and b) 3 tether microphones.

The frequency band associated with sound waves audible to human ears ranges from 20 Hz to 20 kHz. A constant sensitivity across this range, i.e., a flat frequency response, is desired to give equal weight to all signals. Since microphones are fundamentally mechanical devices, they possess resonance frequencies that will amplify those signals unless care is taken to ensure they are beyond the audible range. For the two tether designs shown, we have

resonance frequencies of 48.6 kHz (3 tether) and 77.4 kHz (8 tether), respectively, well beyond the audible range. As resonant frequency increases as the square root of the stiffness, this makes sense as more tethers result in stiffer microphone membranes. Fewer than three tethers could result in membrane deflections that aren't constrained to the membrane normal axis and might result in unwanted torsional modes that will distort the signal. Hence, our designs should perform well with three tethers or greater.

Similarly, we need to make sure the roll-on frequency is sufficiently low to capture low frequency content of interest. Typical audio microphones are designed to cut off at around 100 Hz to eliminate wind and other low frequency noise. As previously mentioned, our low frequency roll-on is strongly influenced by the vent acoustic resistance of the diaphragm gap. Figure 25 shows how roll-on frequencies vary as a function of gap width ranging from 1 μm to 7 μm . As the gap increases, our bandwidth decreases and we lose sensitivity to low frequency signals. Fortunately, our etch process is capable of two micron wide gaps through the 10 micron thick composite film comprising the microphone diaphragm. This sets our roll-on frequency to somewhere between 200 Hz and 400 Hz, depending on the tether design used, which is sufficient for our purposes as a chemical sensing microphone. One alternative option for lower frequency fidelity is to increase our backside cavity compliance by increasing the length of our cavity through packaging means. This is an easier option than using thicker substrates or bonding a second wafer with an aligned cavity hole that would entail much greater process complexity.

4.4. ANSYS Modeling – Membrane Buckling

For the first design pass, we laid out a set of circularly clamped diaphragm membranes where the ring resonator was located at the clamped edge. However, it was soon realized that due to an etch bias error of around 25 microns, it was too difficult to control where this edge was going to land. This placed the diaphragm perimeter either in front of the ring resonator (no strain) or far behind it (minimal strain). This prompted a redesign towards a tethered piston diaphragm comprising a circular membrane supported by a set of tethers fabricated from the same membrane layers. From a simplistic point of view, the circular membrane is a rigid structure mechanically supported and guided by the tethers which are clamped to the more rigid membrane structure that overhangs the edge of the backside cavity. This way, the backside cavity perimeter is designed larger than the perimeter of ring resonators to mitigate the etch bias. It also defines the point of maximum strain by pattern lithography and not by the Bosch etch. However, despite this seemingly elegant solution, another problem arose where the intrinsic stresses of the constituent material layers caused a large degree of diaphragm buckling due to the relaxed mechanical constraints of the tethered design as compared to the more rigid constraints in the circularly clamped diaphragms. Figure 26 shows a SEM image of a version 2, 8-serpentine tether (radially asymmetric) microphone. What is clearly discernible is the gross, out-of-plane deflection of the diaphragm “petals” from the surrounding chip surface ($\approx 28 \mu\text{m}$ to $30 \mu\text{m}$) due to the stress gradient caused by the compressive oxide and tensile polysilicon layers. What is not so apparent from the image is the $-7 \mu\text{m}$ deflection of the diaphragm center from the chip surface causing the overall structure to buckle concave up due to the combination of the stress gradient and the net compressive stress made dominant by the oxide layers. While the latter is not desired, it is the former that raises concerns regarding the acoustic properties of the microphone. This degree

of out-of-plane buckling provides an effective gap that far exceeds the as designed 2 μm gap to provide the desired roll-on frequency. To understand this material structure better and to provide some predictive capability to improve upon our existing process flow and design, a finite element model was created using ANSYS v.14.0 commercial software. This model uses shell elements which simplify the 3D structure by treating it as a 2D structure thus reducing the degree of computational complexity and time. Individual layer thickness and stress is input via shell elements while the 2D geometry is input via SAT files generated by AutoCAD 2011 v.225. Individual layer stress values determined experimentally and used in the model are as follows: -300 MPa for silicon dioxide, +325 MPa for polysilicon and 0 MPa for single crystal silicon. Figure 26 b) shows an ANSYS FEM plot that closely represents this buckling for a similar (radially symmetric) design and material layer stack-up. Discrepancies between the real device and the model can be attributed to a variety of factors including inaccurate layer stress values and bottom oxide erosion during the Bosch etch.

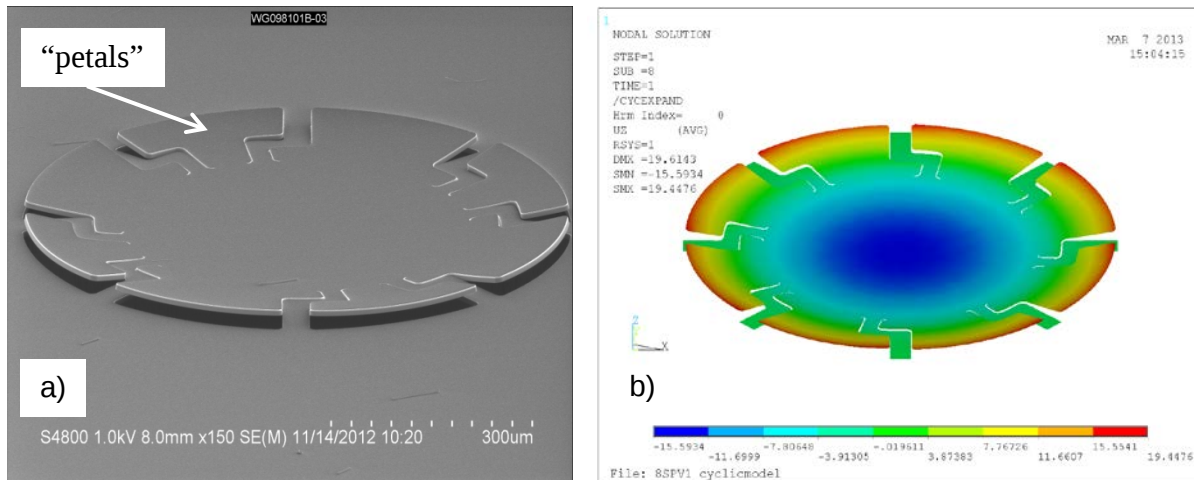


Figure 26. a) SEM of buckled v.2 8 tether diaphragm showing extreme out-of-plane buckling. b) ANSYS FEM plot predicting this degree of buckling in a similar design with the same material layer stack-up.

Guided by the model, the version 3 microphone was redesigned to balance out the overall stress and flatten the diaphragm as much as possible. Simulating different combinations of layer thickness and stress in an iterative fashion helped further our understanding of these two parameters on buckling. It also pointed the way towards an optimal set of process flows bounded by current achievable fab processes. Reducing the number of thickness and stress combinations made it possible to isolate and optimize the effects of geometry in reducing edge deflection. Despite these efforts, there is still a significant amount of buckling in the version 3 diaphragms with a center deflection of $\approx 25 \mu\text{m}$. This is to be expected due to the presence of compressively stressed oxide layers needed for the waveguide optics. However, the edge deflection is substantially reduced to around $-9 \mu\text{m}$ that matches the $-5 \mu\text{m}$ to $-9 \mu\text{m}$ predicted by the model as shown in Figure 27 a,b,c.

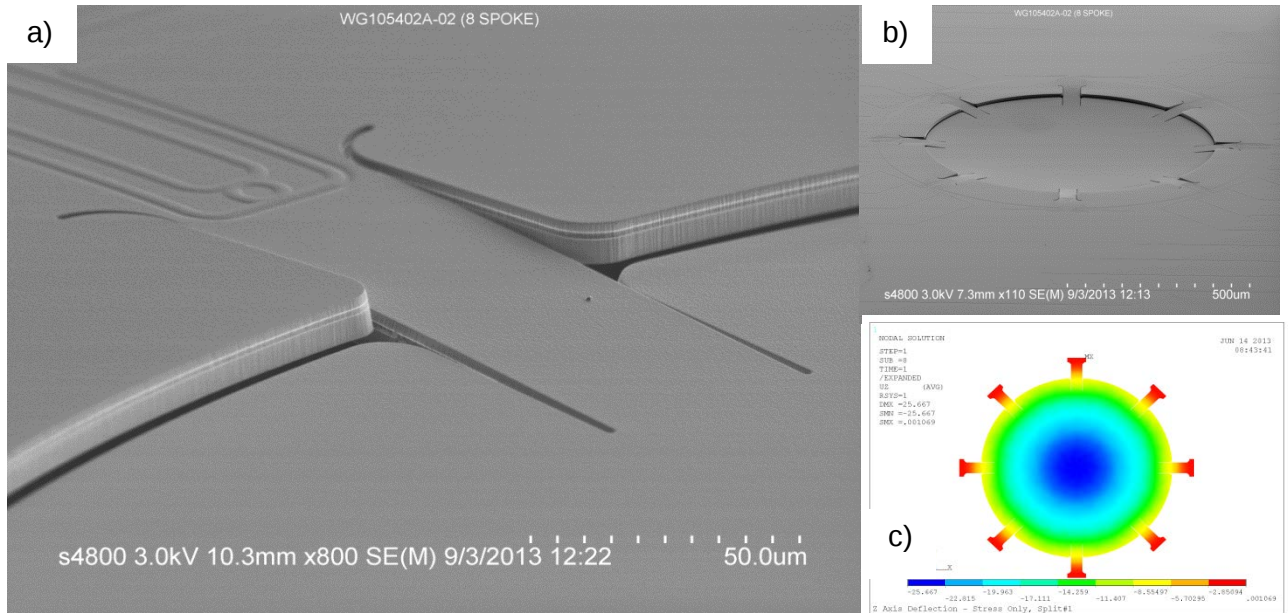


Figure 27. a) SEM close-up image of a single tether showing $\sim -9 \mu\text{m}$ of out-of-plane deflection. b) SEM image of entire 8-tether microphone diaphragm. c) ANSYS FEM displacement plot of this diaphragm design. $-5 \mu\text{m}$ to $-9 \mu\text{m}$ of diaphragm deflection predicted by the model matches the observed value.

4.5. ANSYS Modeling – Strain vs. Pressure Load

The same ANSYS FEM was employed to indicate the X,Y location of maximum strain in the diaphragm tethers as a function of applied pressure loading. This determined the best position to design our ring resonators for maximum sensitivity to sound pressure. A pressure load of 0.1 mPa was applied to the model membrane which included the individual film stresses discussed previously. A linear solution was obtained for this diaphragm with and without a pressure load. The two solutions were subtracted from each other producing strain results purely from pressure loading. A finite grid of nodes with sufficient spatial resolution was generated to map out and locate where the X,Y location of maximum strain was. At first, it was assumed that strain could be enhanced at a spatially determined location by designing a narrow “neck” in the width of a tether via lithography. However, this was determined by simulation to be no different than designing a straight tether of equal width as the neck and was abandoned for this much simpler design option. Figure 28 compares the maximum strain versus distance from the diaphragm center for variations of both notched tethers and straight tethers. The data shows the highest strain is always located at the anchor edge irrespective of there being a notch present or not. Longer tethers impart a greater strain than shorter tethers which was attributed to the effect of a greater moment arm. Tethers with narrow widths also imparted higher strain than those with wider widths. A tether design width of $48 \mu\text{m}$ was set by the size of the ring resonator and accompanying waveguide input/outputs. A tether design length of $131 \mu\text{m}$ was set due to the desire to limit the length of the diaphragm “petals” and to limit the overall footprint of the microphone to fit more optical designs and mechanical designs with 4,6 and 8 tethers.

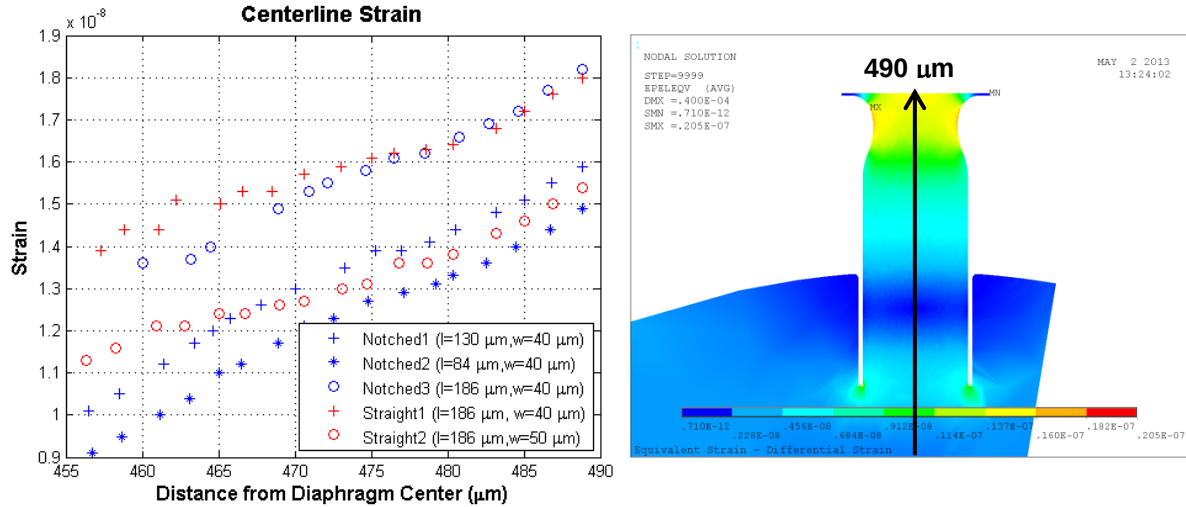


Figure 28. Tether centerline strain as a function of distance from the diaphragm center. The maximum strain is consistently located at the anchors for all tether types.

4.6. Fabrication

4.6.1. Bosch Etch Test Mask

The desire is to create a membrane with an imbedded waveguide that will sense the stress of a deflection in the membrane from a pressure wave. A Bosch etch is used to create an access hole through a standard thickness silicon wafer to access the membrane. One of the problems that have troubled MEMS engineers is compressive residual stress in released devices that causes certain design features (fixed fixed beams, suspended membranes etc.) to buckle. Historically thin membranes created in polysilicon with sacrificial Oxide materials have been problematic and have been shown to buckle and fracture due to compressive stress when the Bosch etch lands on an etch stop layer oxide layer, due to the compressive film exceeding a buckling stress. In this work a method for creating tensile polysilicon was employed where the membrane would be taunt like a drum head rather than in compression which is typical in standard MEMS processes. A test mask was ordered to determine the maximum diameter that would be achievable before the membrane fails. This information would be used to help design the test devices that would survive the Bosch etch. The test mask can be seen in Figure 29. The test wafer film stack can be seen in Figure 30.

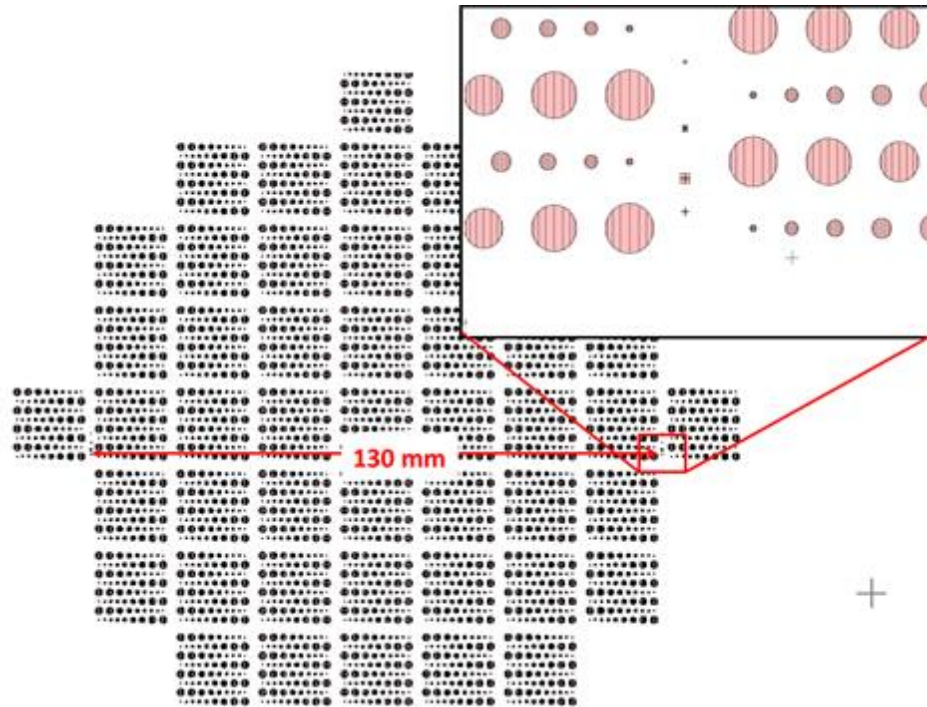
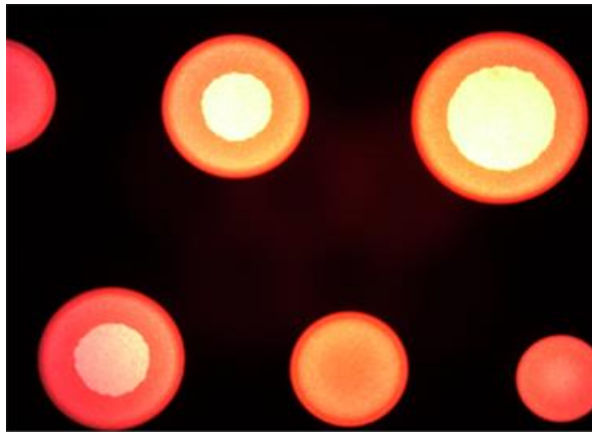


Figure 29. RS924 test mask for interrogating the maximum diameter membrane achievable with Bosch etching on tensile polysilicon.

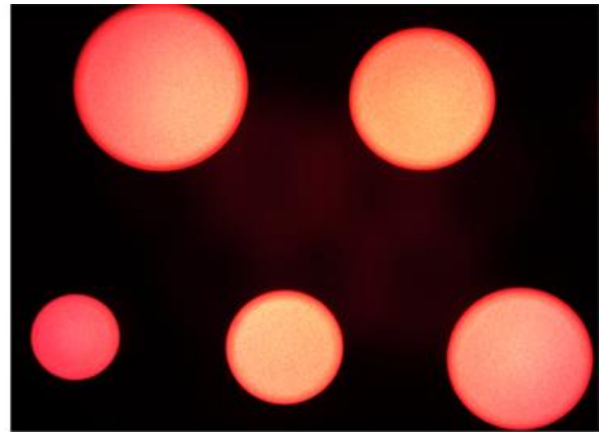
2.0 um USG Oxide
4.0 polysilicon
2.0 um USG Oxide
0.26 um silicon
3.0 um or 1.0 um BOX
WG091501A
3.0 um Thermal Oxide on Backside
0.26 um silicon
4.0 polysilicon

Figure 30. Film stack used for Bosch test wafers

Results from the test wafer Bosch etch showed that tensile polysilicon does not show the typical fracturing of membranes when the Bosch etch lands and would allow up to 1500 um membrane diameters to be fabricated on 3.0 um SOI wafers. SOI wafers with only 1.0 um BOX had breakthrough of the BOX layer when prolonged etching was carried out to clear the smaller features. Results are shown in Figure 31.



1.0 um Buried Oxide (BOX)



3.0 um Buried Oxide (BOX)

Figure 31. Test pattern results from Bosch etch experiment with tensile polysilicon. Features landing on 1.0 um BOX broke through the oxide film on large features. All features were resolved, no membrane fracturing, and no oxide breakthrough on the 3.0 um BOX SOI wafers.

4.6.2. *Gen 1: Device and Fabrication Design, RS915*

Based on the excellent results produced with the Bosch Test Mask a preliminary design was created with waveguide devices. The mask layout shown in Figure 32 is a GDS file showing various configurations of membranes and waveguide geometries.

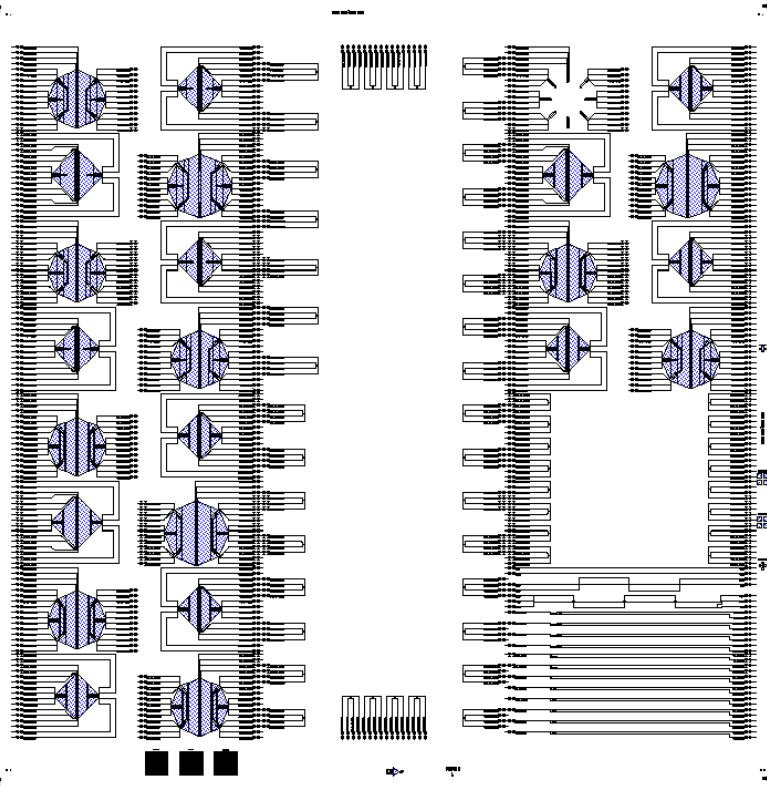


Figure 32. Reticle RS915 device test mask with various configurations of membranes and waveguides.

The method used to create these improved qualities in polysilicon starts with an amorphous silicon (a-Si) deposition at 550°C. Then an oxide layer is applied to the surface at high enough temperature (> 700°C) to cause the silicon to crystallize. The crystallization occurs under the Oxide layer which starts out smooth due to the smooth a-Si surface character and causes the subsequent polysilicon to be very smooth on the surface and uniform in thickness which in some conditions can lead to much stronger mechanical strength [38]. Polysilicon created under similar conditions was demonstrated to be stronger than the typical SUMMIT polysilicon, and based on that work a design of experiment (DOE) was created to investigate a wide variety of stress conditions for this application. The DOE results shown in Table 3 list the pre and post anneal effect on ROC.

Table 3 – Final ROC results post anneal for tensile polysilicon membrane material.

Wafer	CMP	Poly 4 – (4.0 um Poly) 2 – (2.0 um Amorphous Silicon)	Anneal	Etch depth of poly. Land 0.5 um	ROC Pre anneal	ROC post anneal (m) Positive ROC indicates tensile poly
1	No	4	750°C	0.5	+822.43	+185.21
2	No	2	1100°C	land	+240.71	-68.925
3	No	4	1100°C	land	-493.72	-45.753
4	No	2	750°C	0.5	+182.06	+90.205
5	No	4	0	N/A	-1295.8	
6	No	2	0	Holdback	-4496.7	
7	CMP	4	750°C	0.5	+216.29	+113.15
8	CMP	2	1100°C	land	+389.34	-52.102
9	CMP	4	1100°C	land	+1210.0	-39.788
10	CMP	2	750°C	0.5	+34818.0	+198.760
11	CMP	4	0	Hold back	-5100.6	
12	CMP	2	0	N/A	-2200.5	

Images of the first devices fabricated with the RS915 mask set can be seen in Figure 33 where the waveguides are distributed in varying symmetric patterns. Symmetry was deemed necessary to avoid any asymmetric deflection in the films due to the realized stress.

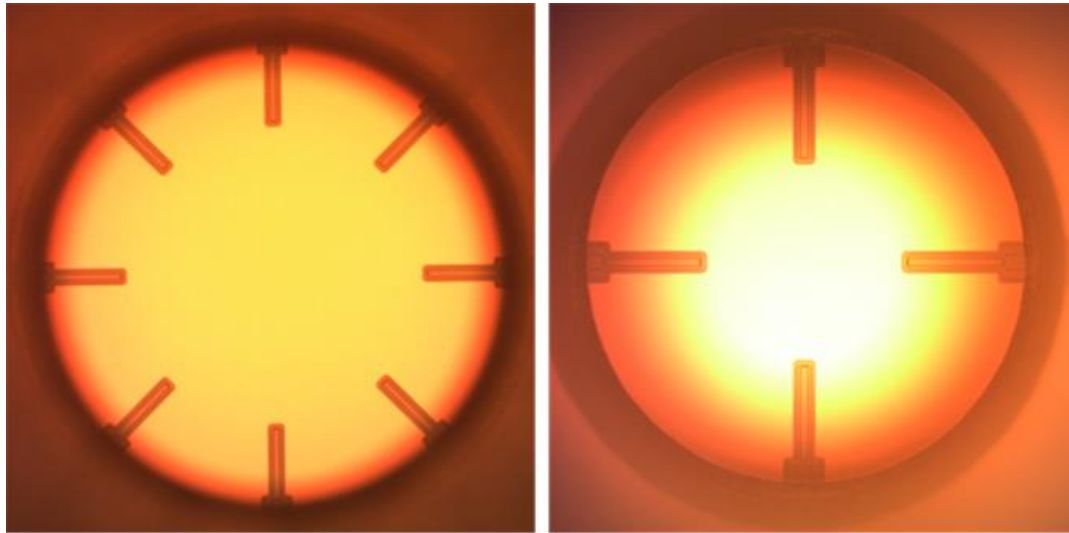


Figure 33. Completed waveguide microphone devices.

The singulation of fabricated die for this work was challenging due to the delicate nature of the thin film membrane. It was feared that dicing with a conventional die saw may be too brutal and could fracture the device. A new dicing strategy was investigated to use a small width trench feature in the Bosch mask that would limit the etch depth in the narrow trench

to less than half the wafer thickness. It was found in previous cleave experiment with a Bosch trench feature that was 20 μm wide there was a risk of fracturing the wafer while on the Bosch etch tools chuck. With that in mind, a mask was created that had a 10 μm wide trench stress concentrators so as to be able to cleave the wafers from the backside and have the cleave travel through the end of the waveguides creating a clean cleaved end facet to launch the laser light into the waveguide. It was found that the cleave was somewhat successful but in many cases left behind an uneven edge at the surface of the wafer and inconsistent cleaves where some of the cleaves would veer off the intended path and find the membrane access hole rather than traveling straight. Another mask was designed that had 15 μm Bosch etch trenches. The 15 μm trenches cleaved in a more consistent way but still did not provide a clean straight cleave and tended to cleave at an angle rather than following a perpendicular cleave to the surface from the trench. Images of both results can be seen in Figure 34 & Figure 35.

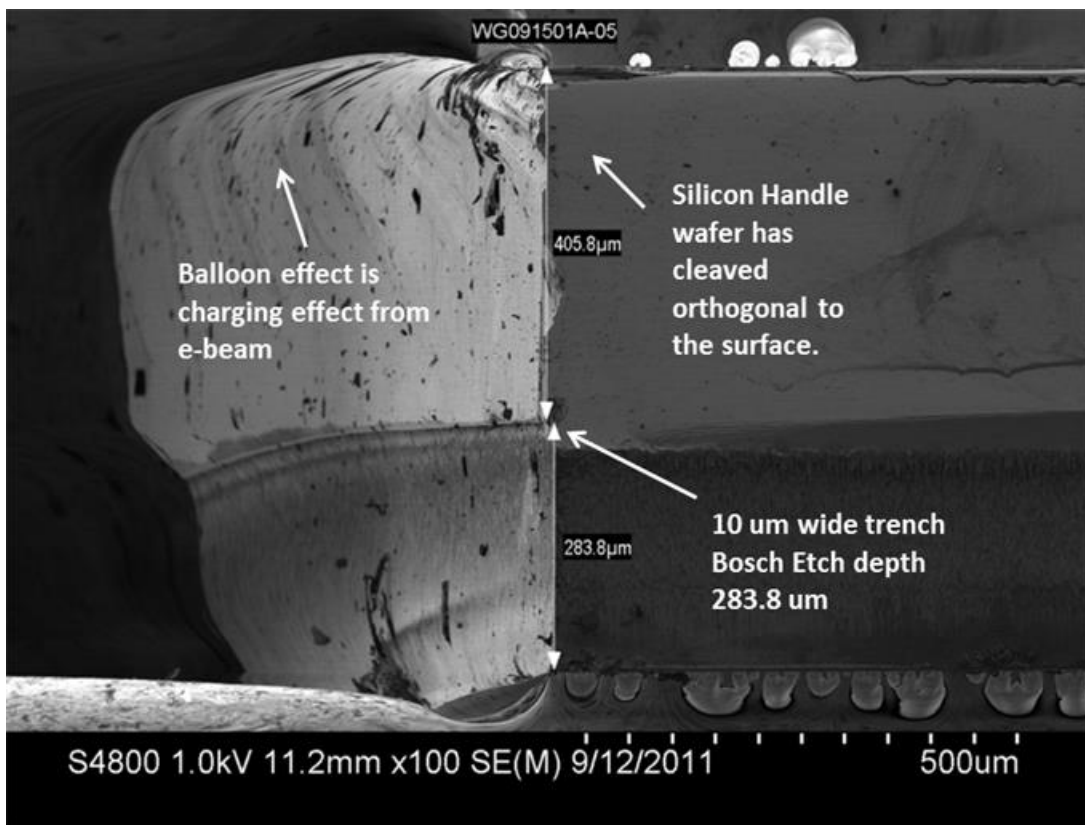


Figure 34. Bosch trench is 283.8 μm deep and 10 μm wide. Cleave was inconsistent and traveled into the die features.

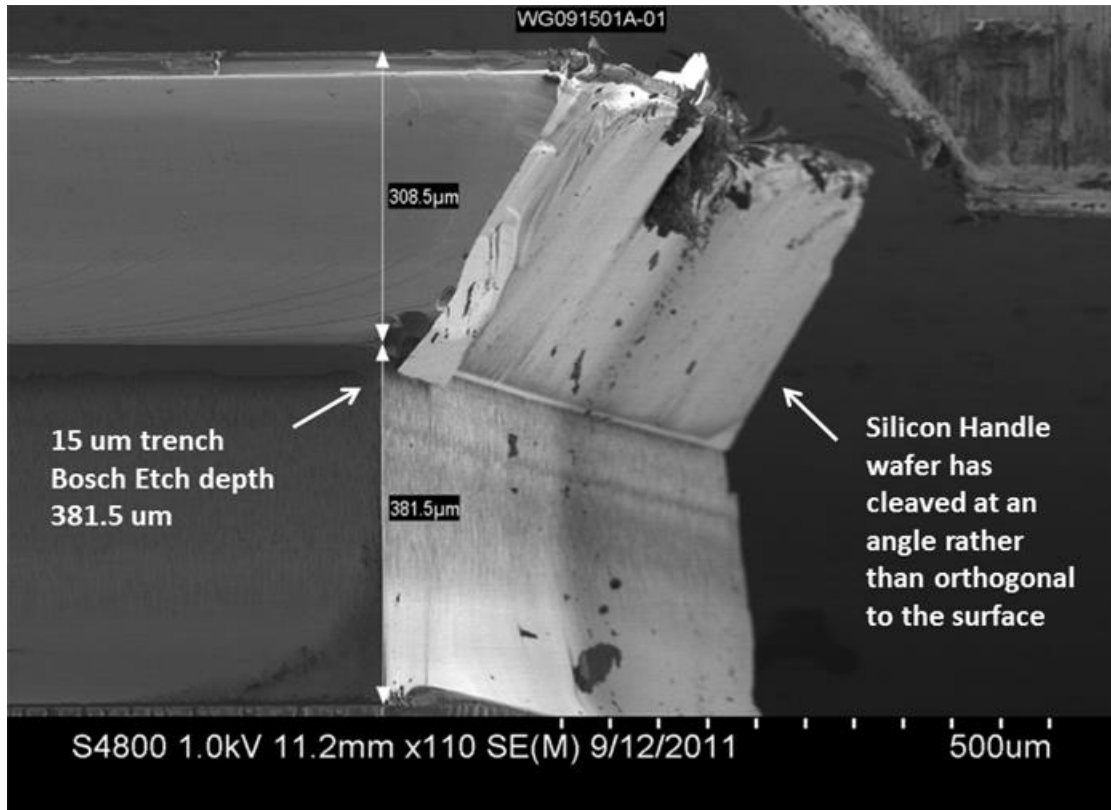


Figure 35. Bosch trench is 381.5 μm deep and 15 μm wide. Cleave was more consistent and singulated die fairly well but cleaved at an angle and provided a rough facet.

It was determined that polishing of the facets would be necessary in order to test the devices with reasonable coupling to the waveguide. The Bosch trench cleaving strategy did not appear to be successful in this case. However, the 15 μm trenches did provide a good way of singulating die, and can be employed in other projects where a clean facet of a waveguide is not needed.

Testing of these polished die proved to be unsuccessful due to what is thought to be a lack of compliance in the membrane to produce a significant signal. While the tensile Polysilicon may have been advantageous in the film strategy to create taunt membranes that survive the Bosch etch, the drum head like film did not deflect enough to produce the desired effect in the waveguides. The singulation process was re-engineered to use a Bosch etch process that will be covered in the Gen2 section.

4.6.3. Gen2: Device and Fabrication Design, RS981

The Gen2 design incorporated three process splits outlined in Figure 36.

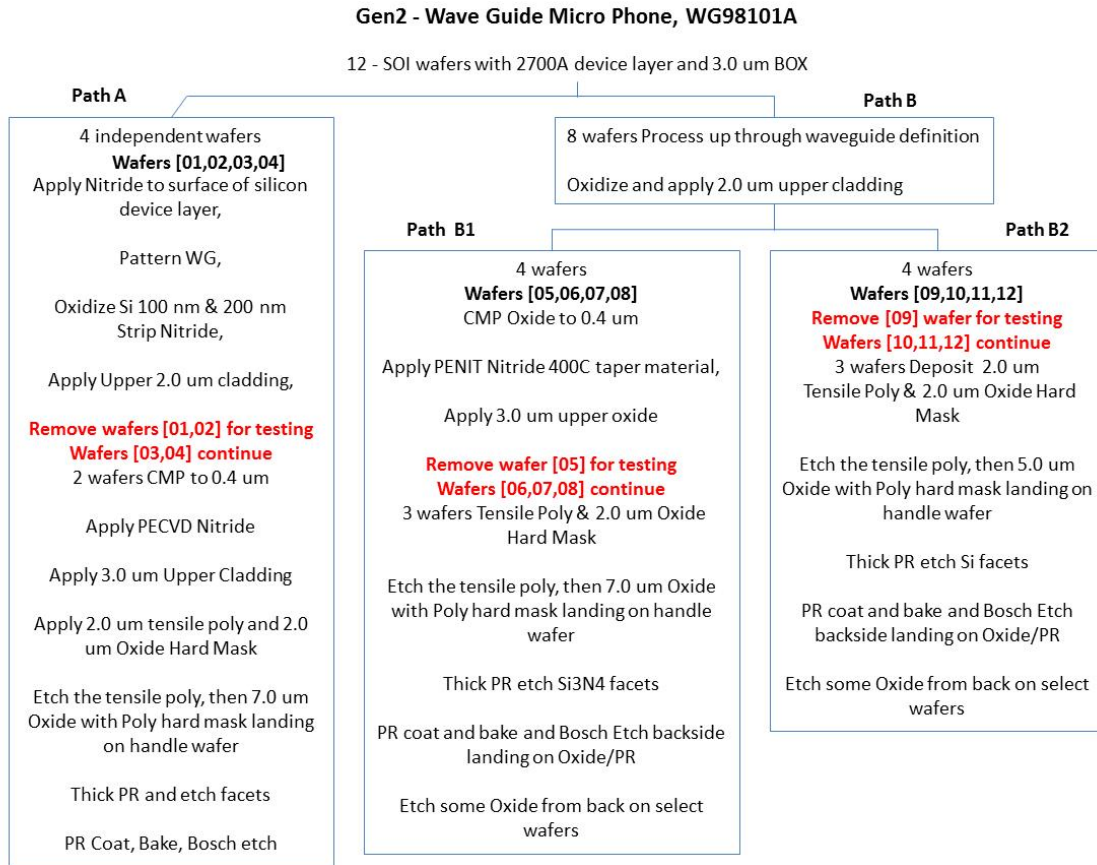


Figure 36. Flow diagram for Gen2 (RS981) Devices.

Split B1 was a basic version of what was done in the full membrane GEN1 design and process that incorporated a tensile Polysilicon layer on the top surface for structural reinforcement to the membrane and a typical waveguide with a taper facet interface for mode matching to the input fiber. Split B2 similarly used a tensile Polysilicon on the upper surface to reinforce the membrane but also included a Silicon Nitride taper at the input facet to reduce insertion loss by better matching the fiber mode and then transitioning the signal to the silicon waveguide using a taper on both materials. This worked out well for reducing insertion losses but required a thick lower cladding (3.5 um) to minimize any losses to the silicon substrate. Split A incorporated an oxidation step to the silicon waveguide using a silicon nitride hard-mask on the silicon surface while allowing the sidewalls to be oxidized and also included the Nitride taper. The oxidation strategy in split A was expected to smooth the sidewall roughness and was based on work detailed in [39]. This strategy was successful in carrying out the oxidation of the sidewalls but loss results were confusing and did not yield lower overall losses than the other types of guides. Images of the oxidation result can be seen in Figure 37. As can be seen the waveguide sidewalls have been oxidized and the Oxide edges look very smooth indicating the silicon sidewalls would also be very smooth.

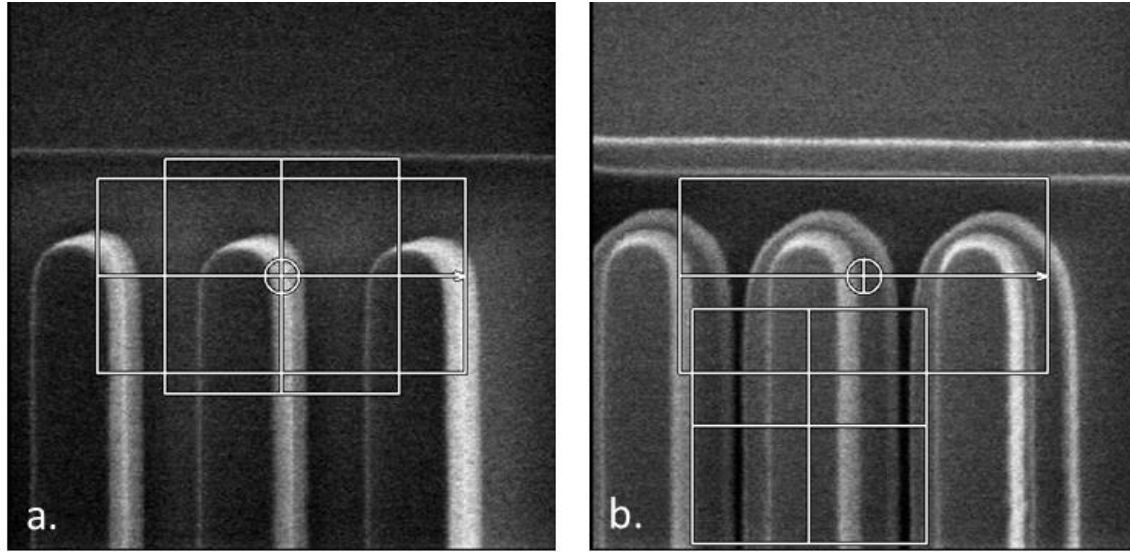


Figure 37. a.) Silicon nitride cap layer on silicon device etched (pre-oxidation). b.) Oxidized sidewalls of silicon waveguides with a silicon nitride cap layer.

4.6.4. Singulation Using Bosch Etch

A novel singulation strategy was developed for this project that simplifies the process and can be seen in Figure 38.

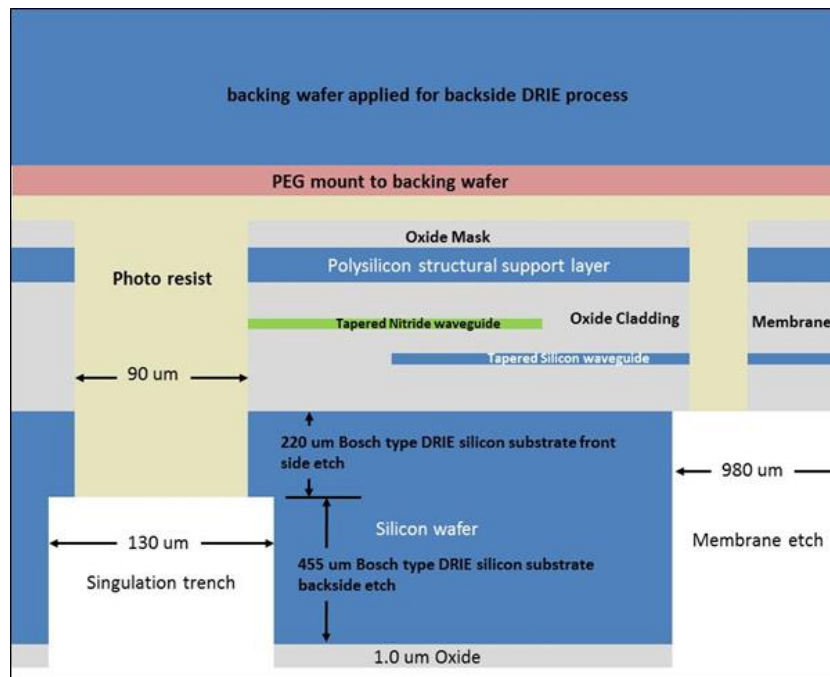


Figure 38. Cross section view of the nominal film structure and Bosch singulation. This singulation process provides a clean facet for lensed fiber coupling to the waveguides as well as a simple way to dice the parts without using a saw.

In this process strategy, a facet is etched through the film stack including the end of the coupling Nitride waveguide. Etching the nitride waveguide facet in this way allows signal

coupling to the singulated die without having to polish a facet on the edge of the die. The Nitride waveguide couples the signal into the silicon waveguide by the use of taped sections on both the silicon nitride and silicon waveguides. The facet etch is continued into the silicon substrate to a nominal depth of 220 μm . This allows the narrow Bosch trench to land at a similar time as the much larger membrane holes. The backside etch is accomplished by first coating the front side structures with photo resist. The Photo resist provides a landing film for the Bosch etch as well as mechanical support for the membranes. The wafer is then mounted on a backing wafer with Polyethylene Glycol (PEG) for further mechanical support. Once the backside Bosch etch is complete, the PEG is soaked off with warm water followed by a solvent photo resist strip to release the parts.

Gen2 devices were able to produce the intended signal, but the signal was very weak compared to expectations. The cantilevered fins that were designed to allow a higher degree of flexibility also were significantly bowed out of plane leaving a large opening at the surface of the chamber opening. This was due to a residual stress in the film stack acting on the cantilevered features of the membrane. Examples of the out of plane deflections can be seen in Figure 39.

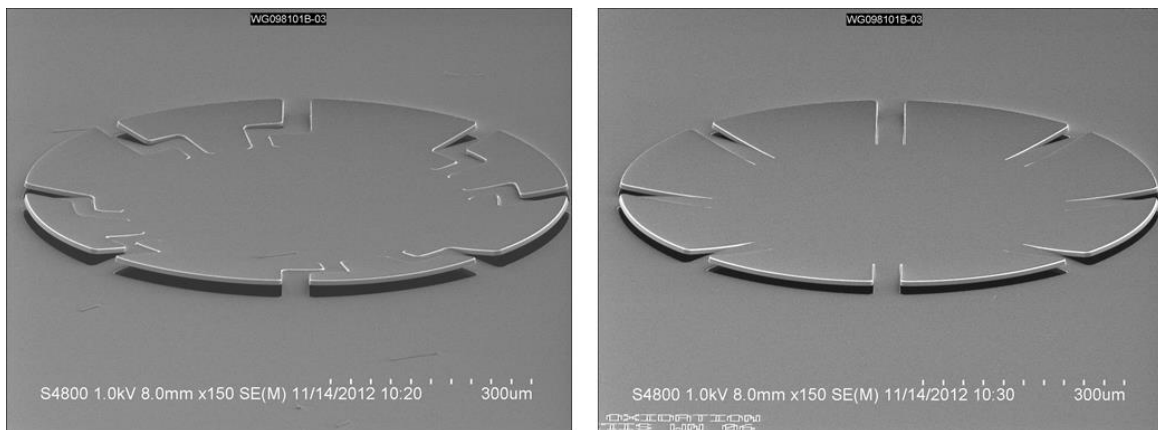
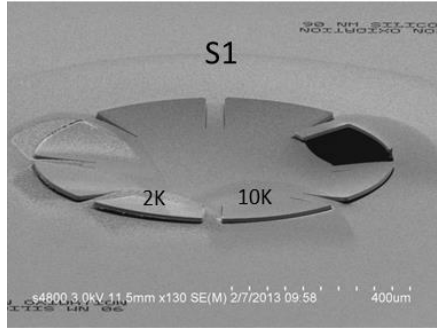
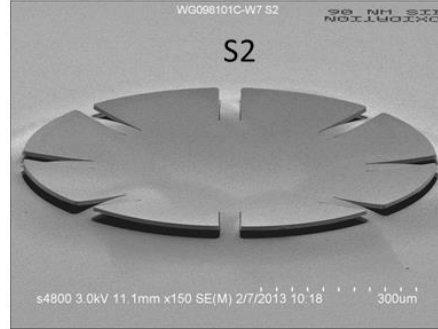


Figure 39. Completed Gen2 (RS981) microphone devices. Notice the out of plane deflections in the cantilevered fins.

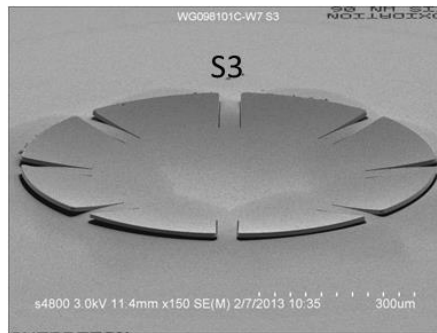
The out of plane deflections seen in Gen2 devices were determined to be a major factor in the low response signal obtained in the testing, where the gaps allowed too much signal to pass through the membrane rather than causing the membrane to deflect. In order to reduce the deflection in the membranes an implant experiment was conducted where the completed devices were subjected to Ion implantation on the front surface to impart compressive stresses in the top of the membrane and reduce the amount of bow in the as-fabricated devices. The best result is displayed in the Figure 40 below where the gap was reduced from 13.89 μm to 5.97 μm with the use of 1 MeV Si to 100 counts. Even with this reduction in gap, the signal was little improved.



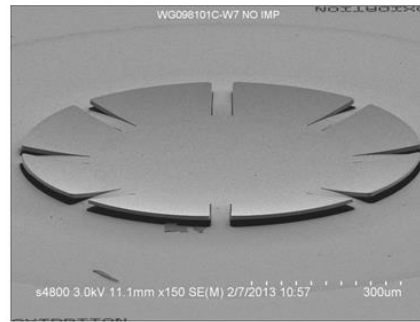
1 MeV Si to various counts



1 MeV Si to 1000 counts



1 MeV Si to 100 counts



No Implant

Figure 40. Ion Implant results on Gen2 devices for reducing the fin gap on completed structures.

4.6.5. Gen3: Device and Fabrication Design, RS1054

Several film stacks were investigated on Gen3 that were designed to move the waveguide further from the center of the film stack, the neutral plane, and to better manage the residual stress and stress gradient. Finite element analysis modeling was carried out to insure the new film stacks would not deflect too much in either direction out of the surface plane. “Mock SOI wafers were also created to simulate the SIO wafers in the process development so as to not consume them more costly SOI wafers material. The MOCK SOI wafers were inserted into each process split to provide a witness wafer of the process that can be cross sectioned as well as provide a expendable wafer that can be broken for cross section SEM analysis during the fabrication. A flow diagram for the process steps can be viewed in Figure 41.

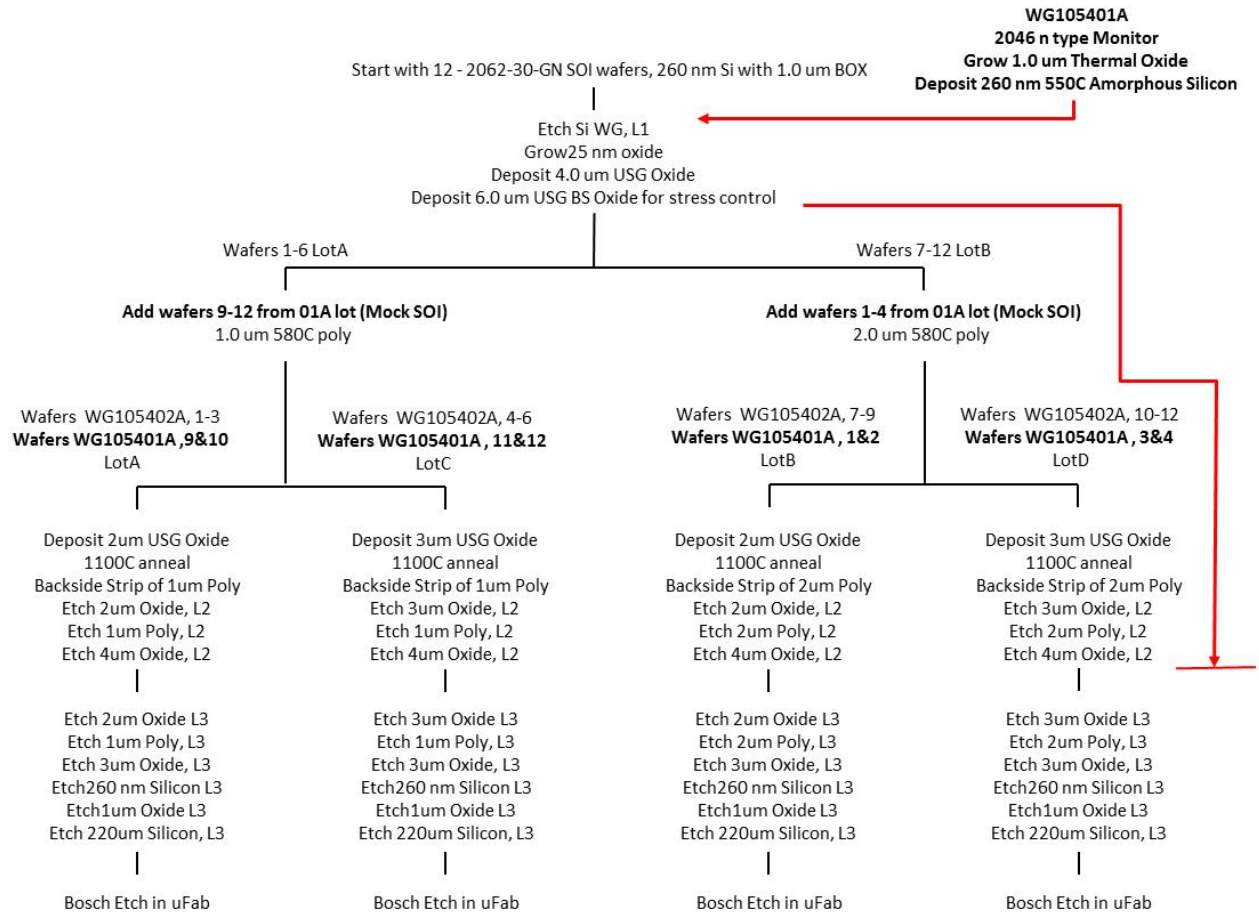


Figure 41. Process flow splits for Gen3 fabrication (RS1054).

The modeling results are covered in a separate section of this report. The design and a cross section view of one of the film stacks can be seen in Figure 42.

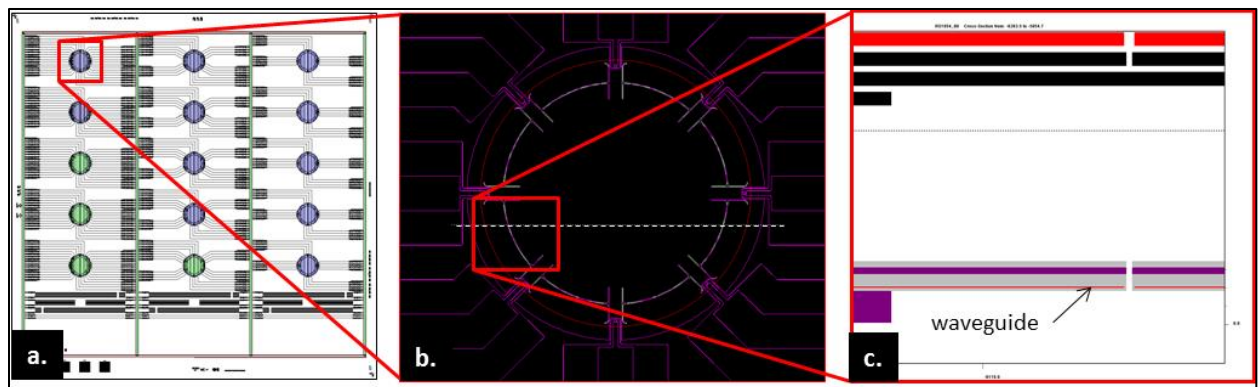


Figure 42. a. Layout of Gen 3 reticle set RS1054. b. Enlargement of one device on RS1054. c. 2D cross section of the device shown in b. showing the waveguide is at the lower side of the film stack far away from the neutral axis.

The etch requirements for this type of fabrication were extreme and the Mock SOI wafers were instrumental in developing an etch that could complete the thickest case of 2.0 um

trench opening and a nominal 13.26 micron thick film stack. An image of the completed etch profile can be seen in Figure 43. Notice the widening of the trench at the top surface and the landed etch on the silicon substrate.

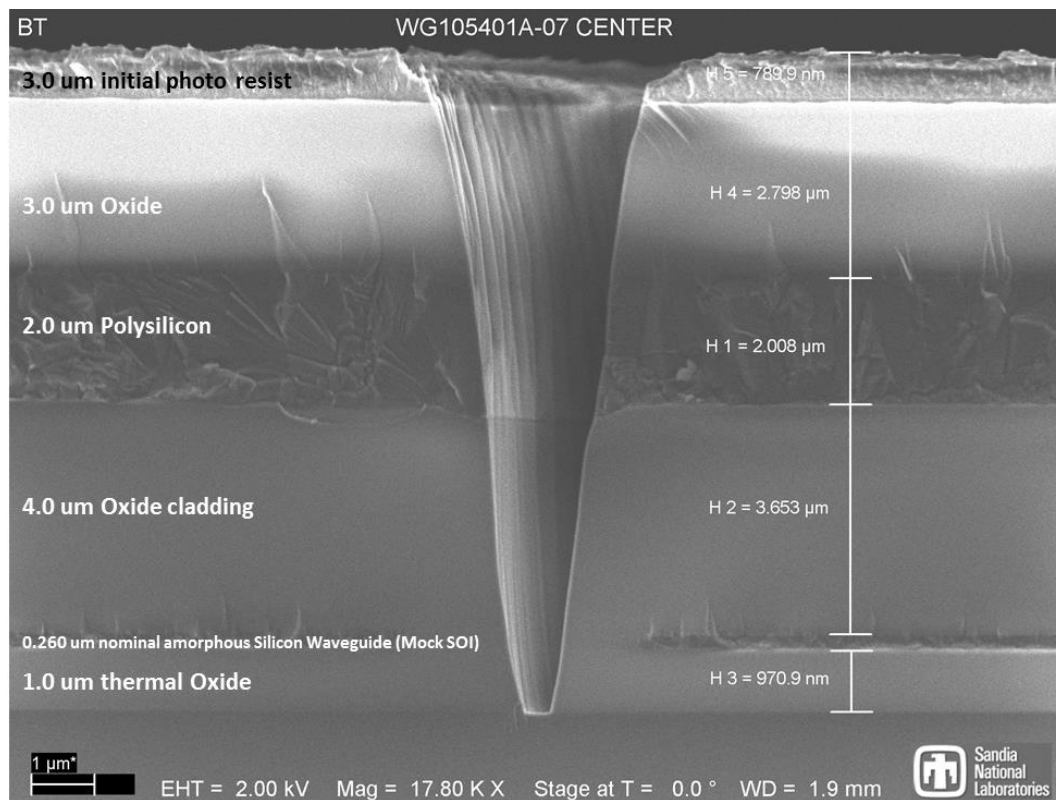


Figure 43. Etch profile of the Mock SOI etch for the thickest film stack case in the process splits for Gen3, RS1054.

In the Gen3 (RS1054) design the Bosch holes were kept equal in diameter (980 um) to allow the Bosch etch to be optimized. Ideally one would like all features to finish etching at the same time to reduce any isotropic etching at the bottom of the Bosch hole sometimes referred to as “notching”. In the Gen 2 devices we created a process that would land the singulation trenches etch at the same time as the larger membrane etches, and in the Gen3 devices the same strategy was employed with similar successful results.

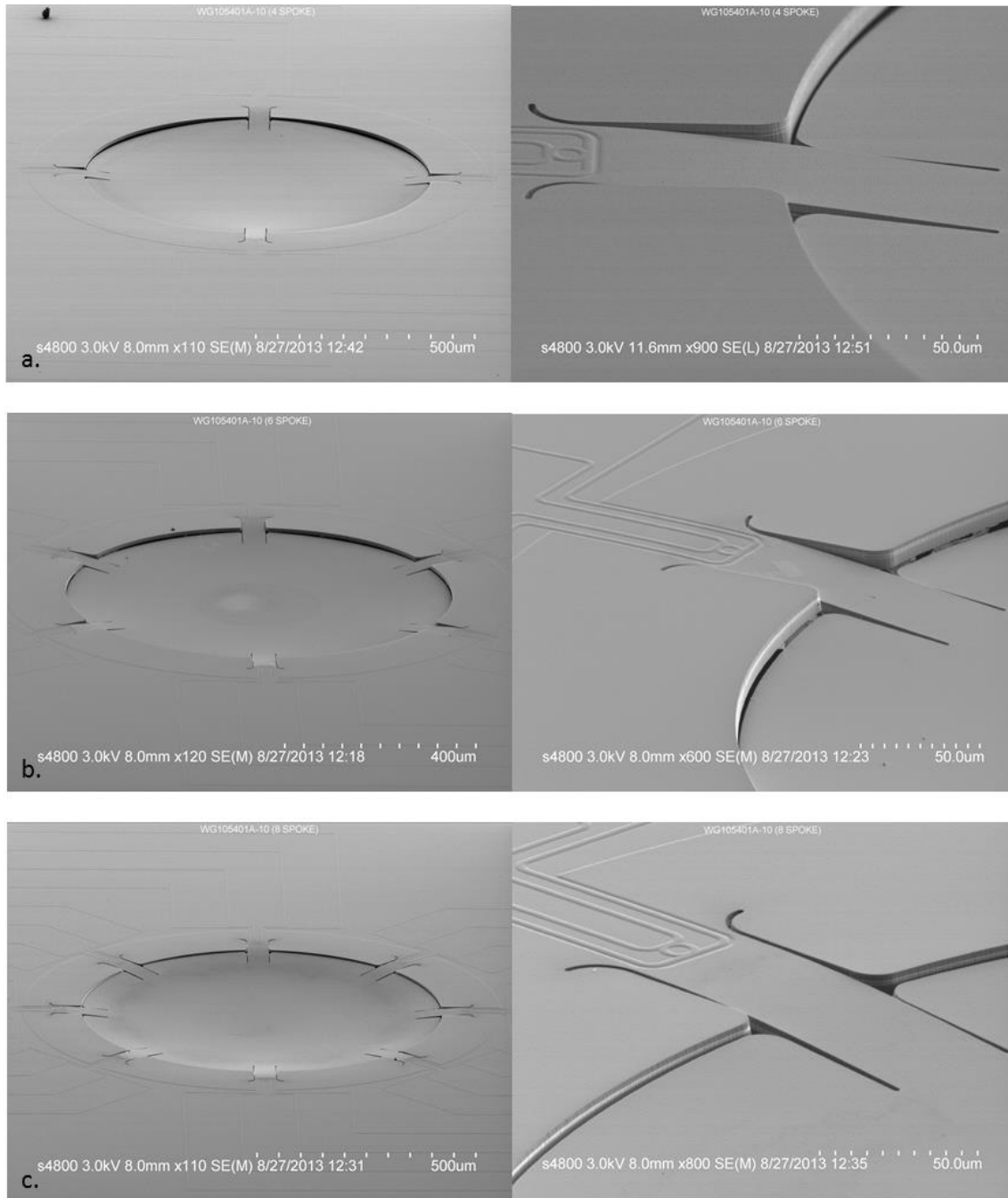


Figure 44. Completed Gen3 devices from the A split. a.) four spoke, b.) six spoke membrane, c.) eight spoke membrane.

Figure 44 shows images of completed Gen3 devices where the membranes now recessed into the Bosch etch opening as desired and as opposed to the Gen2 devices where the fins were deflected out of plane.

4.7. Optical Characterization of Devices

In order to select the best ring resonators for acoustic testing, the ring resonators first needed to be optically characterized. The following sections describe the experimental set up and procedures used in optically characterizing the ring resonator devices that are integrated into the tethers of the microphones. This is followed by set up and procedures used in acoustically testing the microphones.

4.7.1. Description of The Optical Testing Apparatus

A simplified schematic of the optical testing apparatus is shown in Figure 45.

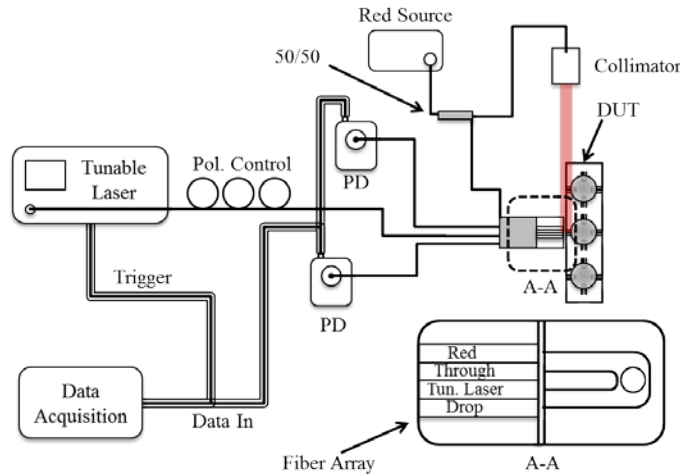


Figure 45. Optical Testing Apparatus.

The optical testing apparatus consists of a tunable laser whose output is connected to a polarization controller which is then connected to one port of a v-groove array of fibers. The v-groove array consists of four, small core single mode fibers that are bonded in v-grooves spaced 127 microns apart. These were purchased from Oz Optics, Ltd. (Ontario, Canada). Section A-A of Figure 45 shows how the ports of the v-groove array map to the waveguides of the ring resonators during testing. A red laser was split using a 1x2 50/50 fused fiber optic coupler. One beam was sent through a port of the v-groove array to aid in aligning the v-groove array to the Device Under Test (DUT). The other beam was sent to a fiber collimator and is imaged onto the v-groove array and the DUT that also aids in alignment process. Two ports of the array were aligned to the Through and Drop waveguides of the DUT. The Through and Drop signal were sent to photodetectors as shown in Figure 45. The voltage signal from the photodetectors was fed into a PC-Oscilloscope that was used for data acquisition. Data acquisition was initiated from the trigger output from the tunable laser that was also fed into the PC-Oscilloscope. The v-groove array and the DUT were placed on holders that were mounted to alignment stages. The stages had multiple degrees of freedom to align the DUT to the V-groove array.

4.7.2. Alignment procedure

The DUT and the v-groove array were mounted to stages that allowed the DUT and V-groove array to be aligned to each other. A photograph of the stages with the axes labeled is shown in Figure 46.

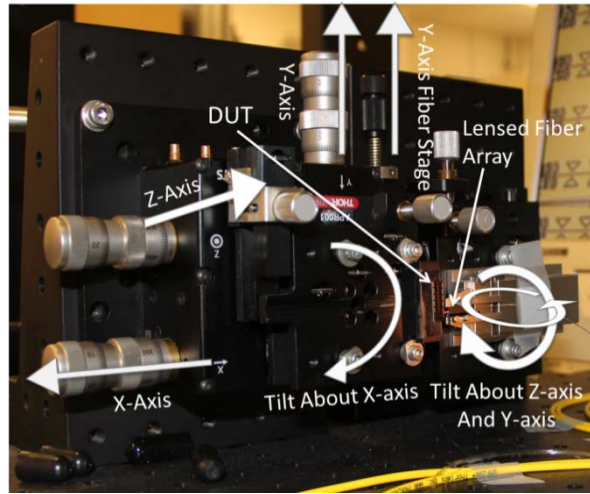


Figure 46. Device Alignment Platform.

The stage on which the DUT was mounted could move in X,Y,Z coordinates and was also able to tilt about the X-Axis. The stage that the v-groove fiber array was mounted to had course movement in Y direction, allowing the array to be moved to any device along the DUT. Additionally, the stage that the v-groove array was mounted to could tilt about the Y-axis and the Z-axis. The stages were used in combination with the collimated red laser to align the plane of the DUT parallel to the v-groove fiber array. The collimated beam was shined onto both the v-groove array and the DUT, and the reflected rays were imaged onto a card far from the stages. The two reflected rays could be aligned by moving the tilt about the X-axis on the stage that the DUT was mounted to, and the tilt about the Y-axis of the stage the V-groove array was mounted to. When the reflected rays were aligned parallel to each other and had the same spacing between each other when imaged close to and far from the setup, the planes of the DUT and the v-groove fiber array were parallel. Further rough alignment was then performed using the red laser beam that was sent through the v-groove array. A microscope and camera were used to view the v-groove array and the DUT. The Z-axis of the stage that the DUT was mounted to could then be used to move the DUT until the red laser beam hit the edge of the device. The course Y-axis on the v-groove array stage could then be adjusted to move the v-groove array to the ring resonator device to be tested. The tilt about the Z-axis could be adjusted such that the edge of the DUT was parallel to the edge of the V-groove array. A photograph of a device aligned to a v-groove array is shown in Figure 47.

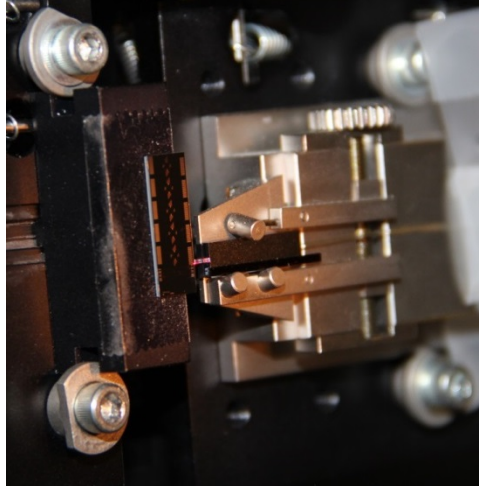


Figure 47. A Device Aligned to a Fiber Array.

Finally, the fine alignment was performed by setting the tunable laser source to 1550 nm and adjusting the X,Y,Z axes and the polarization control until the power measured from the Through port of the ring resonator was maximized. The next section details the tests performed on the ring resonator to optically characterize the different types of ring resonators used in this design.

4.7.3. Optical Tests

To find the best devices for subsequent acoustical testing, static optical characterization of the ring resonator devices was performed. Optical tests were also performed to determine the most sensitive wavelength (mid-point on the side of a resonance) that the tunable laser could be locked into during acoustic testing. The tunable laser was swept through a range of wavelengths while voltage was read from the photodetectors to obtain a graph of the response of the Through and Drop ports of the ring resonator vs. laser frequency. An example of the data taken is shown in Figure 48.

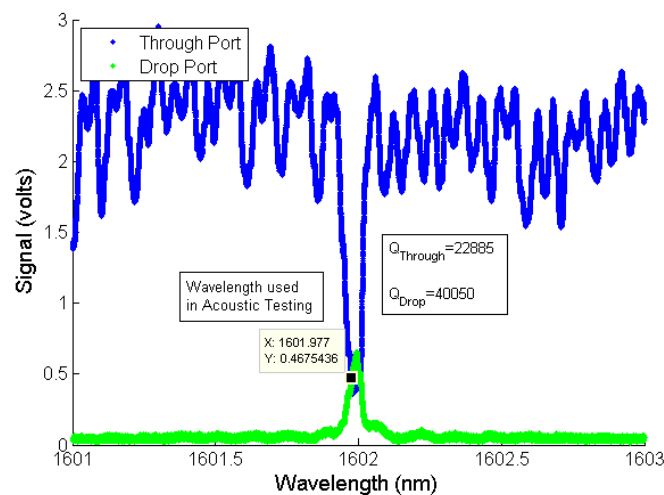


Figure 48. Data From a Ring Resonator.

The characterization parameter measured from the data shown in Figure 48 is the optical quality factor, Q , of the resonance. The quality factor represents the ratio of power stored in the ring resonator to the power lost per optical cycle. In general, a higher resonator quality factor leads to a more sensitive device. Q is defined in the following equation as the operation wavelength, λ_o , measured at the peak (or trough) of the Drop (or Through) port resonance, divided by the Full Width at Half Maximum of the resonance (FWHM).

$$Q = \frac{\lambda_o}{FWHM}$$

The quality factors of a device that was later acoustically tested are labeled in Figure 48. The results of testing the different ring resonator geometries used in this design are shown in this sections Results section below. The ring resonator geometries that yielded the highest Q were then selected for use in the acoustic tests.

The graph in Figure 48 was also used to determine the tunable laser's wavelength during the acoustic characterization experiments. The wavelength at which the resonance of the Drop and the Through ports intersect was chosen for the acoustic experiments so the response of both the Drop and Through ports could be measured at the same time. Figure 49 shows another pair of data traces for the Drop (red) and Through (green) ports of an optical ring with good performance.

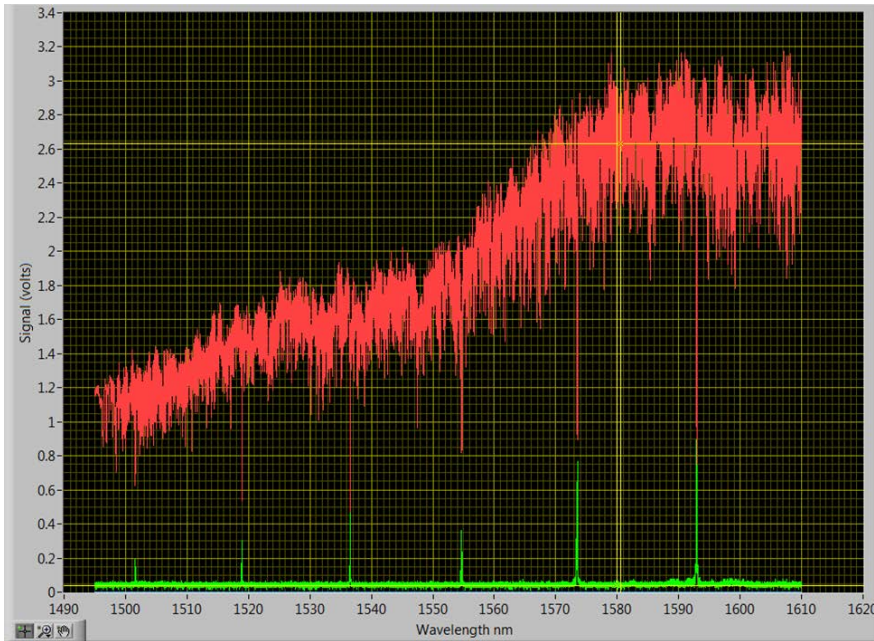


Figure 49. Light from a scanning diode laser collected from the Drop (green) and Through (red) ports of an optical ring resonator microphone. The signal has been converted to volts from a decibel scale. The green peaks correspond exactly with the dips in the red spectrum.

4.7.4. Optical Characterization Results

Despite theory and modeling, it is impossible to fabricate a perfect ring resonator microphone. Dimensional fabrication imperfections, side-wall roughness, and higher order

effects not captured by the models make success a challenge. To optimize the odds of achieving high quality devices, a range of design parameters were varied about their theoretical optimum. Each microphones had 4, 6, or 8 tethers connecting the microphone's membrane to the substrate. For each tether over many microphones, key design parameters were varied, including width of the waveguide ring resonator and the gap spacing between the ring and the bus waveguide. The width of the ring resonator varied from 350 nm to 550 nm. The gap spacing between the ring and the bus waveguide varied from 250 nm to 400 nm. In all, there were 26 different ring resonator designs that were optically characterized. The Q of each device where a resonance was found was measured. Devices with resonator rings of 350 showed zero resonances. Additionally, ring resonators with gaps greater than 350 nm showed zero resonances. The data for ring resonators that produced resonances is shown in Figure 50. It can be seen that the resonator with the highest quality factor had a ring width of 500 nm and a gap of 300 nm between the ring and the bus waveguide. Acoustic testing of this device is presented below.

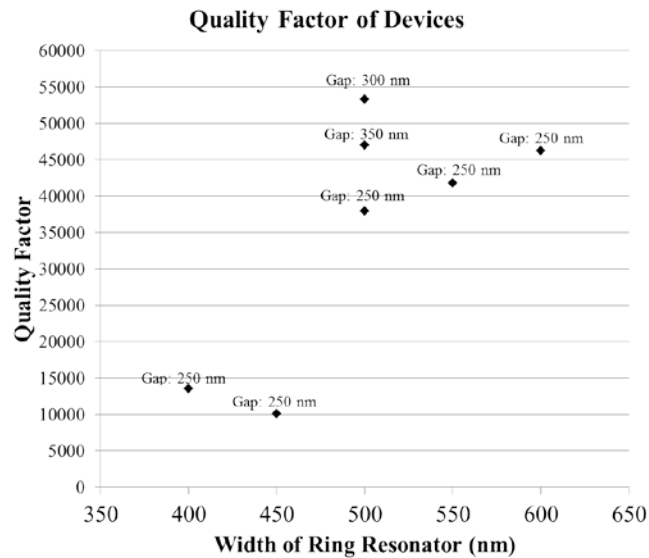


Figure 50. Q of Ring Resonators vs. Geometric Parameters of the Resonator Designs.

4.8. Acoustic Characterization of the Devices

After selecting a ring resonators device from optical performance, the microphone was characterized acoustically.

4.8.1. Description of Acoustic Testing Apparatus

A schematic of the acoustic testing apparatus is shown in Figure 51. The same optical setup described in the previous section was used to align and optically couple into the DUT. Figure 51 shows the additional equipment required for acoustic characterization of the PAS microphones.

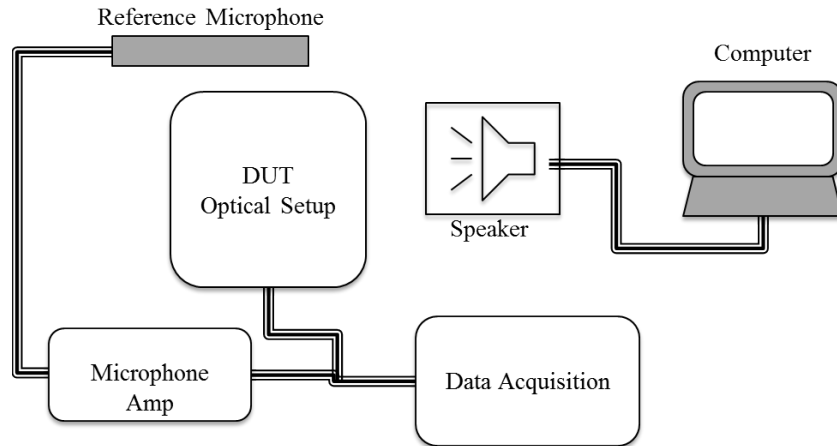


Figure 51. Acoustic Characterization Apparatus.

A reference microphone was mounted in close proximity to the DUT. The output of the reference microphone was routed to a microphone amplifier. The amplified signal was then sent to a PC-Oscilloscope for data acquisition. The outputs of the photodetectors which measure the response from the Drop and Through port of the DUT were also connected to the PC-Oscilloscope. A LabVIEW program was used to generate a sine wave with a specified frequency through the audio jack of the computer. The audio tone was played by a speaker placed in front of the DUT.

4.8.2. Acoustic Tests

After the DUT was aligned and optically characterized, the DUT was acoustically characterized. As mentioned in the section on optical testing, a laser wavelength sitting on the edge of resonances for both the Drop and Through output ports was used. The signal of the reference microphone and both the Drop and Through ports were recorded while the acoustic tone played at different volumes. The PC-Oscilloscope recorded the microphones' outputs vs. time as shown in Figure 52. The peak-to-peak voltage of the response of the reference microphone was used to estimate the pressure that both the reference microphone and the PAS microphone encountered, since the sensitivity of the reference microphone was known. As shown in the Results section, efforts were made to keep the pressure from the speaker roughly constant when testing the PAS microphone over a range of different audio frequencies. The PC-Oscilloscope software computed the Fast Fourier Transform (FFT) of the data to obtain plots of the microphone's response in dBu vs. frequency. A plot of the FFT of Figure 52 is shown in Figure 53.

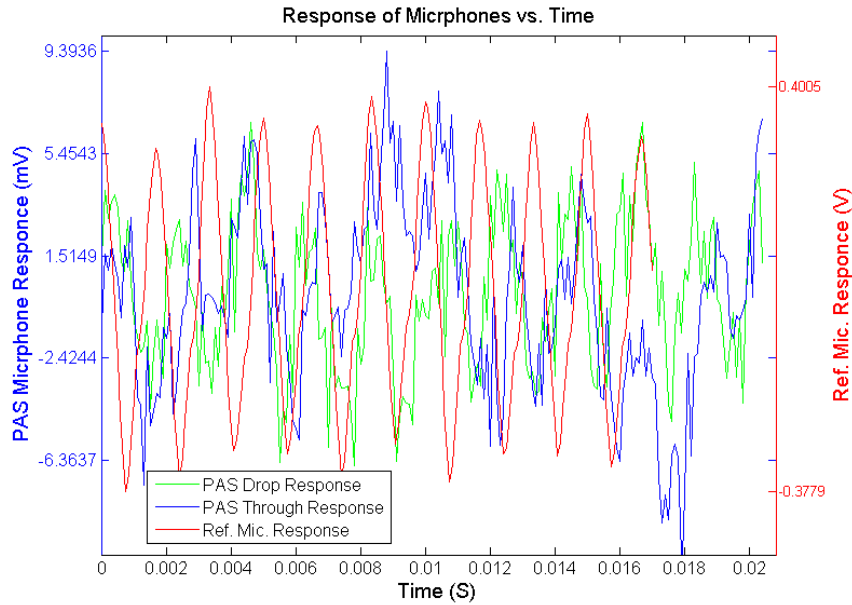


Figure 52. Response of Microphones vs. Time at 500 Hz Audio Excitation.

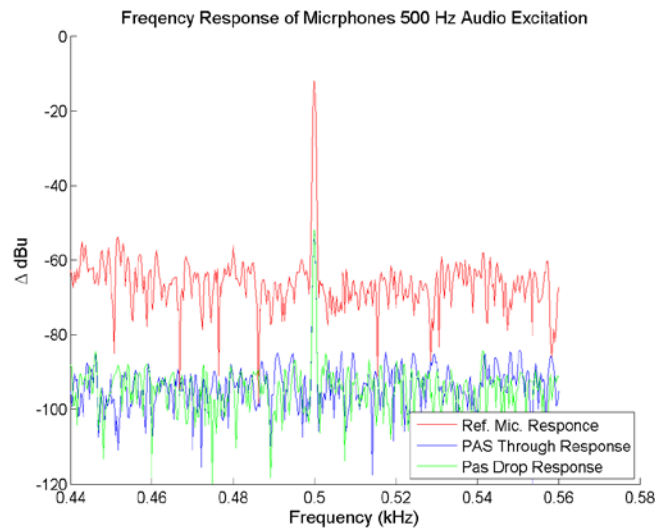


Figure 53. Response of Reference and PAS Mics vs. Frequency at 500 Hz Audio Excitation. The Drop port has a lower noise floor but the dB signal (log scale) show about equivalent response with the Through port.

As can be seen in Figure 53, both the output of the reference microphone and PAS microphone show a peak at the audio frequency, 500 Hz, which was played through the speaker. The response of the reference microphone in dBu was the difference between the peak and average of the surrounding baseline. Response of both the Through and Drop ports were recorded. The output frequency of the speaker was then changed and the test was repeated over a range of frequencies from 500 Hz up to 20 kHz. The results of this testing is shown in the following section.

4.8.3. Acoustic Characterization Results

As mentioned previously, 4, 6, and 8 tether microphones were designed. There were also four different film stacks used during fabrication of these designs. Unfortunately, due to manufacturing issues and time constraints, only one microphone from this final and most successful manufacturing split was tested acoustically. Many designs were tested acoustically with a laser vibrometer and were shown to work. SEM and interferometry data showed that the 6 tether microphone had the smallest air gap between the flaps of the membrane and the substrate edge. It was therefore assumed to have the widest bandwidth response of the three microphone types (for an identical membrane stack – i.e. membrane compliance). Thus, a 6 tether device with the desired ring width of 500 nm and a gap of 300 nm was selected for further acoustic testing. Note: other microphones from the first 2 generations were tested acoustically. The Generation 3 designs were so much better that the others are not discussed further.

The PAS microphone and a reference microphone were tested at audio frequencies varying from 500 Hz up to 20 kHz. The responses of both the reference microphone and the Drop and Through ports of the PAS microphone are shown in Figure 54.

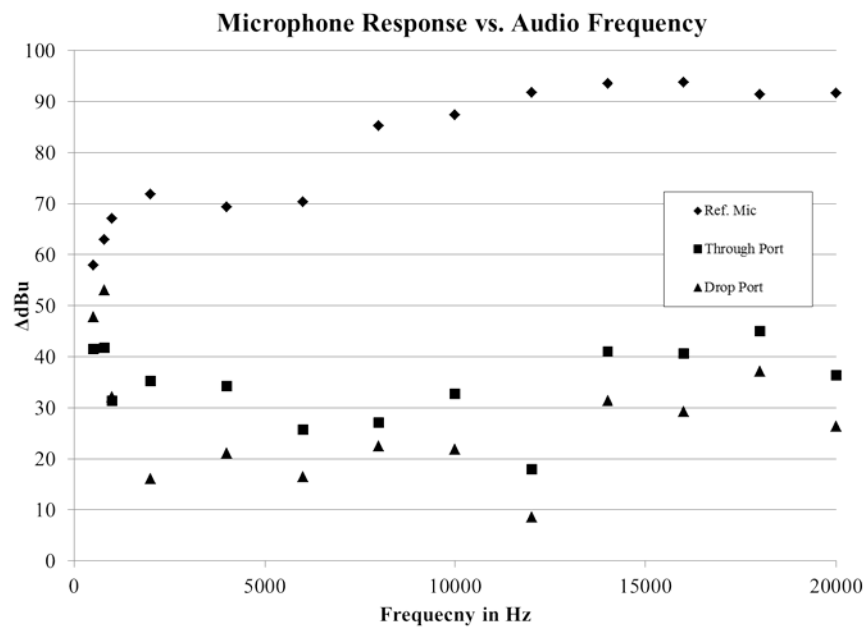


Figure 54. Microphone Response vs. Driven Audio Frequency

The response of the PAS microphones Through and Drop waveguides closely match that of the amplified reference microphone at low audio frequencies. While the response of the reference microphone rises around 5000 Hz and again at 10 kHz, the responses of the PAS microphone output stay fairly consistent throughout the bandwidth tested. It should again be noted that the output of the reference microphone shown in these graphs was amplified while the PAS microphone response was not.

As discussed earlier, the reference microphone was also used to estimate the pressure on the PAS microphone during testing. The volume of the speaker was adjusted during the tests

such that the pressure on the microphones was consistent from one audio frequency to the next. A graph of the pressure output from the speaker as measured from the reference microphone is shown in Figure 55. The pressure varied from 3.2 Pa to 1.8 Pa during these acoustic tests.

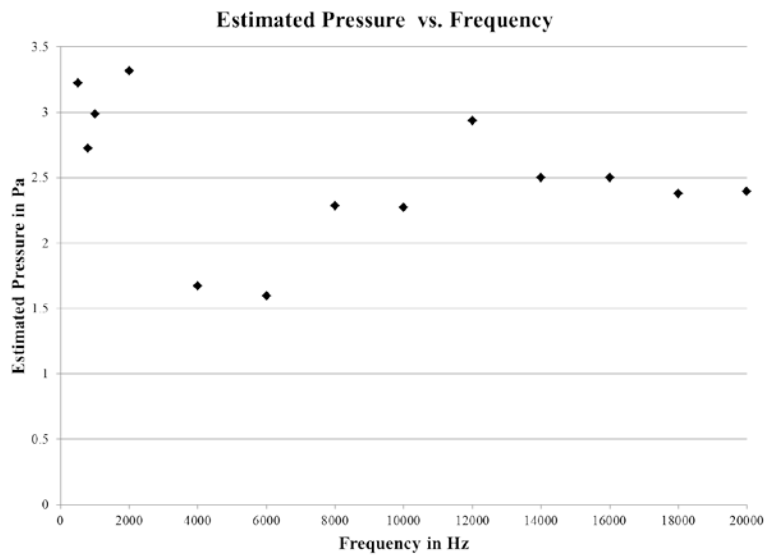


Figure 55. Pressure Output by Speaker vs. Frequency.

5. PHOTONIC CRYSTAL ACOUSTO-OPTIC MICROPHONE

A third, potentially much more sensitive optical microphone design was conceptually designed and modeled. It is based on a photonic crystal (PhC) lattice, where the entire dynamic range of signal occurs within a few tens of nanometers of separation. The design and supporting theoretical treatment are the subject of a Technical Advance [40]. The microphone has a theoretical sensitivity of 7.74×10^6 GHz/MPa. Realization of this concept would enable interesting tradeoffs to improve the microphones, including smaller cavities with thicker membranes for a greater linear dynamic range.

5.1. Acousto-Optic Microphones

In an effort to push the limit on microphone sensitivity, the acousto-optic microphone was developed that takes advantage of strong light-matter coupling in a system of coupled optical cavities [41]. In general, the sensitivity is the product of the pressure sensitivity of the membrane, $\frac{dx}{dp}$, where x is the displacement of the membrane and p is the pressure created

by the acoustic wave, and the sensitivity with which x can be detected. In the case of an acousto-optic microphone, the latter term is related to the change in the optical frequency of the system with x . Typically, such microphones have used a cantilever design consisting of an acoustic cavity containing a small beam fabricated such that it is suspended at only one end to create a pressure sensor. The deflection of this cantilever is interrogated by a laser beam in an interferometer configuration, enabling the acoustic signal to be measured from the detected optical power. This type of acousto-optic microphone has been demonstrated in a PAS system to provide a detection limit of 9.8×10^{10} molecules/cm³ at a laser power of 1.16W for acetylene [42]. In fact, acousto-optic microphones generally have better sensitivity than capacitive microphones fabricated using the same process, due to the limited movement of the membrane in a the capacitive microphone as compared to a cantilever and also the increased movement sensitivity of the optical detection scheme in the acousto-optical microphone. The primary drawbacks of the cantilever design, however, are directly related to the optical detection: careful laser alignment must be maintained, the cost of the system is typically much larger than that of a capacitive microphone, and the system is generally more complex. The factors translate into a photoacoustic spectrometer that has greater sensitivity, but is more delicate and less robust. However, these issues are offset by the use of resonant photonic structures and optomechanical coupling that allow for weaker light sources, which translates into greater versatility in the choice of light sources (i.e. broadband, rather than lasers) and thus the possibility of reduced cost and size with greater robustness of the overall PAS system.

5.2. Photonic Cavity Approach

To achieve optimal sensitivity and in a robust platform, our proposed acousto-optic microphone combines the best characteristics of capacitive and cantilever microphones. The optical detection of membrane motion is utilized for maximum sensitivity, while an integrated photonics platform is used instead of free-space cantilever interrogation, resulting

in a robust and alignment-free package. As shown in Figure 56, the proposed acousto-optic microphone is composed of a membrane suspended over an acoustic cavity, with a PhC cavity and input/output waveguides suspended above the membrane. The PhC cavity is separated from the membrane by a distance s , creating an optomechanical resonator due to coupling of the evanescent field of the optical mode to the membrane. To make fabrication of the device practical, the same PhC structure will be patterned into the lower membrane as well, resulting in an identical cavity. At small separations, the frequency of these two cavity modes will be strongly coupled, form a supermode and modulate the resonance frequency of the modes. This modulation will change the intensity of the light propagating in the output waveguide, providing a detectable signal that is directly related to the excited acoustic signature of the sample. The sensitivity of this acousto-optic displacement detection is $\frac{d\omega}{ds}$, where ω is the optical angular frequency and s is the separation between the membrane and the PhC cavity, which ideally is equal to the membrane displacement, x .

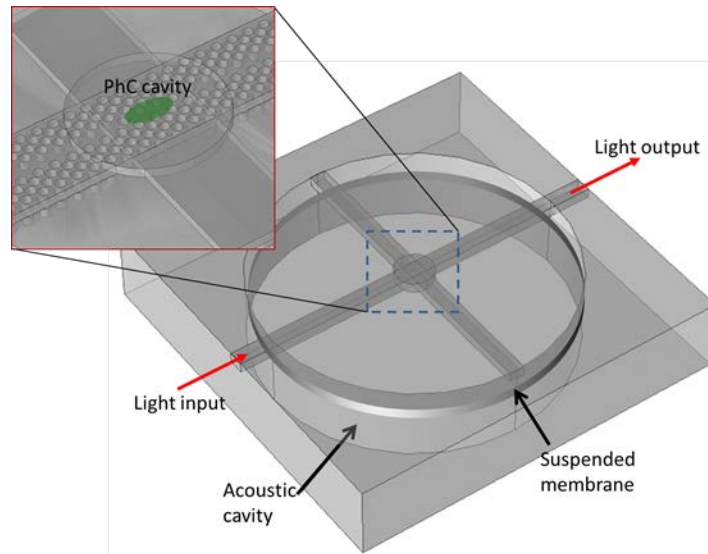


Figure 56. Schematic of the PhC acousto-optic microphone.

5.3. Discriminating Features and Benefits

There are numerous advantages to utilizing a PhC-based acousto-optic microphone for PAS. A summary of the discriminating features and the resulting benefits of this approach are listed in Table 4, and a diagram of our approach summarizing the key innovative features and enabling technologies is shown in Figure 57.

Table 4. Our PhC-based acousto-optic microphone approach offers unique opportunities to develop a definitive solution.

<i>Discriminating Feature</i>	<i>Benefits</i>
Photonic crystal technology	Enables engineering of photonic modes and the creating of slow-light waveguides and compact, high-Q cavities
2D planar topology	Easily integrated with microelectronics or other MEMS devices, large access area, and predictable performance
MEMS-compatible fabrication	High-quality, high-yield fabrication on 6 inch wafers and possible transition to mass production
Silicon-based material system	Structural integrity and corrosion resistance, leverages optomechanics and silicon photonics capabilities at Sandia, large Si electronics industry supports process technology and commercialization opportunities

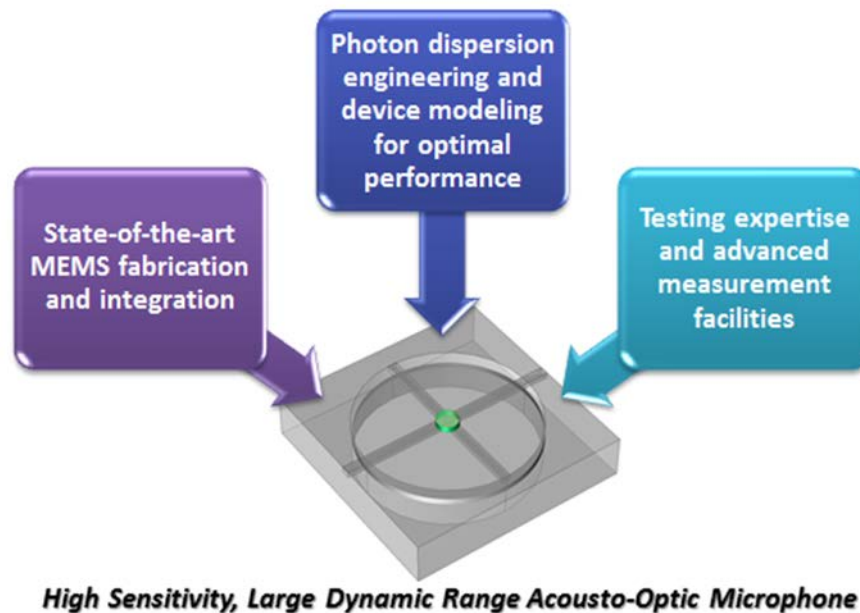


Figure 57. Diagram of our approach summarizing the key innovative features and enabling technologies.

PhCs consist of a periodic arrangement of dielectric materials with a large contrast in refractive index, which can exhibit a complicated band structure (dispersion) as compared to that of the bulk solid or even an unpatterned waveguide due to a combination of Bragg and Mie scattering. The result is the creation of a number of unusual phenomena absent from the bulk case, namely:

- **Photonic bandgaps:** a range of frequencies where light propagation in the PhC is forbidden. Waveguides and cavities can be formed by engineering defects in a PhC within the bandgap.
- **Flat “slow-light” bands:** low group velocity propagating modes that can be engineered by suitable waveguide design and that exhibit enhanced light-matter interactions such as optical nonlinearity.

- **Negative group velocity:** modes with negatively-sloped dispersion that can exhibit negative refraction effects and “superlensing”.

By carefully designing the PhC devices, the first two of these effects can be used to our advantage in the proposed system. Optimization of the PhC topology provides the widest bandgap, enabling wide-bandwidth operation of the PhC devices. The defect modes can be engineered to optimize the cavity topology for maximum confinement (i.e. largest quality factor, Q). Alternatively, using waveguide modes in the slow-light regime maximizes the light-matter interaction over a relatively broad range of frequencies as compared to a high- Q cavity.

5.4. Photonic crystal device designs

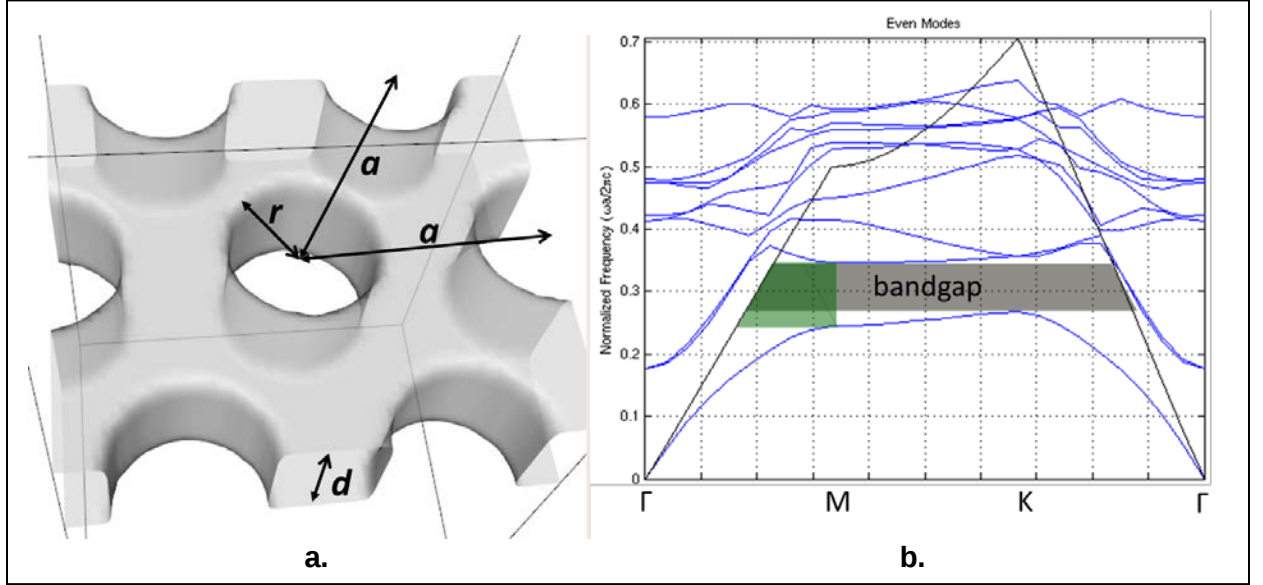


Figure 58. (a) Schematic of a 2D PhC structure. (b) Dispersion diagram for the bulk PhC; the light line is shown in black.

The design of our PhC implementation starts with a hexagonal lattice of air holes in Si, as shown in Figure 58(a), which provides the largest possible bandgap in a 2D slab topology with a geometry that is robust to fabrication defects. The lattice constant a is 460nm and the hole radius r is 138nm, chosen to provide a bandgap at a wavelength of around 1550nm, and the slab thickness d is 250nm, the typical device layer thickness for silicon-on-insulator (SOI) wafers. The dispersion diagram for this structure is shown in Figure 58(b), where a complete bandgap can be observed under the light line for normalized frequencies $0.268 < f_n < 0.345$; the $\Gamma \rightarrow M$ bandgap, which is the direction used for the waveguide and cavity designs, is slightly wider at $0.245 < f_n < 0.345$. All photonic dispersion calculations were performed using the freely available software package MIT Photonic Bands (MPB), a fully-vectorial eigenmode solver for Maxwell's equations with periodic boundary conditions using plane-wave basis functions [43].

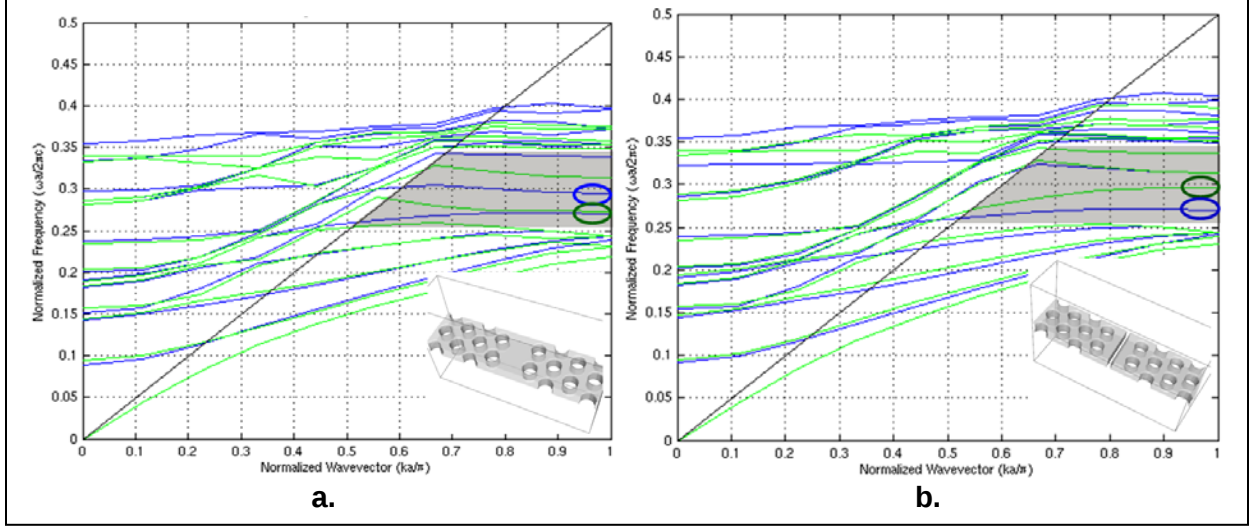


Figure 59. (a) Dispersion diagram for the W1 PhC waveguide; blue indicates even modes, green indicates odd modes, and the light line is shown in black. Inset: schematic of structure. (b) Dispersion diagram for the W1 slot PhC waveguide.

A W1 waveguide was created by removing a complete row of air holes as shown in the inset of Figure 59(a), along with a slot waveguide created by adding a continuous air slot with a width w of 80nm to the W1 design (inset of Figure 59(b)). Photonic bandgap mirrors consisting of 4 lattice periods are used above and below the guiding region. The modes even waveguide modes are indicated in blue, and the odd modes in green; circles highlight the guided modes that were chosen for analysis. The modes were examined at the M point, where their dispersion is the flattest, indicating the lowest group velocity.

5.5. Acousto-optic microphone sensitivity estimates

To calculate the expected sensitivity of the acousto-optic microphone and estimate the overall dynamic range and sensitivity of the microphone, the motional sensitivity of the optomechanical PhC device must be determined and combined with an estimation of the pressure sensitivity of the membrane itself from structural mechanics simulations. The sensitivity of the optomechanical PhC structure can be assessed by calculating the dispersion of a system of two stacked parallel waveguides, as shown in the insets of Figure 60, as the separation between them is changed. The resulting data are plotted for the two modes of both the W1 (Figure 60(a)) and slotted (Figure 60(b)) waveguides for separation distances s varying from 20 to 460nm. Note that only the even supermode is plotted here, since the odd supermode exhibits equivalent behavior, although mirrored about the frequency at $s \rightarrow \infty$. As expected, the mode frequency is modified exponentially with separation distance, since the evanescent electromagnetic field coupling the two membranes decreases exponentially with increasing distance from the surface of the membrane. Thus, the change in normalized frequency $\omega_{n,f}$ can be fit with a function of the form $\omega_{n,f}(s) = c_1 - c_2 e^{-c_3 s}$ where c_1 , c_2 , and c_3 are fitting parameters, which are shown for each of the four curves in Figure 60. The

motional sensitivity can then be determined by taking the derivative $\frac{d\omega_{n,f}}{ds}$ of these curves, which for the largest case (even mode of Figure 60(b)) gives $3.9 \cdot 10^{-4} e^{-0.01s}$. As an example, a

membrane separation of 100nm, the sensitivity would be $1.43 \cdot 10^{-4} \text{cm}^{-1} = 93.2 \text{GHz/nm}$, or in terms of $\frac{1}{\omega} \frac{d\omega}{ds}$, $0.485 \mu\text{m}^{-1}$. The slot waveguide generally outperforms the un-slotted W1 waveguide because of the greater field penetration into the air cladding above and below the PhC slab due to the introduction of the air slot, which strengthens the coupling between the two membranes.

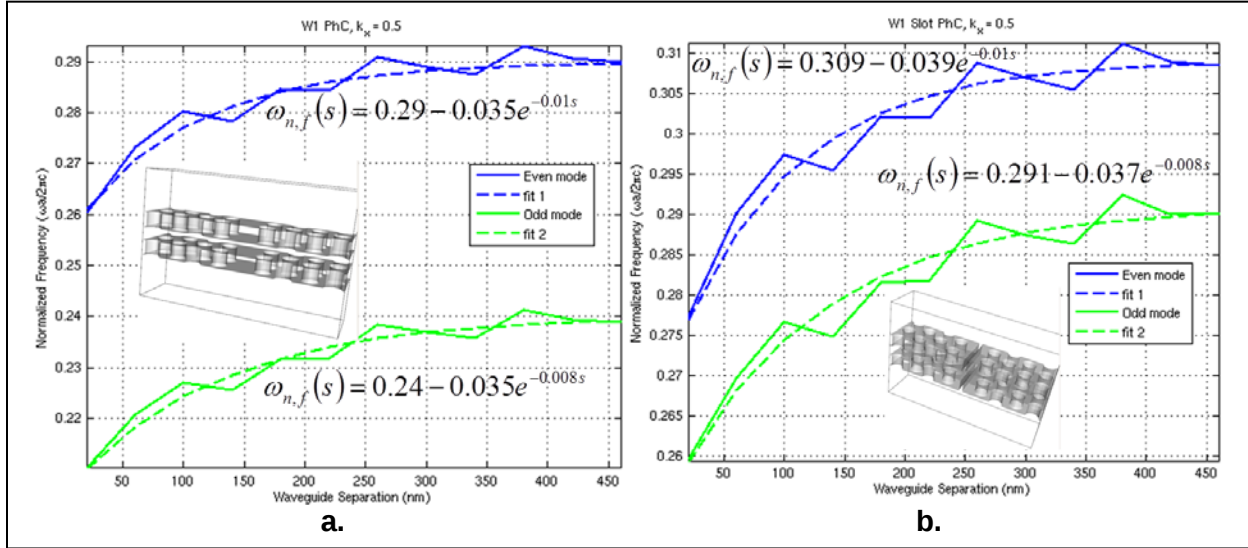


Figure 60. Wavelength versus membrane separation for the even and odd guided modes of (a) W1 and (b) slot PhC waveguides.

The pressure sensitivity of the membrane was calculated using analytical expressions for the flexural rigidity of a circular drum-like membrane and the resulting pressure deflection sensitivity at the center of the microphone where the detecting PhC device is located. The small holes drilled into the lower membrane are ignored, since they should be small enough that any airflow through them will be highly restricted and thus this pressure leakage path can be neglected. Also, any movement of the upper membrane is ignored since it is not directly exposed to the pressure gradient, it is assumed to be rigid enough that its movement will be negligible, and it has large open areas etched away that will prevent any pressure gradient from building up on either side of the membrane. The results of these calculations

are shown in Fig. 6(a); the pressure sensitivity $\frac{ds}{dp}$ has a functional form of $8.3 \cdot 10^4 \text{s}^{-3}$. As

an example, for a membrane separation of 100nm, the sensitivity would be $8.3 \cdot 10^4 \mu\text{m/MPa}$. By multiplying this with the motional sensitivity calculated in above, the overall sensitivity of the acousto-optic microphone can be determined, as shown in Fig. 6(b). Clearly, the smallest separation between membranes provides the greatest sensitivity, however practical limitations such as fabrication tolerances and the possibility of stiction failure at high membrane displacements constrain the minimum separation distance to approximately

100nm, thus limiting the maximum achievable sensitivity of the microphone $\frac{d\omega}{dp}$ to about $7.74 \cdot 10^6 \text{GHz/MPa}$.

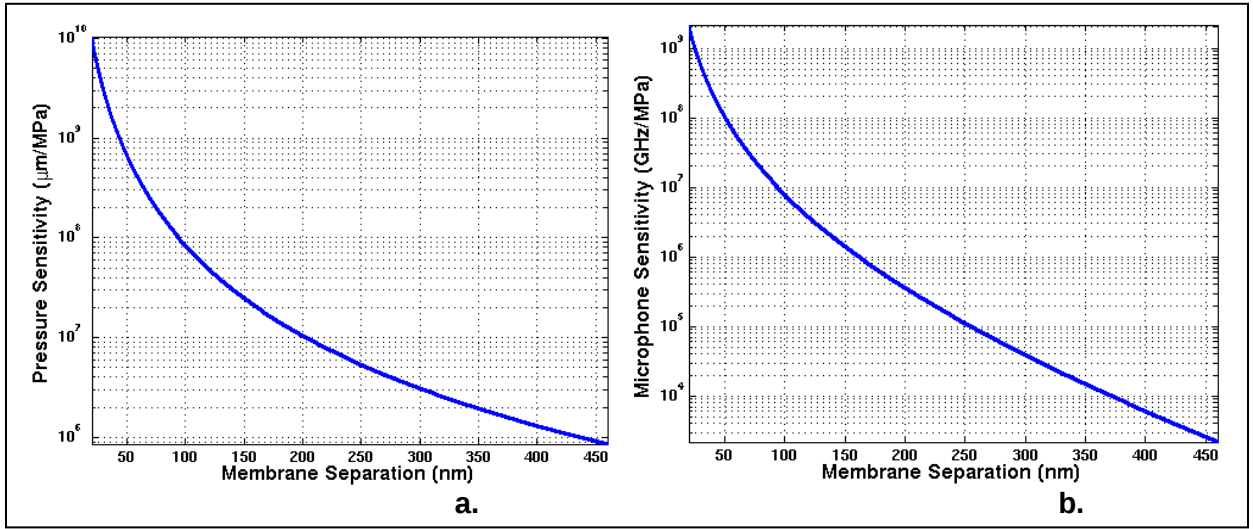


Figure 61. (a) Calculated pressure sensitivity of the lower membrane versus membrane separation distance. (b) Overall sensitivity of the acousto-optic microphone versus separation distance.

Photonic crystal cavity modes can also be used for optomechanical coupling between parallel membranes, with the advantage of increase Q , although at the expense of reduced bandwidth since the two are inversely related. Thus, an L3 cavity was also designed by completely removing only 3 air holes in an otherwise unperturbed PhC lattice, as shown in Figure 62. Cavities of this type have been optimized by modifying the air holes immediately surrounding the cavity to produce measured Q values as high as 100,000 [44].

5.6. Fabrication process

A process flow based on the MEMS fabrication facilities at Sandia has been developed that will enable test samples of the acousto-optic microphone designs shown here to be made. As shown in Figure 62, the process starts with an SOI wafer that has a layer of oxide grown or deposited on it which acts as a spacer layer between the membrane and PhC layers. Next, a second inverted SOI wafer is bonded to the first wafer and its Si handle and BOX layers are removed. The PhC pattern is etched through both Si device layers and BOX layers, and then a second pattern is transferred to just the top Si layer. Finally, the bottom Si handle layer is patterned with a backside dry etch and the Si device layers are released by removing the BOX layers only in the regions where the Si handle was etched.

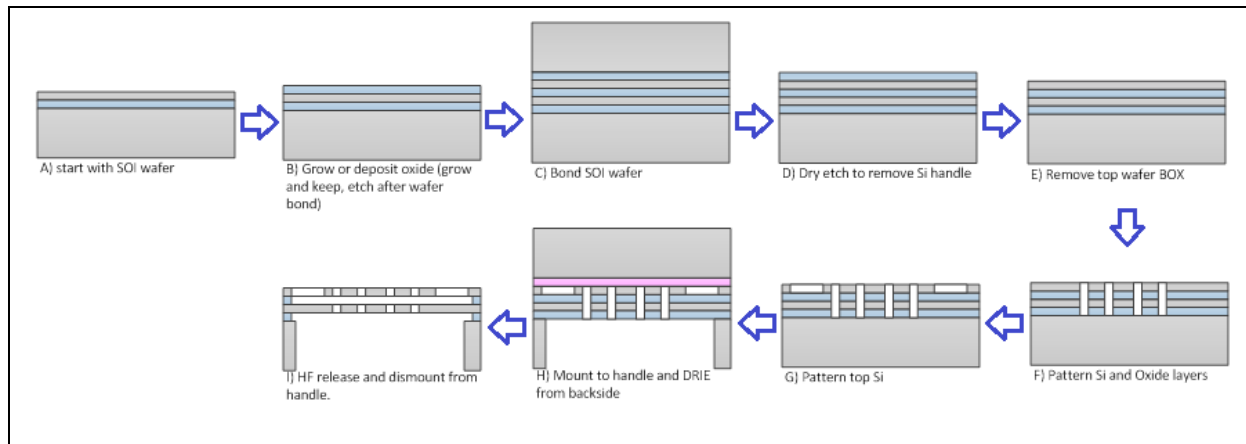


Figure 62. Process flow for PhC microphone.

6. PHOTOACOUSTIC TESTING

6.1. FTIR PAS

Most work involving trace detection of vapor phase compounds with PAS involve the use of lasers. CO and CO₂ are particularly well suited to tuning over narrow regions to excite very weak bands in the IR. The vibro-rotational bands often overlap with a number of other compounds, making application to complex mixtures impossible. In fact, most trace detection papers do not even attempt binary compositions.

A much more practical solution to complex mixtures is to use broadband, scanned excitation. While the light sources are generally much weaker, the detection limits are lower. However, despite the directly proportional sensitivity of light intensity versus signal, the much stronger fundamental absorption bands become accessible, making the tradeoffs less significant. However, parts per thousand to parts per million can be reached with certain molecules. For many applications, percent concentrations are of interest – as when explosive compounds near their LFL (lower flammability limit).

There are two broadband methods that can be used. The first, illustrated in Figure 1, diffracts the broadband light into component wavelengths, typically with a diffraction grating. An optical chopper and lock-in amplifier bring out any signal at a given wavelength and then the grating is moved to the next wavelength step. This process is slow and requires precision alignment. The other option is FTIR PAS. We chose to pursue this option for simplicity, versatility, and the array of commercial accessories to optimize our chances of success.

The first photoacoustic spectra we took used the Model PAC300 Thermo-Nicolet ESP photoacoustic detector from MTEC Photoacoustics, Inc. (Ames, IA) in a model 6700 Thermo-Nicolet FTIR spectrometer. While designed for solid samples, we used the sample cup to secure vapor samples in the PAS chamber. Initially, carbon dioxide from the air was qualitatively increased (Figure 63). Humidity, acetone, isopropanol, and ethanol were also sampled in nitrogen (Figure 64). While the recommended background gas is helium, for optimal properties of heat capacity, nitrogen worked quite well. While the cup volume was easy to determine, the volume above it was not.

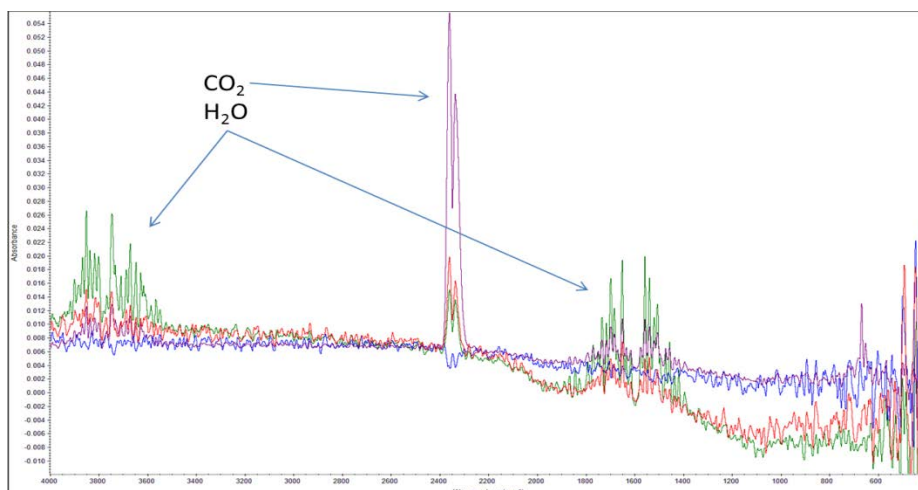


Figure 63. FTIR of partial air samples captured in PAC300 PAS system.

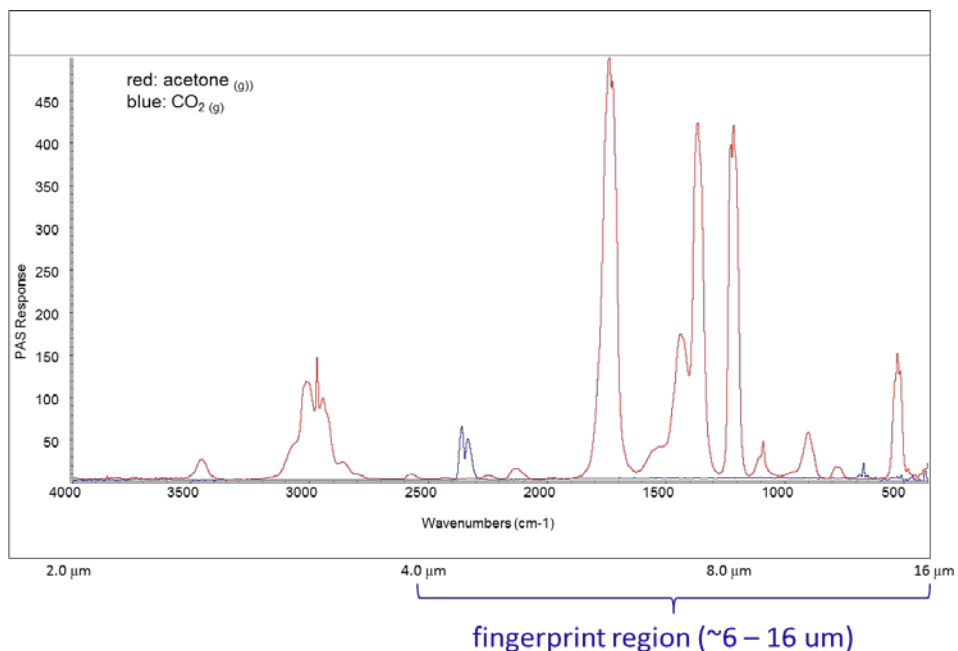


Figure 64. FTIR of microliter sample of acetone and CO₂ in PAC300 PAS system.

In another experiment, microliter volumes of acetone were dispensed into the sample cup and flushed momentarily with nitrogen before sealing the volume. Signal intensity of the carbonyl CO stretch near 1700 cm^{-1} was observed to increase over time, providing data for the rate of vaporization in nitrogen at room temperature (Figure 65). By comparing the final signal intensity versus the volume dispensed, a knee in the curve would indicate the liquid volume required to saturate of the volume. Together with vapor pressure and temperature, the volume of the sealed area could be determined. Unfortunately, the signal suddenly became very weak between experiments, and we suspect the microphone was mechanically damaged from a pressure pulse when the nitrogen flow rate was suddenly increased. This also prevented us from determining the signal strength of saturated vapor during the rate of vaporization experiments, so the rate was not determined.

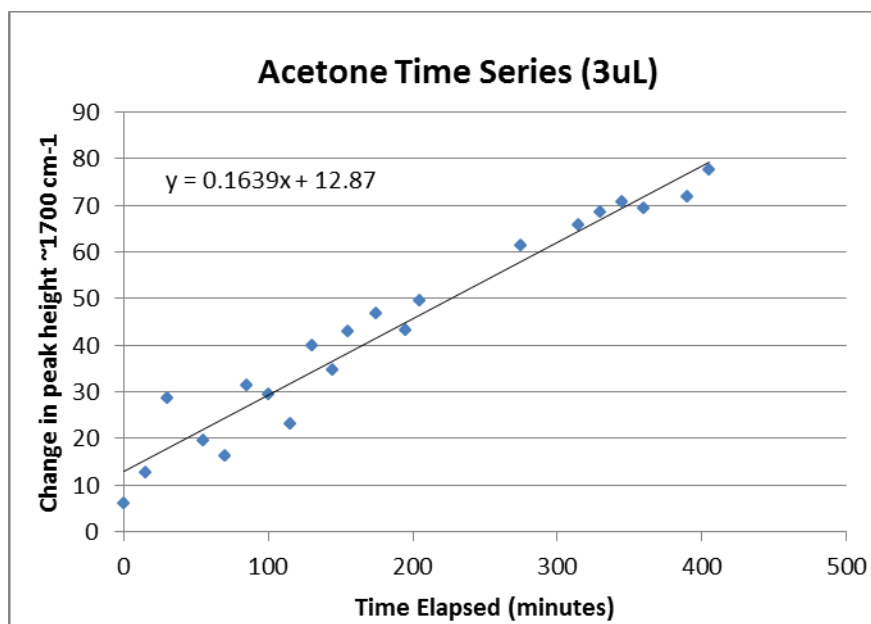


Figure 65. Evolution of carbonyl C-O stretch near 1700 cm⁻¹ with time for acetone.

6.2. Fully Assembled System

6.2.1. Setup

Based on readiness of the interferometric microphones, one was selected for full integration in the optical MEMS PAS setup. The experimental setup described in 3.4 Acoustic Testing was integrated with the FTIR spectrometer. A degree of customization was required. First, the microphone was glued into the PAS cell. A single PIR fiber optic was secured into the PAS cell ferrule, which was then compressed into the cell's receptacle. The other end of the fiber was interfaced with the launch port of a FiberMate-2[®] fiber optic probe coupler (Harrick Scientific, Inc.). (Figure 67).

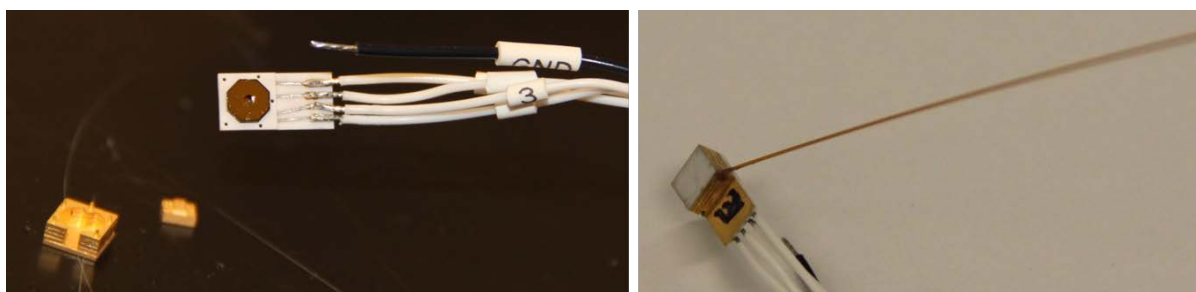


Figure 66. Left: PAS cell, FO ferrule, and microphone prior to final assembly. The substrate has electrostatic control wires soldered in place. Right: PAS cell after assembly, with capillary tube in place at corner outlet. Inlet port is also visible at corner to the lower left.

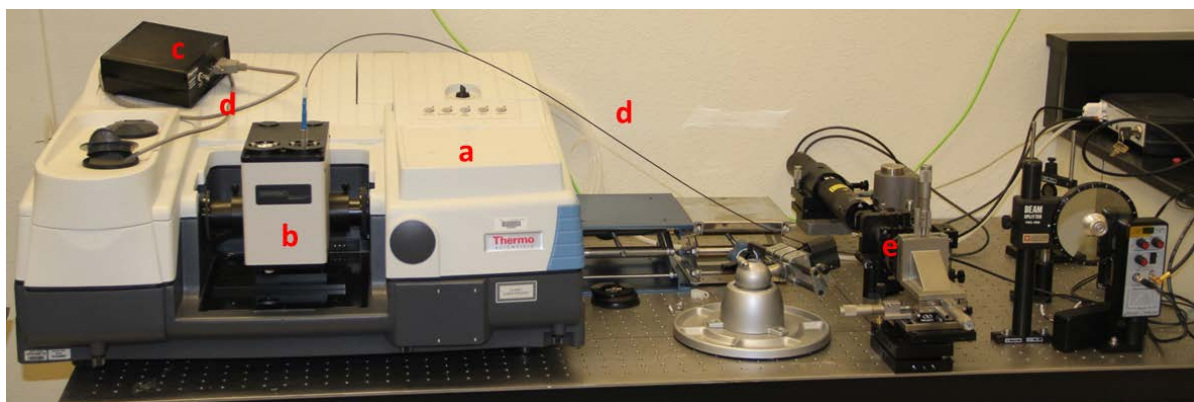


Figure 67. FTIR experimental setup. (a) FTIR; (b) fiber optic interface; (c) universal detector interface; (d) PIR fiber to PAS cell in (e). Not shown: photodetector amplifier and cable connecting it to (c). The setup at the right side of the figure is described in Figure 15.

Laser light bounced off the microphone's diffraction grating was once again captured on the differential photodetector. The resulting electrical signal was amplified by an Endevco model 133 Isotron® signal conditioner. The static signal was balanced to zero amplitude using a continuously variable neutral density filter and an oscilloscope. The oscilloscope was used to capture signals from acoustic impulses created by lifting one end of hex wrench and gently dropping it from up to an inch onto the optical bench. Any motion to the table was observable. The impulses enabled fine tuning of the electrostatic voltage. When the positive and negative peaks were equal in magnitude, the grating/membrane separation was at the most sensitive part of the transduction curve.

The differential photodetector signal was then connected to the FTIR. A Nicolet Magna-IR/Nicolet X700 External Detector Interface was interfaced to the FTIR through an auxiliary detector port. The entire setup, with the exception of power supplies and amplifiers, were located on a floated optical bench. The system was then ready for FTIR PAS. Unfortunately, a great deal of vibration and electrical noise were present on the system. These were visible with the oscilloscope, and the FTIR spectrometer's interferograms. They varied periodically, especially the 60 Hz signal that was able to pass through the differential photodetector's band pass filters (set to high pass 100 Hz and low pass 100 kHz). These sources of noise were not nearly as prevalent in the optical test labs, where thicker optical benches sat on noise-isolating floors. Further extremes were likely necessary for these very sensitive microphones.

6.2.2. Testing

Nonetheless, vapors of acetone and isopropanol were tested. Liquids were dispensed into Tedlar bags, which were then filled with helium. Up to 24 hours was allowed for vapor phase equilibrium to establish. Samples were then extracted via 25 mL syringe, or the entire bag was used. Either way, a 200/360 micron deactivated capillary column was used to direct vapors directly into the photoacoustic cavity through the corner port. A second capillary 0.5 meters long was sealed to the vent port. FTIR spectra were collected. Despite many variations on methods to eliminate and control sources of noise, spectral averaging, and digital filtering, spectra could not be teased from the data.

IR transmission through the PIR fibers and fiber optic probe coupler was previously verified by connecting two 1 meter long PIR fibers with an SMA barrel connector and jumping the launch and receive ports together. The level of CO₂ present in the cell was readily detectable by its IR absorption peak, and it could be modified by nitrogen purging. The C-H stretch of acetone was significantly muted, as transmission through the PIR fiber optic is reduced in this region.

Microphone verification was performed with the oscilloscope. The hex wrench impulses were varied in amplitude by dropping the wrench end from up to an inch in height. This was generally enough to saturate its response. The signal could be amplified by less, but when the interferometric cavity exceeds a half-wavelength of the 633 nm laser wavelength, the signal wraps and requires more sophisticated algorithms to correct. The amplifier gain was set so a signal that did not appear to wrap spanned just under ± 10 V. Figure 68 shows such an impulse, raw and band pass filtered (post-processing) to improve noise.

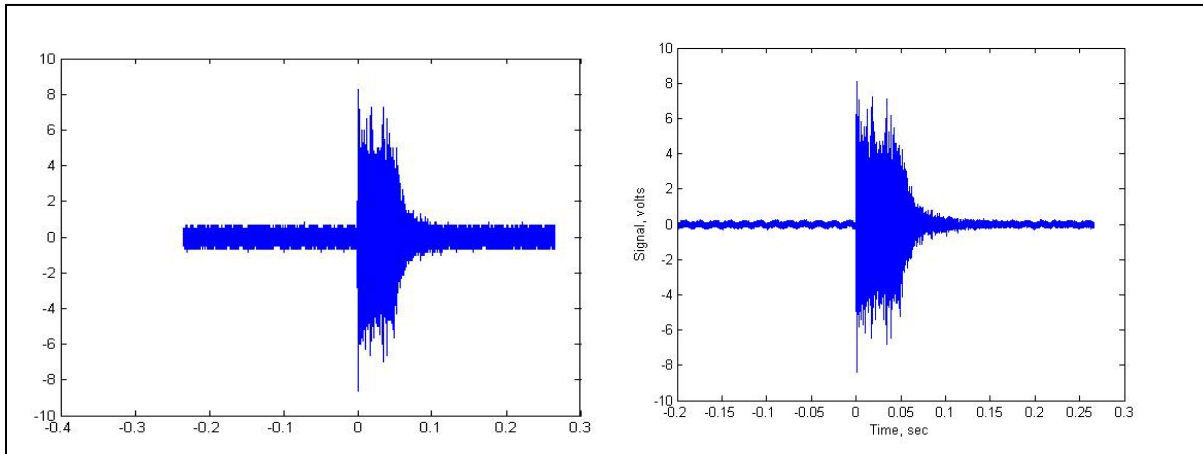


Figure 68. Raw impulse signal (left) and filtered signal (right).

Taking the power spectrum of the raw acoustic signal (Figure 69) was insightful. The strongest peak occurred at 23.685 kHz. This was in the right region for the calculated fundamental natural resonance of the microphone. It was clearly excited by the impulse of the falling wrench. Another much broader band of frequencies was found in the region of 9-15 kHz. This was likely responsible for the high pitched ringing that was clearly audible. There was very little frequency content beyond the natural resonance. The few observable peaks appeared as a dampened series, and may have been combination tones or higher order resonances. These were not aliasing artifacts, as the sampling rate was 10 MHz. The signal could be significantly cleaned up by band passing between 1 to 30 kHz to reduce random noise.

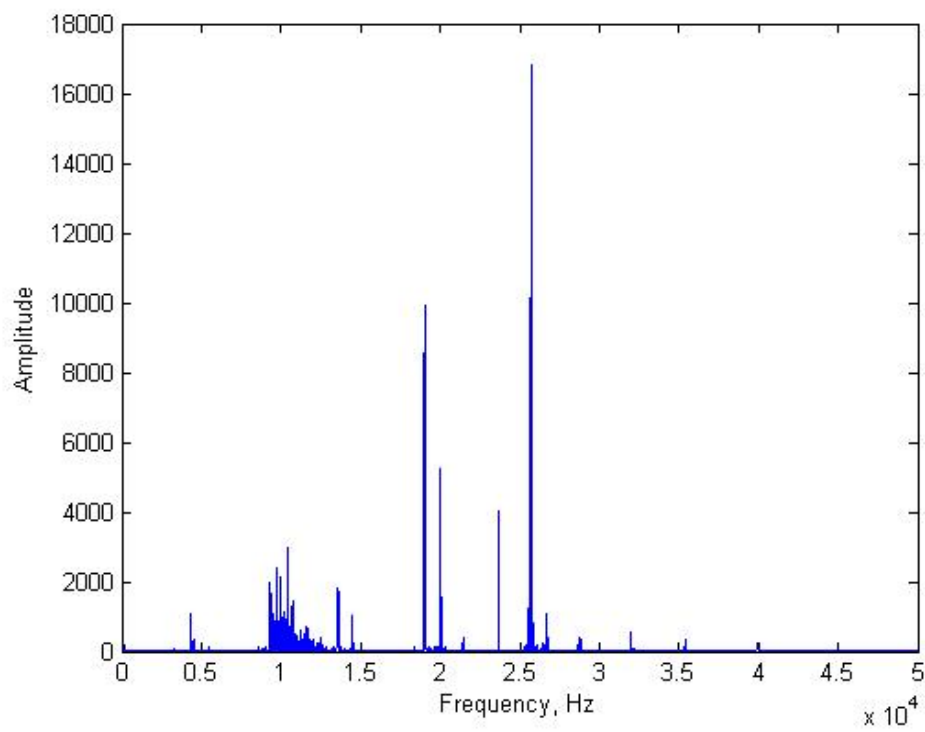


Figure 69. Power spectrum of raw acoustic signal in Figure 68.

7. CONCLUSIONS/FUTURE DIRECTIONS

A great number of advances were made during this project, especially with regard to optical microphones. An existing, proven microphone design was further refined to nanometer displacement detection. An optical micro-ring microphone was taken from concept to a fully functional design. The sensitivity was similar to the interferometric design. The complex membrane stackup proved more challenging to construction than the 3 mask design would indicate. Finally, a photonic crystal lattice microphone was conceived, modeled, and had its performance projected into a photoacoustic application. Realization of this concept would enable interesting tradeoffs to improve the microphone applications. Smaller cavities and thicker membranes could be built for a greater linear dynamic range. The stiffer designs would have higher Q factors, improving fidelity of sound. Single chip microphone arrays could be built, which has implications from ultrasounds to collision mitigation for UAVs.

Advances were also made in understanding and prediction of MEMS-scale photoacoustic cells. The temporal pressure forcing was modeled along with thermal effects and breathing modes. The simulations developed here guided the design of our cells. Other applications that could benefit include other microphone and hydrophone work, micro-mixers, micro-coolers, and any application were a small orifice interfaces with a cavity that can induce breathing modes.

While photoacoustic spectra using the FTIR spectrometer were elusive, there is much additional work that could be done. Clearly, noise and vibration isolation must be improved. Electrical noise sources can be improved with line filtering power line conditioners. Other types of IR fibers could be used. Hollow core silver/silver iodide fibers look promising for short distances. Finally, it may be that the tradeoff of using lasers more than compensates for the lack of broadband tuning if environments have low complexity.

Figure 70 shows a conception endpoint. The PIR fiber optic can be interfaced with a materials compatibility/accelerated aging vessel with a MEMS PAS cell inside. The vessel may be placed in an oven with the fiber connected for real-time measurement of evolving environments within the vessel. Or, the vessel, and many others, could be pulled from an oven at a periodic basis for in-situ analysis without breaking the air-tight seal. If still in a laboratory environment, the lossy IR fibers can be replaced with open-air paths to focusing optics aligned with internal, free-standing PAS cell. Interferometric detection a small standoff distance will allow for acquiring PAS spectra without direct contact of the interior atmosphere. Indeed, there is much more that could be done.

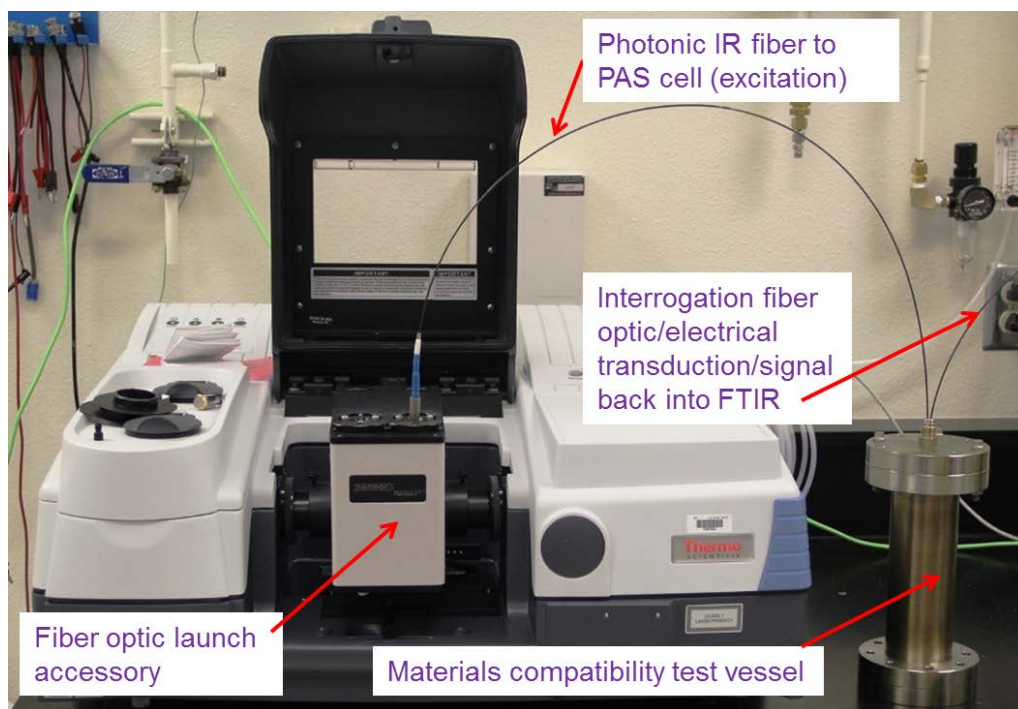


Figure 70. Concept of monitoring materials compatibility test with a MEMS PAS cavity within the test vessel, possibly in an oven.

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