BROADBAND ALL-OPTICAL ULTRASOUND TRANSDUCERS FOR HIGH-RESOLUTION ULTRASOUND IMAGING

by

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A dissertation submitted in partial fulfillment Of the requirements for the degree of Doctor of Philosophy (Electrical Engineering) In The University of Michigan 2008

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To my family

ACKNOWLEDGEMENTS

I must first thank Matt O'Donnell for providing everything I wanted for my graduate school journey. His passion for research and his deep insights into ultrasound imaging wowed me countless times. He's also a class act and a great role model in life. I sincerely mean it when I tell other fellow graduate students that I have the BEST adviser in the world!

I would also like to thank Professor Norris, Professor Mycek, Professor Winful, and Dr. Ye to serve on my committee and give me valuable advice on my research.

Shai Ashkenazi was the first person I met in the lab, who taught me all I could handle about the etalon and shared plenty of MATLAB codes which saved me a lot of time afterwards. Tak Buma spent many hours in the hot summer of 2005 to show me his home-built laser, and how to get signals from the black PDMS films. Sheng-Wen Huang is like a big brother to me. I got tremendous help from him on the beamforming codes, and chatting with him around our optics table always kept my spirits up no matter how boring experiments were. These three guys are not only great colleagues, but also mentors to me. I can't put into words how much I appreciate their help.

The BUL lab has been a warm and supportive environment. Every member has helped me directly or indirectly: Hua, Congxian, Russ, Todd, Marwa, Christine, Ragnar, Yan, Kang, Zhen, Becca, Kyle, Binh, and Tim. Dan and Wei from Shu Takayama's lab taught me everything about PDMS, which was a huge part of my work. Brian Johnson from the cleanroom also helped tremendously. The machine shop folks at both the engineering school and the physics department deserve a lot of credit for making the water tanks and countless other components in my setup. I can't thank enough the administrative staff in Biomedical Engineering, Electrical Engineering, and Physics. I also thank Professor Kirk Shung from the Resource Center for Medical Ultrasonic Transducer Technology at the University of Southern California for supplying the high frequency piezoelectric transducers.

I want to thank Professor John Holland and his books for inspiring me into science, and making the University of Michigan one of my dream schools. I'm grateful for every beautiful football Saturday in the fall, where I share joy and disappointments with 110,000 other 'Michigan Men'.

Finally, I would like to give my special thanks to my wife for her love and support. She makes home the sweetest place to be. I owe so much to my mom, who sacrificed a lot for me over the years. She's the real hero and warrior. All my family members have encouraged and supported me in a big way, and I will never forget that.

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CHAPTER 1

INTRODUCTION

1.1 Motivation

Biomedical imaging techniques, including ultrasound imaging, X-ray computed tomography (CT), magnetic resonance imaging (MRI), and optical imaging, play critical roles in medical diagnosis and therapy. Each imaging modality has its unique operating principle, with certain advantages and disadvantages compared to other technologies. Ultrasound imaging relies on the detection of mechanical properties within biological tissues, and has been irreplaceable because of its real-time imaging capabilities and ease of use for both doctors and patients.

During the past few decades, the use and development of high-frequency ultrasound has dramatically expanded the frontiers of ultrasound imaging, making better image resolution possible in various clinical applications. Typical applications include ophthalmology [1-4], dermatology [5-7], intravascular imaging (IVUS) [8] and small animal imaging [9-11]. An integral part of any ultrasound imaging system is a transducer or transducer array responsible for the generation and detection of acoustic waves. Regardless of the type of transducer used, the spatial resolution is primarily determined by the size, spacing, physical and electrical properties of the array elements. Currently, the unavailability of two-dimensional (2D) high-frequency transducer arrays is a major bottleneck preventing further development of high-frequency ultrasound, especially in real-time 3D high-resolution applications.

There are three major types of ultrasound transducer, based on the methods used to generate and detect acoustic waves. The first is piezoelectric technology, the dominant technology since the birth of medical ultrasonics. The second is called capacitive micromachined ultrasound transducers (CMUTs), a rapidly surging technique since the 1990s. However, it is extremely difficult to build 2D high-frequency arrays using these two types of transducers. This provides motivation for the investigation of a third transduction mechanism: optoacoustics, an attractive alternative potentially overcoming the major limitations of piezoelectric and CMUT devices. The present work addresses the design and development of optoacoustic transmitters and detectors, and then focuses on the combination of these two into an integrated device for practical imaging applications.

1.2 Piezoelectric transducers and arrays

Piezoelectric materials are able to transform an applied voltage into pressure waves, and likewise, they respond to an external acoustic field by generating a voltage. These processes provide the generation and detection mechanisms for piezoelectric transducers. Immediately after World War II, piezoelectric crystals like quartz were used, and were quickly replaced with much more efficient materials, the piezoceramics like lead zirconate or PZT [12-13]. Since then, virtually all commercial medical ultrasound systems use piezoelectric transducers for imaging arrays.

There are two major schemes to form an imaging aperture. The first mechanically scans a single element transducer, and the second utilizes either 1D or 2D assembled transducer arrays. These two configurations are shown in figure 1.1.

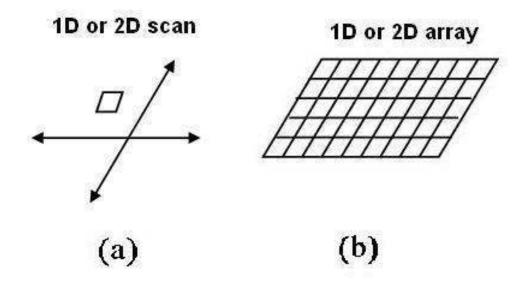


Figure 1.1: Two schemes utilizing piezoelectric transducers to form an imaging aperture: (a) 1D or 2D mechanical scan of a single element; (b) 1D or 2D assembled array

Mechanical scan of a single element transducer to form an array is often referred to as a synthetic array approach. Its biggest advantage is that the frequency range of the transducer can far exceed that used in the other approach. For example, state of the art individual elements can operate near 100 MHz [14-15]. Also, the spacing between elements is defined by the step distance of the scan, and can be easily reduced to a few microns using commercial motor systems. However, the disadvantages of this scheme are also significant. First, the very low f-numbers of these high-frequency transducers severely restrict the depth of the field. Second, a bulky scan system needs to be attached to the transducer, which makes it much more difficult to build a convenient and compact system. Third, it takes significant time to scan the required imaging aperture, meaning that the real-time imaging capabilities of ultrasound are abandoned. This is certainly not a desirable situation in real clinical applications.

On the other hand, transducer arrays, whether 1D or 2D, are extremely difficult to fabricate with piezoelectric techniques, especially at higher frequencies. The best arrays developed to date include a 64-element 35 MHz 1D array [16] and an 8-element 55 MHz annular array [17]. Fully sampled 2D arrays have not even been attempted at frequencies above a few MHz [18]. This means that we either sacrifice high resolution and retain high frame rates using a low-frequency array, or sacrifice frame rates and retain high resolution by mechanically scanning a high-frequency single element transducer. Nonetheless, real-time 3D high-resolution imaging is not feasible with piezoelectric technology at present.

Major difficulties and challenges of designing and fabricating piezoelectric arrays using the conventional "dice and fill" approach have been investigated by numerous researchers, and are well documented [14-25]. The fundamental constraint in a phased array is the element spacing required, normally half of the acoustic wavelength for full beam steering. For example, 10 µm element spacing is required for a 75 MHz phased array, well beyond the capabilities of current construction methods. Meanwhile, dicing piezoceramics to micron scale elements is also a tough task. Large numbers of electronic connections pose an even bigger challenge: an electronic wire connection is required for each array element; therefore large, 2D arrays consisting of thousands of elements require thousands of wire connections, which are extremely difficult to integrate into a

commercial ultrasound scanner. Other factors include crosstalk between elements, as well as lack of quality high frequency materials and electronics.

Piezoelectric technology continues to grow with extensive efforts from numerous investigators. However, building 2D high-frequency arrays remains difficult despite recent improvements in the mature technology for building low-frequency arrays.

1.3 Capacitive micromachined ultrasound transducers

The idea of capacitive transducers is at least as old as piezoelectricity. Recent advances in microfabrication technology, however, have made it possible to build highperformance CMUTs [26-36] to rival piezoelectric transducers in practical applications. A CMUT consists of a capacitor cell with a metalized membrane as the top electrode, and a heavily doped silicon substrate below the membrane as the bottom electrode. A DC voltage is applied between the two electrodes, so that the membrane is attracted toward the bulk due to the electromagnetic force while the induced stress works otherwise. An AC signal applied on top of the DC-biased capacitor generates ultrasound, and the reverse process holds as well: acoustic waves modulate the capacitor spacing, producing a corresponding electronic signal. CMUTs have challenged many assumptions about ultrasonic transduction, and have enabled revolutionary advances in ultrasound imaging. Several notable advantages are listed as follows.

First, fabrication of 2D arrays with large element counts is possible using standard silicon integrated circuit fabrication technology, and 128 by 128 element 2D arrays have been successfully fabricated and tested for 3D imaging [34-35]. Second, a CMUT inherently has wide bandwidth: single elements operating at 60 MHz [37-38], and 1D 64

element array operating at 45 MHz [39] have been reported. Third, convenient integration of front-end electronics during the fabrication process seems promising at this moment [40-42], which might solve the problem of electronic wire connections. Note that this wire integration technique is being developed but is not yet well-established.

As promising as CMUT technology is, it's still in its childhood, and there are some major challenges left to overcome before 2D high-frequency CMUT arrays can be fabricated and used in practical biomedical applications. First and foremost, integration of electronic wire connections, though promising right now, has not been realized for large arrays. Second, even though 2D arrays have been fabricated and evaluated, they have been limited to a few MHz, while high-frequency CMUT arrays have been limited to 1D. Breakthroughs in fabrication techniques are needed to make this leap. Third, high cross coupling between elements remains an issue. Finally, reducing element size and spacing to a few microns is also not a trivial task. Therefore, there is still great need to explore other transduction mechanisms for 2D high-frequency arrays enabling real-time 3D high-resolution ultrasound imaging.

1.4 Optoacoustic systems

Optoacoustic transduction has been a subject of study for nearly as long as its rivals. Recent advances in high-power lasers, as well as micro and nano fabrication techniques, have sparked the revival of optoacoustic research, and have made it a realistic alternative to piezoelectricity and CMUT. Optoacoustic transducers rely on optical generation and detection of ultrasound, where two laser beams are used as input/output

vectors instead of electronic signals, one for ultrasound generation and the other for ultrasound detection.

A variety of mechanisms have been studied for optical generation of ultrasound, including electrostrictive mixing [43-45], radiation pressure [45-46], stimulated Brillouin scattering [47], ablation [48-50], and thermoelastic expansion [51-54]. Historically, all methods suffered from weak acoustic signals and low transduction efficiencies. Ablation normally generates the largest signals, but its destructive nature makes it unrealistic for biomedical applications. Thermoelastic expansion is usually the most efficient mechanism among these candidates. A typical configuration, as shown in figure 1.2, focuses a laser pulse onto the film surface, and optical absorption rapidly causes a temperature rise in a localized volume in the film, where thermal expansion launches an acoustic wave into the overlying sample.

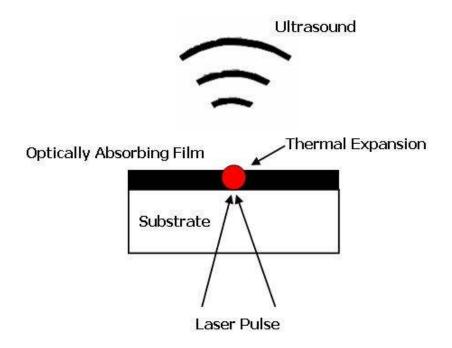


Figure 1.2: Typical configuration for optical generation of ultrasound using thermoelastic expansion

Early studies of thermoelastic expansion for optical generation of sound concentrated on utilizing metal films and generating acoustic waves in solids [51, 55-56]. 1D arrays employing multiple laser sources were also constructed [57-59], even though the number of elements was limited to less than sixteen. Surprisingly, very little research has been done to use this phenomenon for medical transducers [60-62].

The low transduction efficiency has excluded optoacoustics as a valid option for medical ultrasonics for many years. Metal films were widely used because they are easy to deposit on substrates, but their coefficients of thermal expansion are generally in the range of $10-20 \times 10^{-6}$ /K, and seldom exceed 25×10^{-6} /K, small compared with polymer materials. Apparently, transduction efficiency can be significantly improved by using materials with higher thermal expansion coefficients. For example, polydimethylsiloxane (PDMS) has a coefficient of 310×10^{-6} /K, more than ten times higher than common metals. However, PDMS is a transparent polymer, thus does not absorb optical energy.

Dr. Takashi Buma of the University of Michigan managed to make 25 µm thick black PDMS films using a mixture of PDMS and carbon black [63-65], where carbon black serves as efficient optical absorbers. An increase of over 20 dB in transduction efficiency was observed with this state of the art black PDMS film. The major drawback is that the black film attenuates the generated ultrasound by about 1dB/µm. Therefore, transduction efficiency can be further improved by reducing film thickness.

Optical detection of ultrasound has also been a subject of interest for several decades [66-85]. The most straightforward principle is called passive optical detection, as shown in figure 1.3. A laser source probes a transducing surface consisting of an optical

reflector, which can simply be a thin highly reflective optical pellicle. Acoustic waves incident on the reflector surface phase modulate the probing laser beam in proportion to surface displacements. The sensitivity of passive optical detection relies on the magnitude of the optical phase shift, generally very small because the amplitude of surface motion is typically less than the optical wavelength. In reality, the sensitivity of optoacoustic detection must be improved to rival piezoelectricity, and active optical detection, most notably in the form of resonant optical ultrasound transducers (ROUT), has been developed for this purpose.

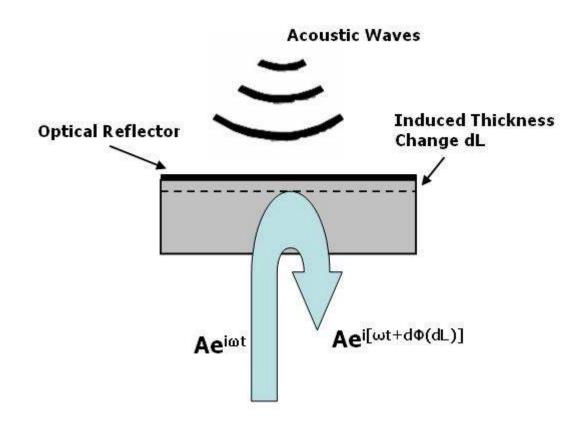


Figure 1.3: Typical scheme for passive optical detection: ultrasound waves modulate the phase of the probing laser beam.

ROUTs utilize active optical detection, and rely on the interaction of an optical field with ultrasound waves in a resonance cavity. Acoustic strain induces modulation of the probing optical field, especially the amplitude rather than the phase. With a sharp cavity resonance, the optical response is amplified, which significantly improves sensitivity of optoacoustic detection. ROUTs include etalons [66-80], microrings [81-82], fiber gratings [83], and dielectric multilayer interference filters [84].

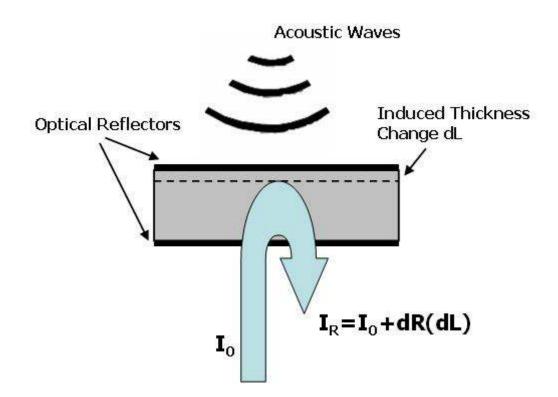


Figure 1.4: Typical scheme for active optical detection using an etalon: ultrasound waves modulate the amplitude of the probing laser beam

One of the most effective and simplest approaches uses an etalon, also known as a Fabry-Perot interferometer. The etalon consists of two reflecting mirrors. Figure 1.4 shows the typical scheme for active optical detection of ultrasound using the etalon. Light

incident from an external source undergoes multiple beam interference in the etalon and produces a reflected signal intensity that depends on the optical path length and wavelength. A resonance condition occurs when the optical path of two-way travel in the etalon bulk equals an integer multiple of the wavelength. A sharp drop in the reflected intensity is observed at or near resonance, creating a mechanism for sensitive ultrasound detection. The strain associated with an ultrasound wave passing through the etalon modulates its thickness, and therefore changes the resonance condition. This in turn changes the reflected optical intensity, thus the acoustic pressure can be measured by measuring the intensity change of the reflected signal.

Other than being used as ultrasound hydrophones [73-75], etalons have been widely employed as ultrasound detection array elements for photoacoustic imaging [80]. Photoacoustic imaging represents a hybrid imaging technology that takes advantage of both the high contrast of pure optical imaging and the high resolution of conventional ultrasound imaging. Typically, the contrast is based on the optical properties of biological tissues, and resolution is scaleable with the frequency of the ultrasound wave. Just like conventional ultrasound imaging, photoacoustic imaging relies on a transducer array to detect acoustic waves. Similarly, lack of 2D high-frequency ultrasonic detection arrays is also the bottleneck of 3D real-time high-resolution photoacoustic imaging. Etalons provide an attractive alternative for photoacoustic imaging. Up to now, most studies involving etalons have been conducted in the frequency range of 1 to 40 MHz [66-80], where conventional piezoelectric techniques and surging CMUT devices are mature and virtually irreplaceable. Etalons with broader bandwidth over 50 MHz will significantly expand the frontiers of high-frequency ultrasound and photoacoustic imaging.

The most significant advantage of optoacoustic transduction over piezoelectricity is that the size and spacing of each transmit/receive array element is defined by the focal spot of a laser beam, and can be easily reduced to several microns using conventional optics. Small element size and spacing make it suitable for synthetic aperture imaging at frequencies over 100 MHz. Also, no electronic wire connections are required because optical beams are used instead of electronic signals. Plus, an array can be easily formed and is conveniently configurable by splitting the primary laser beam and focusing the resultant secondary beams onto a programmable array of spots, avoiding the trouble of dicing and assembling the transducing surface. Another exciting advantage of optoacoustic transducers is the potential for miniaturization. Generation and detection arrays can be made small enough to fit into small volumes, such as biopsy needles, catheters, optical fibers, and guidewires.

1.5 Dissertation overview

Although both optoacoustic transmitters and detectors have been significantly improved during the past decade, an integrated device combining the two has yet to be designed and developed. This has to be done before optoacoustic transduction can be considered and used for any practical biomedical applications. This dissertation describes the optimization of optoacoustic transmitters and detectors, and focuses on the design, fabrication and evaluation of integrated broadband all-optical ultrasound transducers.

Chapter 2 describes the optimization of black PDMS films, as well as the design and evaluation of a novel optoacoustic transmitter using a 2D gold nanostructure. The bandwidth, acoustic pressure, thermal damage threshold, and transduction efficiency of both structures are characterized. The unique optical properties of the gold structure are emphasized because it provides a convenient approach for integration with optoacoustic detectors.

Chapter 3 describes how the etalon's bandwidth can be improved to well above 50 MHz by reducing etalon thickness. The optical resonance, frequency response, noise equivalent pressure, and angular response of this thin polymer etalon are presented. Photoacoustic imaging experiments are also conducted using etalon arrays, where axial and lateral resolutions of better than 20 µm are achieved.

Chapter 4 describes the design, fabrication, and evaluation of the first integrated all-optical ultrasound transducer array element. This structure combines the etalon and the gold nanostructure. Pulse-echo experiments are performed to demonstrate the acoustical performance of this device. A 1D optoacoustic array is formed by mechanically scanning an imaging target. Detailed characterization of the optical and acoustical properties, as well as preliminary imaging results, show that all-optical ultrasound transducers are extremely promising for 2D high-frequency arrays.

Chapter 5 describes the design and implementation of a theta-array imaging system combining the etalon and the black PDMS film. Pulse-echo signals display broad bandwidth of above 40 MHz, and the first 3D image from an optoacoustic array is obtained and presented.

Chapter 6 summarizes work to date and introduces possible future work. Various methods to further improve optoacoustic transmitters and detectors, as well as several immediate next steps for device integration and array expansion will be discussed.

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CHAPTER 2

OPTOACOUSTIC TRANSMITTERS

2.1 Optimization of black PDMS films

The state of the art optoacoustic transmitter is a 25 μ m thick black PDMS film consisting of a mixture of PDMS and carbon black deposited on top of a glass substrate [63-65]. The optoacoustic transduction efficiency of this film is over 20 dB better than that of metallic films, because the thermal expansion coefficient of PDMS is significantly higher than most common metals. The optical absorption depth of the black PDMS film is estimated to be about 1 μ m [65], thus the region where the thermoelastic effect takes place is a thin layer 1 μ m from the interface into the film. However, the film is about 25 μ m thick, which means that more than 20 μ m of the overlying film doesn't contribute to the thermoelastic effect at all, but simply attenuates the generated ultrasound, especially at frequencies higher than 50 MHz. As a result, a thinner film will lead to improvements in thermoelastic conversion efficiency, as well as the center frequency and bandwidth of the generated acoustic waves. This motivates us to improve the fabrication techniques to reduce the thickness of the black PDMS films.

The use of carbon black is the key constraint to reducing film thickness. Generally, PDMS can be spin coated into very thin films with thickness of a few microns. However, PDMS is a transparent silicone, so carbon black must be added as an optical absorber to convert light to sound. Our old 25 μ m thick sample is a mixture of PDMS, toluene, and carbon black (25 nm particle size, low structure; Raven 2500 Ultra, Columbian Chemicals Inc.), spin coated onto a glass substrate at 2000 rpm for 2 minutes. The key to making a thinner black PDMS film is to significantly improve dispersion of carbon black into PDMS, thus producing a uniform film when spin coated at a higher speed. Dispersion can be improved by choosing a different kind of carbon black with larger particle size, and high structure (increasing oil absorption). We also observed that although toluene decreases the viscosity of the PDMS mixture, it actually prevents carbon black (45 nm particle size, high-structure; Raven 14, Columbian Chemicals Inc.) with a ratio of 5.5:1. The mixture is spin coated (WS-400A-6NPP/LITE, Laurell Technologies Corp.) at 5500 rpm for 2 minutes, and then cured at 100 degrees for an hour. The thickness of the film is 11 μ m as measured with a surface profiler (Alpha Step 500 Profilometer, KLA Tencor Inc.).

The basic experimental setup for thermoelastic generation of ultrasound is shown in figure 2.1. The black PDMS film is mounted on a sample holder at the water surface. A laser pulse is focused onto the black PDMS film through the glass substrate. Ultrasound is detected by a spherically focused transducer sealed at the bottom of the water tank by two 'O' rings.

In our first experiment, the excitation source is a directly modulated, single transverse mode InGaAsP diode laser (ML976H6F, Mitsubishi Electric & Electronics USA Inc.) emitting at a wavelength of 1550 nm, combined with 980 nm pump light (SDLO-2400-110, SDL Inc.), followed by an erbium-doped fiber amplifier (EDF-555,

Thorlabs Inc.). This produces a 10ns (50 % fractional) laser pulse with energy of 30 nJ, focused to a spot size of 25 µm using a 3.1 mm focal length aspherical lens (C330TM-C, Thorlabs Inc.). An f/1.8, LiNbO₃ transducer (Resource Center for Medical Ultrasonic Transducer Technology, University of Southern California) with a focal length of 3 mm and a center frequency of 45 MHz was used to record the acoustic signal at a distance 3 mm from the absorbing film (i.e., at the focal length of the transducer). The detected signal was amplified by 30 dB (5910R, Panametrics Inc.) before data was captured by a digitizer board (CS8500, Gage Applied Technologies Inc.) with 8 bit resolution and a sampling rate of 500 MS/s.

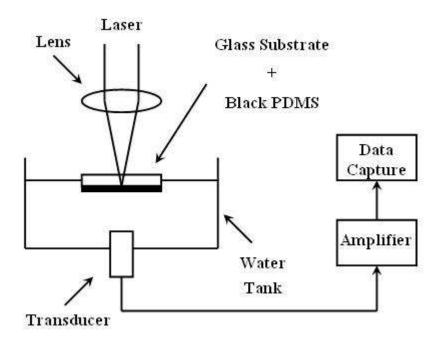


Figure 2.1: Experimental setup for thermoelastic generation of ultrasound.

For comparison purposes, both the 11 μ m black PDMS film and the old 25 μ m film are measured. Figure 2.2 (a) shows the detected signal from our new 11 μ m black

PDMS film, which has been averaged 1000 times using MATLAB (The Mathworks Inc.). Meanwhile, the detected signal from our old 25 μ m black PDMS film, also averaged 1000 times, is shown in figure 2.2 (b). Thermoelastic transduction is increased by 6 dB compared to the old film.

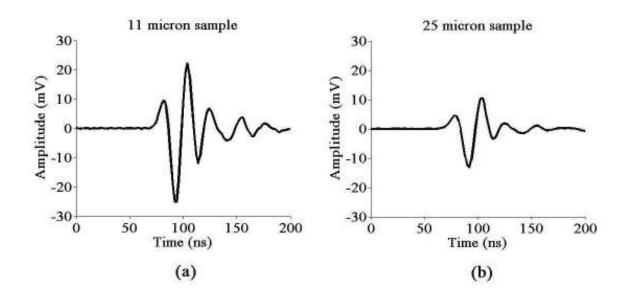


Figure 2.2: With a 10 ns laser pulse, the signal from (a) new 11 μ m black PDMS film and (b) old 25 μ m PDMS film.

Corresponding spectra (each has been normalized to its own peak) for the two films are shown in figure 2.3. Clearly, the new film excels at higher frequencies (above 40 MHz). This demonstrates that the majority of the black PDMS film (i.e., the part overlying the absorption region), behaves as an attenuator for high frequency acoustic waves, and attenuation is relatively larger at higher frequencies. Faster transient heating will generate ultrasound with higher frequency components. From figure 2.3 it is clear that a 10 ns long laser pulse can generate ultrasound with center frequency of 45 MHz and -6 dB bandwidth of 56 %. The actual bandwidth might be higher than observed, because it is limited by the 55 % bandwidth of the transducer.

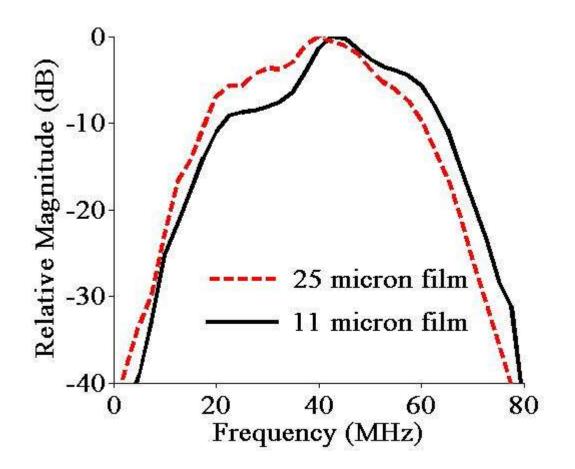


Figure 2.3: Spectrum comparison of the new 11 μ m film (black solid curve) and the old 25 μ m film (red dashed curve) with a 10 ns laser pulse. Magnitude is relative to spectral peak of each signal separately.

The optical pulse length must be decreased to about 5-6 ns to demonstrate the capability of the film to generate acoustic waves with frequencies above 50 MHz. We switched the optical source to a commercial high energy solid state laser (Surelite, Continuum, Inc.), which produces a 5 ns laser pulse with energy of 200 mJ (λ =1064 nm). We coupled the laser beam into a multimode fiber with core size of 200 µm, and output it through a collimator (11 mm focal length; F220SMA-B, Thorlabs Inc.); this reduces the pulse energy to about 500 µJ. Before the pulse is focused onto the film, it goes through a

set of neutral density filters (optical density=3.0), which further reduces the energy to about 500 nJ. An f/1.4, LiNbO₃ transducer (Resource Center for Medical Ultrasonic Transducer Technology, University of Southern California) with a focal length of 4.1 mm and a center frequency of 80 MHz is used to record the acoustic signal. Again, the thermoelastic source is placed at the acoustic focus of the transducer.

Figures 2.4(a) and (b) show the detected signal, averaged 1000 times, from our new 11 μ m and old 25 μ m films respectively. The new film shows an increased efficiency of 11 dB compared to our old sample when using the 5 ns laser source. This again confirms that higher frequency components are attenuated more in the black PDMS film.

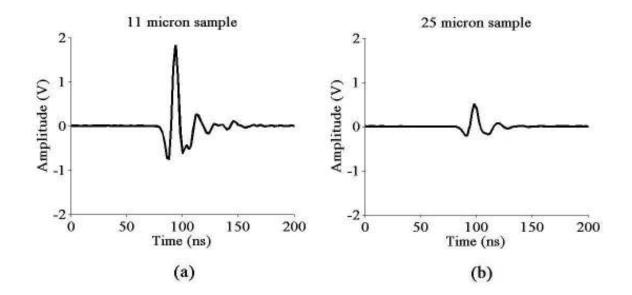


Figure 2.4: With a 5 ns laser pulse, the signal from (a) new 11 μ m black PDMS film and (b) old 25 μ m PDMS film.

Corresponding spectra (each has been normalized to its own peak) for the two films are shown in figure 2.5. The center frequency of the generated ultrasound is 60 MHz, with a -6 dB bandwidth of 80 %. Both the center frequency and the bandwidth have been improved compared to that generated by 10 ns pulses.

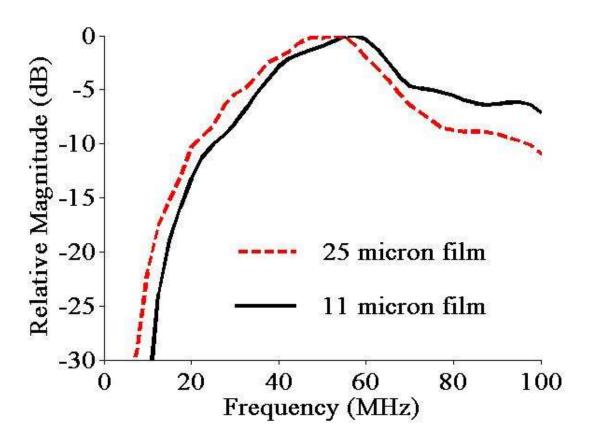


Figure 2.5: Spectrum comparison of the new 11 μ m film (black solid curve) and the old 25 μ m film (red dashed curve) with a 5 ns laser pulse. Magnitude is relative to spectral peak of each signal separately.

In addition to the thermoelastic transduction efficiency, center frequency, and bandwidth of the 11 µm black PDMS film, the acoustic power produced on each optical excitation also plays a key role for the film to be potentially used for high-frequency realtime imaging, because sufficient acoustic power leads to a high signal to noise ratio (SNR). High SNR per pulse can eliminate the need for signal averaging, an important consideration for real-time imaging. As an initial test of the ultimate power possible with this film, the optical power per pulse was increased significantly from the 500 nJ used to create the signal presented in section four. Up to a pulse power of 50 μ J, the acoustic signal increased linearly with optical power (i.e., the 50 μ J signal was 40 dB larger than the 500 nJ one). Above 50 μ J, the acoustic power no longer increased linearly with optical power and the film was thermally damaged at a threshold of about 150 μ J delivered to a spot size of 25 μ m, representing a fluence of 24 J/cm².

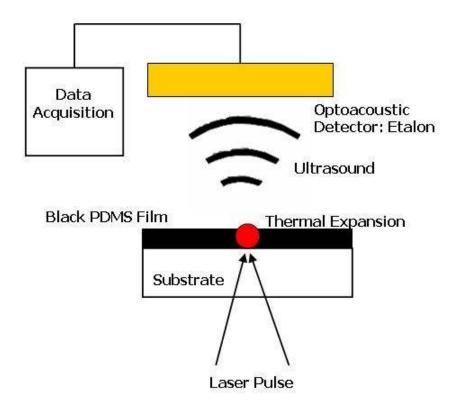


Figure 2.6: Experimental scheme for measuring the acoustic pressure from the black PDMS film using an etalon.

The acoustic pressure produced at an optical power of 50 μ J delivered to an optical spot size of 25 μ m is measured using an etalon structure [66-80]. The experimental scheme is shown in figure 2.6. First, a 10 MHz piezoelectric transducer was

calibrated using a fiber-optic hydrophone [86]. Then this 10 MHz piezoelectric transducer is used to calibrate the etalon placed at the acoustic focus of the transducer. The etalon output was 360 mV at an acoustic pressure of 3 MPa. The piezoelectric transducer was then removed and the black PDMS film was placed 10 mm from the etalon. With an optical power of 50 μ J delivered to an optical spot size of 25 μ m, the etalon output was 100 mV. Assuming that the response of the etalon is linear, and also taking into account the frequency response of the etalon, the acoustic pressure is estimated to be about 800 kPa at a distance of 10 mm. This pressure should be sufficient for potential applications in real-time acoustic microscopy.

2.2 Theoretical spectrum

In this section, the theoretical spectrum of the generated ultrasound signal from a thermoelastic source is derived, and is compared to the experimental spectrum. First, it is beneficial to show that theoretically, an ideal thermoelastic source generates ultrasound with its temporal profile proportional to the time derivative of the input optical pulse. Thermoelastic generation of acoustic waves is governed by two equations: the heat conduction equation and the acoustic wave equation. The heat conduction equation, which determines the temperature distribution, is shown in the following expression [87]:

$$\frac{\partial T}{\partial t} = \kappa \nabla^2 T + \frac{1}{\rho C} h(\vec{r}, t)$$
(2.1)

where κ is the thermal diffusivity, ρ is the density, *C* is the specific heat capacity, and *h* is the rate of energy deposited per unit volume. In our case, energy is deposited by optical absorption.

The wave equation for the scalar potential ϕ is given by [88]:

$$\nabla^2 \phi - \frac{1}{c^2} \frac{\partial^2 \phi}{\partial t^2} = \frac{3B}{\rho c^2} \alpha_L T \tag{2.2}$$

where *c* is the longitudinal wave speed in the medium, *B* is the bulk modulus, α_L is the linear coefficient of thermal expansion, and *T* is the temperature rise. The pressure is related to the scalar potential by $p = -B\nabla^2 \phi$. The temperature distribution determined by (2.1) acts as a source term in (2.2).

A solution for equation (2.2) can be written as:

$$\phi = -\frac{1}{4\pi} \frac{3B\alpha_L}{\rho c^2} \int d\vec{r} \frac{\vec{r} \cdot \vec{r} \cdot \vec{r}}{|\vec{r} - \vec{r}|}$$
(2.3)

In the far field, the distance (a few millimeters) is much larger than the optical spot size (less than 50 μ m). Thus the pressure can be written as

$$p = \frac{1}{4\pi} \frac{3B^2 \alpha_L}{\rho c^2} \frac{1}{r} \int d\vec{r} \frac{\partial^2 T(\vec{r}, t - \frac{r}{c})}{\partial t^2}$$
(2.4)

Now we turn to equation (2.1). The laser pulse duration used here is generally less than 20 ns; therefore, neglecting thermal diffusion (typically takes over 1 μ s) during acoustic generation is a reasonable approximation, yielding:

$$\frac{\partial^2 T}{\partial t^2} = \frac{1}{\rho C} \frac{\partial h(\vec{r}, t)}{\partial t}$$
(2.5)

Substituting (2.5) into (2.4) yields:

$$p = \frac{1}{4\pi} \frac{3B^2 \alpha_L}{\rho c^2} \frac{1}{r} \int d\vec{r} \frac{\partial h(\vec{r}, t - \frac{r}{c})}{\partial t} = \frac{1}{4\pi} \frac{3B^2 \alpha_L}{\rho c^2} \frac{1}{r} \frac{\partial I}{\partial t}$$
(2.6)

where I is the total optical energy incident onto the film. Therefore, with appropriate approximations, the pressure in the far field is proportional to the time derivative of the laser pulse:

$$p_{far} \propto \frac{1}{r} \frac{\partial I}{\partial t}$$
 (2.7)

In an ideal situation, the spectrum of the generated acoustic signal can be derived by taking the Fourier transform of the derivative of the laser signal. Figure 2.7 (a) shows the recorded signal of the 5 ns laser pulse used for optoacoustic generation in the previous experiment, and figure 2.7 (b) shows the spectrum of its derivative.

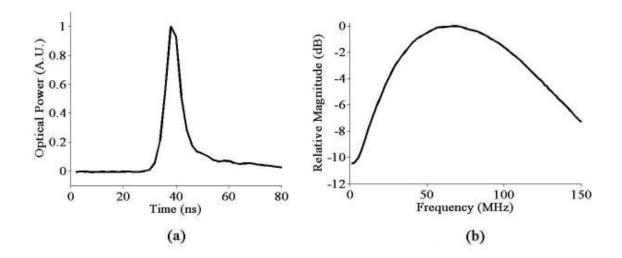


Figure 2.7: (a) Signal of the 5 ns laser pulse (b) Spectrum of the derivative of the 5 ns laser pulse.

To determine the spectrum of the actual ultrasound signal recorded by the 80 MHz transducer, the frequency response of the transducer must be taken into account. A pulse-echo signal from the transducer is shown in figure 2.8 (a), while its spectrum is displayed in figure 2.8 (b). The receive-only frequency response of the transducer can be approximated as the square root of the spectrum of the pulse-echo signal.

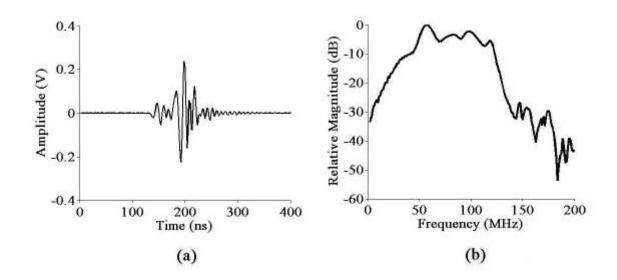


Figure 2.8: (a) Pulse-echo signal from the 80 MHz transducer (b) Spectrum of the pulse-echo signal.

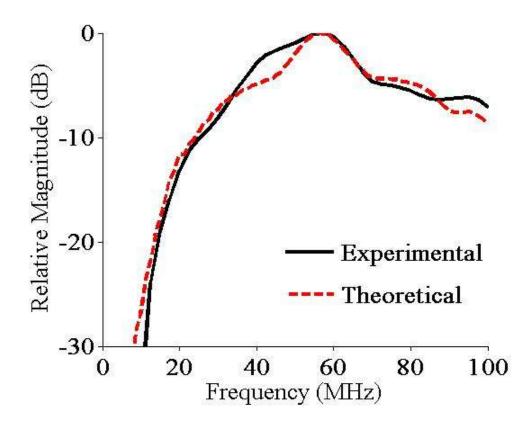


Figure 2.9: Experimental spectrum from the 11 μ m thick black PDMS film (black solid curve) and the theoretical curve (red dashed curve).

The theoretical spectrum of the detected ultrasound signal can be derived by taking the time derivative of the laser pulse, multiplying the spectrum of this pulse with the square root of the pulse echo response of the transducer, then taking into account the attenuation in water (0.0022dB/cm/MHz²[89]). The calculated theoretical spectrum curve, together with the experimental spectrum from the 11 µm black PDMS film, is shown in figure 2.9. The two curves are in good agreement with each other, indicating that the temporal profile of the generated ultrasound is dominantly determined by the input laser pulse. Therefore, the center frequency and the bandwidth of the generated ultrasound can be further improved with shorter laser pulses. Most importantly, the temporal response of the 11 µm thick black PDMS film is short enough to generate ultrasound at frequencies above 50 MHz, and this film closely approaches an ideal thermoelastic source. When the laser pulse duration is reduced to roughly the same as the heat transmission time, the simple laser pulse derivative relationship will no longer hold for the generated ultrasound waves. Heat transition from each individual carbon black particle to PDMS is about 2 ns, and that would be the limiting factor for the highest frequency possible using the thermoelastic effect in this device.

2.3 Surface acoustic pressure and transduction efficiency

The acoustic pressure at the surface of the film and the optoacoustic transduction efficiency are important indicators of the performance of optoacoustic transmitters. The surface acoustic pressure at the moment of generation can be calculated based on the value of the far field acoustic pressure using standard Rayleigh-Sommerfeld diffraction formulas [90-91]:

$$P_{far}(\vec{r}',t) = \iint_{\Sigma} \frac{1}{2\pi c |\vec{r}' - \vec{r}|} \frac{dP_{near}(\vec{r},t - \frac{|\vec{r}' - \vec{r}|}{c})}{dt} dS$$
(2.8)

Here *c* is the acoustic velocity, \vec{r}' is in the far field, \vec{r} is at the surface of the optoacoustic transmission element, P_{far} is the far field acoustic pressure, and P_{near} is the surface acoustic pressure. Assuming that the element size is much smaller than the distance, which is normally the case, the above formula can be rewritten as:

$$P_{far}(t) = \frac{S}{2\pi cr} \frac{dP_{near}(t - \frac{r}{c})}{dt}$$
(2.9)

On the other hand, we have previously shown that [2.6]:

$$P_{far}(t) = \frac{1}{4\pi} \frac{3B^2 \alpha_L}{\rho^2 c^2 C} \frac{1}{r} \frac{dI(t - \frac{r}{c})}{dt}$$
(2.10)

Here *I* is the total optical energy incident onto the film, ρ is the density, *C* is the specific heat capacity, *B* is the bulk modulus, and α_L is the linear coefficient of thermal expansion. Comparing the above two equations, it is not difficult to show that P_{near} is proportional to the total optical energy, and thus should follow the formula of the incident laser pulse:

$$P_{near}(t) = A_{near} \exp(-\frac{4\ln 2 \cdot t^2}{\tau^2})$$
(2.11)

Here A_{near} is the amplitude of the surface acoustic pressure, and τ is the laser pulse duration. Therefore, substituting (2.11) into (2.9), the far field pressure is:

$$P_{far}(t) = -\frac{8\ln 2}{\tau^2} \frac{S}{2\pi cr} A_{near} \left[(t - \frac{r}{c}) \exp(-\frac{4\ln 2(t - \frac{r}{c})^2}{\tau^2}) \right]$$
(2.12)

Thus, the amplitude of the far field acoustic field A_{far} is:

$$A_{far} = \frac{8\ln 2}{\tau^2} \frac{S}{2\pi cr} A_{near} \cdot \max[(t - \frac{r}{c})\exp(-\frac{4\ln 2 \cdot (t - \frac{r}{c})^2}{\tau^2})]$$
(2.13)

From this, we conclude the following:

$$A_{far} = \sqrt{\frac{8\ln 2}{e}} \frac{S}{2\pi c r \tau} A_{near}$$
(2.14)

Consequently, A_{near} can be expressed as:

$$A_{near} = \sqrt{\frac{e}{8\ln 2}} \frac{2\pi cr\tau}{S} A_{far}$$
(2.15)

The surface acoustic pressure can also be written in the form of the incident optical energy by substituting (2.15) into (2.10):

$$P_{near} = \frac{3B^2}{2\rho^2} \frac{\alpha_L \tau}{cC} \frac{1}{S} \frac{dI(t)}{dt}$$
(2.16)

Therefore, the surface acoustic pressure is proportional to the incident optical fluence, while the far field acoustic pressure is proportional to the optical power. Knowing the value of the far field acoustic pressure at a specific distance, the surface pressure can be estimated using (2.15). In our current experiment, the transmission element diameter is 25 μ m, the laser pulse duration is 5 ns, and the far field acoustic pressure is 800 kPa at a distance of 10 mm away from the film, when the incident optical energy is 50 μ J/pulse. This yields a surface acoustic pressure of 500 MPa. When the incident optical power reaches the thermal damage threshold of 150 μ J/pulse delivered to a spot size of 25 μ m,

the corresponding surface pressure could reach as high as 1500 MPa. High surface pressures at this level enable good SNR and high-quality imaging even though the transmitter emits a far-field, diverging radiation pattern where intensity falls off as the inverse square of distance.

The optoacoustic transduction efficiency η can be conveniently calculated using the surface pressure:

$$\eta = \frac{E_{acoustic}}{E_{in}} = \frac{S\int \frac{P_{near}^2(t)}{Z} dt}{E_{in}} = \sqrt{\frac{\pi}{8\ln 2}} \frac{S\tau A_{near}^2}{ZE_{in}}$$
(2.17)

Here E_{in} is the input optical energy, and Z is the acoustic impedance. In our case, when the incident optical energy is 50 µJ/pulse, the transduction efficiency is about 0.6 %. Note that because A_{near} is proportional to E_{in} , and is inversely proportional to S, it's easy to conclude that the transduction efficiency is proportional to E_{in}/S . Therefore, the efficiency can be greatly enhanced by increasing input optical energy and reducing the element spot size. Clearly, when the incident optical power reaches the thermal damage threshold of 150 µJ/pulse delivered to a spot size of 25 µm, the corresponding transduction efficiency could reach as high as 1.8 %.

2.4 Gold nanostructure film

With black PDMS films, the transduction efficiency and the bandwidth of the generated ultrasound are mainly limited by the thickness of the acoustically absorbing PDMS film. The optical absorption depth of the black PDMS film is estimated to be 1 μ m, therefore only a 1 μ m layer from the substrate contributes to the thermoelastic effect.

For the new 11 μ m thick film, there is still a 10 μ m thick layer that attenuates the generated ultrasound by roughly 1 dB / μ m. Clearly, reduced film thickness is highly desired to further improve transduction efficiency. However, with carbon black as the optical absorber, it is extremely difficult to reduce the thickness below 10 μ m because of the increased viscosity of the mixture. Such high viscosity prevents the polymer mixture to be spin cast into thinner films. This motivates us to consider other absorbing structures to replace carbon black in the PDMS film. A novel 2D gold nanostructure has been developed for this purpose.

The gold nanostructure consists of a 2D gold nanoparticle array sandwiched between a transparent substrate and a 4.5 µm thick PDMS layer. Each gold nanoparticle is 20 nm thick, and 128 nm by 110 nm in cross section. Gold nanoparticles serve as efficient optical absorbers because surface plasmons localized around the particles strongly absorb light at a resonant wavelength that depends on the size, shape, and local dielectric environment of the gold nanoparticles [92-103]. When a pulsed laser beam is focused onto the gold nanostructure layer through the glass substrate at the resonant frequency, a large fraction of incident energy is absorbed. Energy transferred over a very short time interval from the absorbing layer to PDMS rapidly heats a localized volume, in which an impulsive thermal expansion launches an acoustic pulse into the overlying sample. Because no carbon black is added, the PDMS layer is transparent and does not attenuate the generated ultrasound.

The first and key step is to fabricate SiO_2 stamps, and the process flow is illustrated in figure 2.10. A 200 nm thick SiO_2 layer is thermally deposited on a Si wafer, and a 10 nm chrome (Cr) layer is deposited on top of the SiO_2 layer using radiofrequency

(RF) sputtering. The Cr layer serves as an etch mask to the SiO₂ layer and is also used to improve adhesion between photoresist and the SiO₂. A 90 nm thick photoresist layer is then spin-coated on the Cr layer. Laser interference lithography (LIL) [104-106] is performed to form a grating pattern with a 220-nm pitch on the photoresist layer. The Cr layer is etched with Cl₂ and O₂ reactive ion etching (RIE), and then the SiO₂ layer is etched with CHF₃ RIE. After etching the SiO₂, the Cr mask is removed by Cr wet etchant. The final SiO₂ stamp consists of a 1D 200 nm thick SiO₂ pattern with period of 220 nm on top of a Si wafer. These stamps determine the pattern of the ultimately desired gold nanostructure.

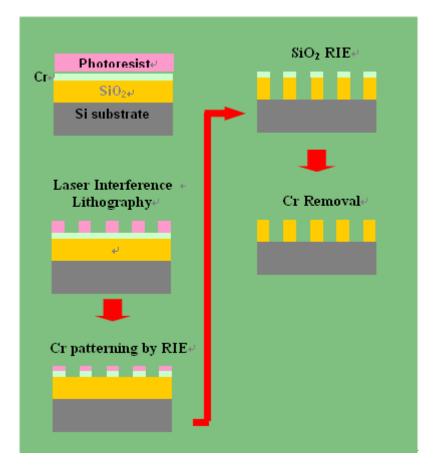


Figure 2.10: Process flow for fabricating SiO₂ stamps on a Si wafer.

A schematic of laser interference lithography is illustrated in figure 2.11. A He-Cd laser with a wavelength of 325 nm and power of 50 mW is used. The periodic line grating patterns are obtained through the laser interference between the direct beam and the reflected beam from the mirror. The period of the line patterns can be adjusted simply by changing the angle of the stage. A longer distance between the laser and the wafer makes it possible to form the desired pattern over a large area.

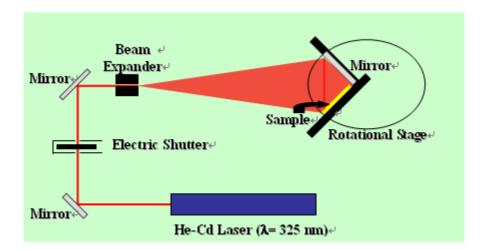


Figure 2.11: Scheme of using later interference lithography to fabricate 1D SiO_2 stamps on a Si wafer.

The next major step is to fabricate a 2D mold using the 1D SiO_2 stamp produced in the previous step. The mold consists of a 2D array of 200 nm thick SiO_2 nanosquare pillars with period of 220 nm on a Si substrate. Nanoimprint lithography [107-111] with an A90A scheme is employed because of its convenience and cost-effectiveness. This process is demonstrated in figure 2.12.

A 200nm thick SiO_2 film is first deposited on a Si wafer, and then a thin 10nm film of Cr is deposited using an electron beam evaporation system. This Cr film serves

as a mask layer. Plasma etching recipes with high-selectivity of SiO_2 over Cr and Si enable Si to behave as an etch stop. Before imprinting, the fabricated grating stamp with 220nm period is pretreated by an anti-sticking layer (1H, 1H, 2H, 2H Perfluorodecyl trichlorosilane) to prevent the stamp from adhering to the imprint pattern during the demolding step.

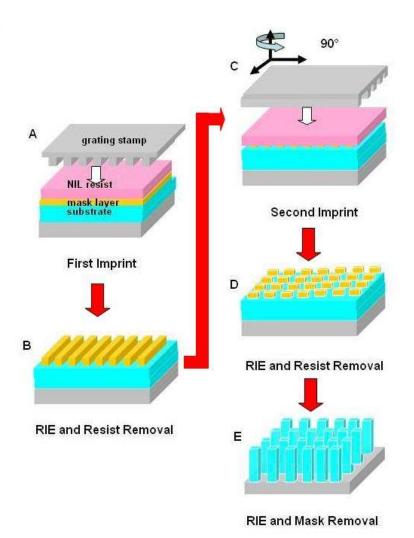


Figure 2.12: Process of fabricating a 2D SiO_2 nanosquare mold using the 1D SiO_2 grating stamps.

The nanoimprint resist is spin coated into a 140nm thick film, baked on a hotplate to remove residual solvent and imprinted in a custom-built nanoimprinter. RIE is used in a two-step manner to remove the residual polymer layer and Cr patterning. The nanoimprint resist is then dissolved in acetone and the sample is rinsed using methanol and IPA and dried in N_2 . The same process is repeated for the second imprint with the grating rotated 90°. SiO₂ is reactive-ion etched to its full depth of 200nm and then the Cr layer is removed using a wet etchant.

Now the desired gold nanostructure is fabricated using the mold consisting of a 2D array of SiO₂ pillars with thickness of 200 nm and period of 220 nm. Note that each pillar is 128 nm by 110 nm in cross section. It is then imprinted on a 200 nm thick photoresist (mr-I 8020, Micro Resist Technology, Berlin, Germany) layer, which is spin-coated onto a glass substrate. After separation, a 200 nm thick photoresist layer with 2D arrangements of air holes spaced every 220 nm is obtained, where each air hole is 200 nm deep and 128 nm by 110 nm in cross section.

A 20 nm layer of gold is then deposited on top of the structure using an electron beam evaporator, not only leaving a 2D array of gold nanoparticles at the bottom of the air holes, but also covering the top of the photoresist layer. This structure is called the non-liftoff gold nanostructure, which is an important mid-product and will be used in chapter 4. Figure 2.13 (a) shows a scanning electron microscope (SEM) picture of the top view of this nanostructure, and figure 2.13 (b) shows a sketch of the side view.

Liftoff is then performed by soaking the sample in acetone within a low-power ultrasonic bath, removing the polymer layer, as well as the overlying gold layer. The sample is rinsed with methanol and IPA, then dried with N₂. A 2D array of 20 nm thick gold nanoparticles with period of 220 nm remains on the glass substrate. Each gold nanoparticle is 128 nm by 110 nm in cross section. The structure obtained is called the liftoff gold nanostructure, and will be used for ultrasound generation experiments. A scanning electron microscope (SEM) picture of the top view of the structure is shown in figure 2.14 (a), and a sketch of the side view is shown in figure 2.14 (b). A mixture of PDMS and toluene, with a ratio of 2:1, is then spin coated into a 4.5 µm thick layer on top of the gold nanostructure layer.

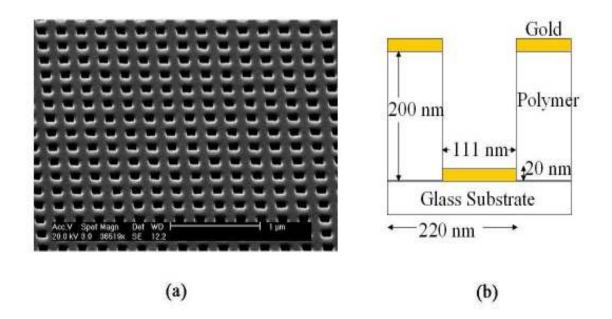


Figure 2.13: SEM picture of the top view (a) and sketch of the side view (b) of the non-liftoff gold nanostructure.

Optical transmission measurements were done using a Nikon TE300 Eclipse inverted microscope (20x objective; NA=0.44) with transmitted light coupled into an Ocean Optics SD2000 fiber coupled spectrometer using an achromatic lens. As illustrated in figure 2.15, transmission through the liftoff structure varies with wavelength.

Maximum absorption (minimum transmission) occurs at 675 nm, and there is a factor of 3 greater transmission at 980 nm compared to 675 nm.

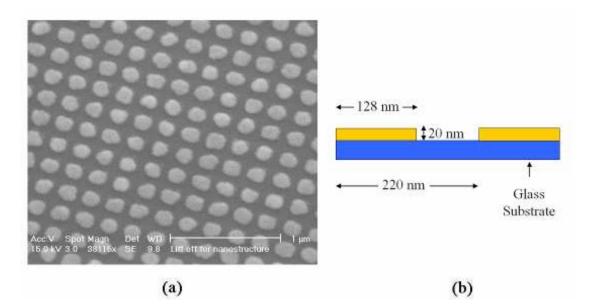


Figure 2.14: SEM picture of the top view (a) and sketch of the side view (b) of the liftoff gold nanostructure.

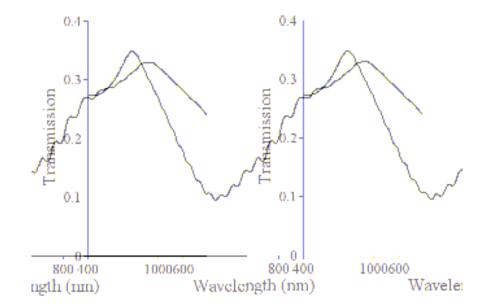


Figure 2.15: Optical transmission vs. wavelength.

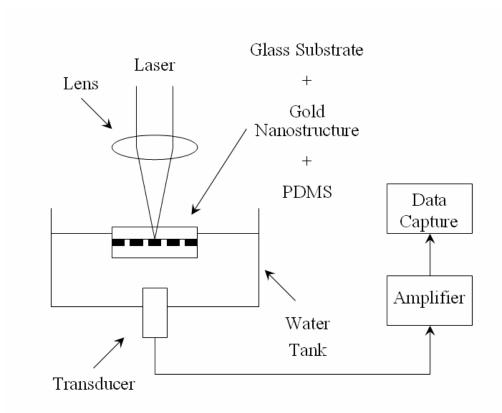


Figure 2.16: Experiment setup for thermoelastic generation of ultrasound using the gold nanostructure film.

The experimental setup for thermoelastic generation of ultrasound using the gold nanostructure film is shown in figure 2.16. The gold nanostructure film is mounted on a sample holder at the water surface. The laser excitation source is a commercial high energy solid state laser with tunable wavelength (Surelite, with OPO Plus, Continuum, Inc.), which produces a 5 ns laser pulse with energy of 100 mJ. The beam is coupled into a multimode fiber with core size of 200 μ m and output through a collimator; this reduces the pulse energy to about 100 μ J. Before the pulse is focused onto the film, it goes through a set of ND filters (OD=3.0), further reducing the energy to about 100 nJ. It is then focused onto the gold nanostructure film through the glass slide with a 3.1 mm focal

length aspherical lens. A f/1.4, LiNbO₃ transducer with a focal length of 4.1 mm and a center frequency of 80 MHz records the acoustic signal at a distance 4.1 mm from the absorbing film (i.e., at the focal length of the acoustic lens). The detected signal is amplified by 30dB before data capture.

Figure 2.17 (a) shows the detected signal from the gold nanostructure film, averaged 1000 times, for optical excitation at 700 nm. In comparison, the detected signal from the 11 µm black PDMS film, also averaged 1000 times, is shown in figure 2.17 (b). Although the gold nanostructure film is not able to absorb 100 % of the laser energy as the black PDMS film does, the acoustic signal from the gold nanostructure film is still larger than that from the black PDMS film. We attribute this to the reduced thickness of the gold nanostructure film, which means less attenuation to generated acoustic waves.

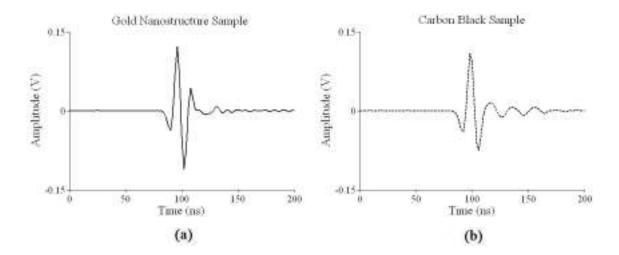


Figure 2.17: Signal from (a) 4.5 μ m film with a single layer of gold nanoparticles and (b) 11 μ m black PDMS film.

Corresponding spectra for the two films are shown in figure 2.18. The reduced thickness has an even bigger impact on the signal spectrum. As can be observed from figure 2.18, the spectrum from the gold nanostructure film shows significant

improvement at frequencies above 50 MHz. This also reconfirms that the higher frequency components are attenuated more in the PDMS layer overlying the acoustic source. The heat transition time from the gold nanoparticles to PDMS is about 100 femtoseconds, which is much shorter than the pulse duration. Fast heat transfer like this will generate ultrasound with higher frequency components.

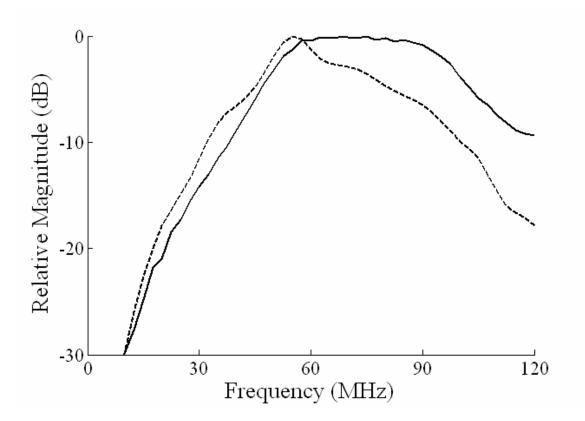


Figure 2.18: Spectrum comparison of the gold nanostructure film (solid curve) and the black PDMS film (dashed curve).

Because the absorption of the gold nanostructure film depends on the incident laser wavelength, and the amplitude of the acoustic signal should be a linear function of the absorbed energy, we expect the output amplitude to change with wavelength similar to the optical absorption. With our laser system, we were able to change the incident wavelength from 675 nm to 1000 nm, and measure the acoustic amplitude at different wavelengths. Figure 2.19 shows how the output changes with incident wavelength, for both the gold nanostructure film and the black PDMS film. Note that the laser energy at different wavelengths is different, so we normalized the output to an incident energy of 100 nJ. For the black PDMS film, the output amplitude does not change much with wavelength, as is expected because the film is 100% absorbing over this whole wavelength range. For the gold nanostructure film, the maximum output is located near 700 nm, while the minimum output is near 1000 nm. The corresponding amplitudes have a ratio of 7.5:1. The maximum and minimum locations agree fairly well with the optical transmission curve, but the extinction ratio is larger than expected. This mismatch can be corrected if optical reflection from the gold nanoparticle layer is taken into account.

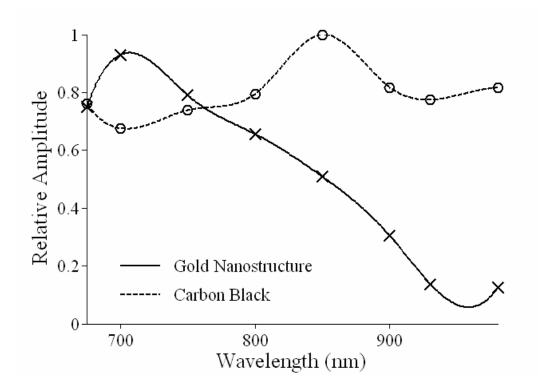


Figure 2.19: Acoustic signal vs. wavelength from the gold nanostructure film (dashed) and the black PDMS film (dashed).

We have previously measured the acoustic pressure from the black PDMS film, and we can estimate the acoustic pressure for the gold nanostructure film based on amplitude comparisons. When a 5ns pulse with optical power of 100 nJ is delivered to a spot size of 25 μ m, the acoustic pressure generated using the gold nanostructure film is about 2 kPa at a distance of 10 mm, representing about 1.5 MPa at the film surface. The acoustic pressure will increase linearly with input laser energy, and the ultimate pressure possible is determined by the thermal damage threshold of the film, which reaches an optical power of 25 μ J delivered to a spot size of 25 μ m, representing a fluence of 4 J/cm². At this light level, the acoustic pressure can reach 500 kPa at a distance of 10 mm, and 375 MPa at the surface, which should be sufficient for real-time ultrasound imaging.

The high extinction ratio of the gold nanostructure film provides a possible method to integrate it with optoacoustic etalon detection arrays for ultrasonic imaging. The etalon typically requires a separate laser beam from the one used for ultrasound generation, where the detection wavelength is tuned to the minimum absorption wavelength of the gold nanostructure film. Because of the high extinction ratio of the gold nanostructure film, most of the laser energy at the maximum absorption wavelength is transduced into ultrasound, whereas most of the laser energy at the minimum absorption is transmitted for ultrasound detection. An ideal element would absorb 100% of the generation beam and transmit 100% of the detection beam. A structure with optical absorption close to the ideal might be achieved by optimizing the gold nanoparticles' size, spacing and shape.

2.5 Simulation of optical absorbance

Further understanding of the optical absorbance of the gold nanostructure is desired to maximize the performance of the device. Extensive efforts have been investigated in theoretical studies of nano-scale noble metal particle arrays by numerous researchers [112-115]. Because of the complexity in the physics of gold nanoparticle arrays, this section will focus on simulations of an individual gold nanoparticle.

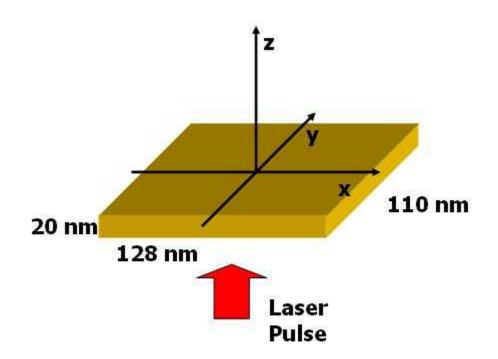


Figure 2.20: Geometry of an individual gold nanoparticle.

As shown in figure 2.20, each gold nanoparticle is 20 nm thick, and is 128 nm by 110 nm in cross section. The direction of the laser pulse is perpendicular to the cross section of the nanoparticle. The absorption coefficient can be calculated using Mie scattering theory, which yields the flowing formula [96, 115]:

$$C_{abs}(\lambda) = \frac{2\pi}{\lambda} \frac{\operatorname{Im}((\varepsilon_r - 1) \oiint \vec{E} dV)}{\sigma_{in}}$$
(2.18)

Here λ is the wavelength, ε_r is the relative dielectric constant of gold which is dependent on the wavelength [116], σ_{in} is the cross section of the nanoparticle, and $\iiint \vec{E} dV$ is the volume integration of the electric field within the nanoparticle when a unit external electric field is applied.

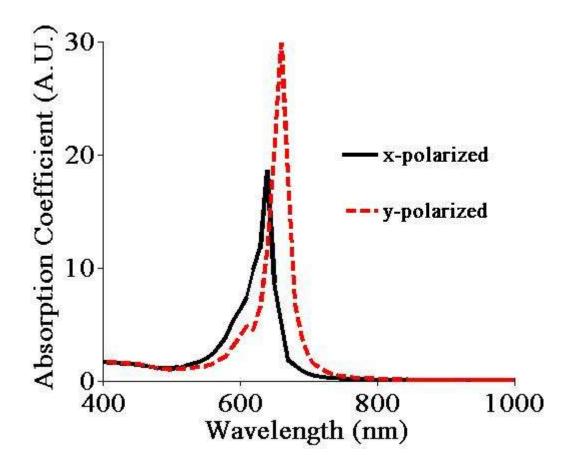


Figure 2.21: Optical absorption coefficient of a single gold nanoparticle when the incident laser pulse is (a) x-polarized (b) y-polarized.

FEMLAB's electro-static module is utilized for simulation. At each wavelength, the program uses the preconfigured geometry and the corresponding dielectric constant of gold, and calculates the electric field distribution given a unit external electric field. Two cases are calculated, one is when the external electric field is along the x direction, which corresponds to x-polarized incident laser pulse, while the other is when the external electric field is along the y direction, which corresponds to y-polarized incident laser pulse. Then the volume integration of the electric field within the gold nanoparticle is done automatically with FEMLAB's internal functions. Figure 2.21 shows how the optical absorption coefficient changes with wavelength for both x-polarized and y-polarized incident pulses. Clearly, the resonant wavelength is between 600 nm and 700 nm, consistent with experimental results. The FWHM of the resonance in the simulation results is about 45 nm, much smaller than the experimental value of roughly 200 nm. The reason is that there are several linewidth broadening mechanisms that were not taken into account in these simulations.

The first broadening mechanism is called electron surface scattering. In bulk gold, the electron collision frequency is determined by the Fermi velocity. However, when the dimension of the gold particle is reduced to nanometer level, size-dependent electron surface scattering becomes important and therefore the experimentally measured dielectric coefficient of bulk gold must be modified for gold nanoparticles. Even though several models [117], both classical and quantum mechanical, have been developed, precise understanding of electron surface scattering remains difficult. In addition, the distribution of nanoparticle sizes also leads to inhomogeneous broadening of the absorption resonance. Clearly, the experimentally measured curve is the collective

behaviors of numerous gold nanoparticles and these nanoparticles differ vastly in their shapes and sizes, as supported by figure 2.14 (a). In our case, it is expected that the size distribution is the dominant contribution to the observed linewidth.

In-depth simulations and analysis still need to be conducted on the optical absorption of gold nanoparticles and arrays. The preliminary results in this section demonstrate that Mie scattering theory is a good approximation in terms of determining the resonance wavelength.

2.6. Comparison between gold nanostructure and black PDMS films

Two structures, gold nanostructure films and black PDMS films, have been developed for optical generation of high-frequency ultrasound. They both utilize the thermoelastic effect for optoacoustic transduction, but employ different optical absorbers. A 2D array of gold nanoparticles is used in the gold nanostructure film, while carbon black particles dispersed into PDMS are used in the black PDMS films. This has led to many differences in properties and performance of the two structures, as summarized here.

- a. Thickness. The gold nanostructure film is about 4.5 μm thick, and the black PDMS film is 11 μm thick. The gold nanostructure film is thinner because no carbon black is added to PDMS, which significantly lowers the viscosity and thus reduces thickness.
- b. Transduction efficiency. The transduction efficiency of the gold nanostructure film is about 20 % higher than the black PDMS film. At stated in previous texts, the black PDMS film attenuates generated ultrasound by about 1 dB/ μ m.

Therefore, a transparent PDMS layer can significantly improve optoacoustic transduction efficiency. Meanwhile, the gold nanostructure can't absorb 100 % of the optical energy like the black PDMS film, thereby limiting the overall conversion efficiency.

- c. Bandwidth. The bandwidth of the gold nanostructure film is clearly better than that of the black PDMS film. For example, with a 5 ns laser pulse, the black PDMS film produces a center frequency of 60 MHz with -6 dB bandwidth of 48 MHz, while the gold nanostructure film generates a flat spectrum from 60-90 MHz with -6 dB bandwidth of 60 MHz. Again, this originates from the fact that the black PDMS film attenuates the generated ultrasound, especially high frequency components, while transparent PDMS does not.
- d. Thermal damage threshold. The thermal damage threshold of the black PDMS film reaches an optical fluence of 24 J/cm², 6 times higher than that of the gold nanostructure film. The most important reason is that the optical absorbing ability of each individual gold nanoparticle is much higher than a carbon black particle. Thus, with the same incident optical energy, the gold nanoparticle is more likely to be damaged. Note that the largest surface acoustic pressure available is 1500 MPa and 375 MPa for black PDMS and gold nanostructure films respectively.
- e. Integration capabilities with optoacoustic detectors. The gold nanostructure holds an edge in its abilities to be combined with optoacoustic detectors, because it has a high extinction ratio and allows laser beams with wavelengths far from resonance to penetrate through the structure. On the other hand, black PDMS films are absorbing over the whole optical spectrum, and thus do not allow the co-

existence of two separate laser beams. Therefore, with black PDMS films, optoacoustic generation and detection elements must be geometrically separate, while integrated single elements are possible with the gold nanostructure.

In summary, both structures can produce broad bandwidth and sufficient acoustic power for high-resolution ultrasound imaging. The black PDMS film is suitable for applications where extremely high acoustic pressure is desired, and the gold nanostructure film holds the edge when a fully-sampled array geometry is required.

2.7 Other structures

Other than the gold nanostructure and black PDMS films, optoacoustic transmitters utilizing different optical absorbers have been developed and tested. Even though they produced poor transduction efficiencies, it is beneficial to discuss their performance to further understand the thermoelastic effect and identify technical challenges.

As the first alternative, black polyimide is used instead of a mixture of PDMS and carbon black, and is spin cast into a 1 μ m thick film on top of a glass substrate. This sample is tested with a 10 ns laser pulse using the experimental setup shown in figure 2.1, and is compared to the 11 μ m black PDMS sample. The signals from the black PDMS film and the black polyimide film, both averaged 1000 times, are shown in figure 2.22 (a) and (b) respectively. The transduction efficiency of the black PDMS film is 31 dB higher than that of the black polyimide film. The main reason is that the thermal expansion coefficient of black polyimide is much lower than PDMS. Even though the black

polyimide film is much thinner, with much less acoustic attenuation, it is unable to make up for the huge difference in thermal expansion coefficient.

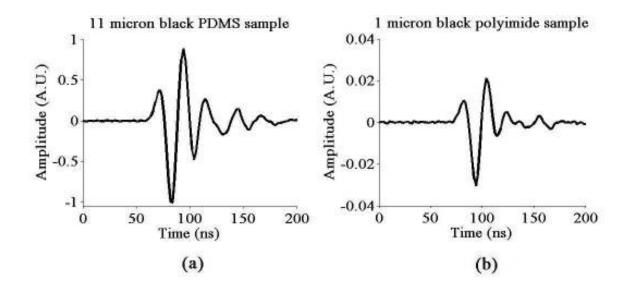


Figure 2.22: With a 10 ns laser pulse, the signal from (a) the 11 μ m black PDMS film and (b) the 1 μ m black polyimide film.

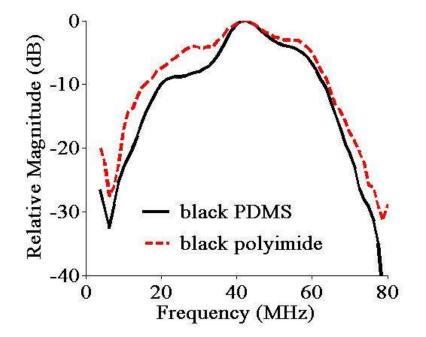


Figure 2.23: Spectrum comparison of the 11 μ m black PDMS film (black solid curve) and the 1 μ m black polyimide film (red dashed curve) with a 10 ns laser pulse.

Corresponding spectra (each has been normalized to its own peak) for the two films are shown in figure 2.23. Clearly, the black polyimide film has a broader bandwidth, due to its much reduced thickness. This again verifies the claim that black films attenuate the generated ultrasound, especially the higher frequency components.

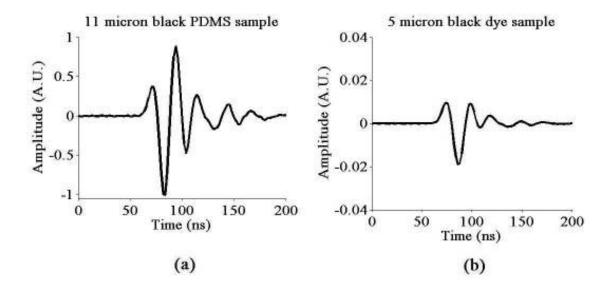


Figure 2.24: With a 10 ns laser pulse, the signal from (a) the 11 μ m black PDMS film and (b) the 5 μ m black dye film.

The second alternative utilizes the mixture of PDMS and black dye (black 220, Gayson, Inc., Barberton, OH, USA). Unlike carbon black, black dye is extremely easy to disperse into PDMS, and does not increase the viscosity of the mixture. PDMS, black dye, and toluene are mixed at a ratio of 1:1:0.5 and the mixture is spin cast into a 5 μ m film. The main drawback of using black dye is that its optical absorption is much lower than carbon black, and as a result, this 5 μ m film is only capable of absorbing 75 % of the incident laser energy. This sample is also tested with a 10 ns laser pulse using the experimental setup shown in figure 2.1, and is compared to the 11 μ m black PDMS

sample. The signals from the black PDMS film and the black dye film, both averaged 1000 times, are shown in figure 2.24 (a) and (b) respectively. The transduction efficiency of the black PDMS film is 36 dB higher than that of the black dye film. This is attributed to the fact that the film consists of only 40 % PDMS. When PDMS is mixed with an equal amount of black dye, its unique chemical structure, where its high thermal expansion coefficient originates from, is destroyed. Therefore, the thermal expansion coefficient of the film is significantly reduced, causing the transduction efficiency to be more than 30 dB lower than black PDMS films. The concentration of PDMS can be increased by reducing the amount of black dye, which is not desired because it will further sacrifice the optical absorbing capabilities of the film. Corresponding spectra (each has been normalized to its own peak) for the two films are shown in figure 2.25. Again, the thinner film has a broader bandwidth.

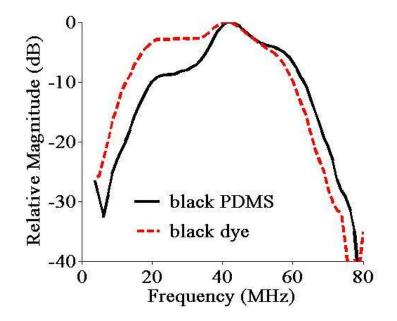


Figure 2.25: Spectrum comparison of the 11 μ m black PDMS film (black solid curve) and the 5 μ m black polyimide film (red dashed curve) with a 10 ns laser pulse.

2.8 Summary of optoacoustic transmitters

In summary, 11 µm thick black PDMS films have been fabricated for thermoelastic generation of high frequency ultrasound with improved fabrication techniques. Meanwhile, 5 µm thick PDMS films with a 2D gold nanostructure as optical absorber have also been developed using nanoimprint lithography. Both devices improve the conversion efficiency of the thermoelastic effect by about 11dB compared to the 25 µm thick black PDMS films fabricated previously. We have also demonstrated that the temporal responses of both structures are short enough to generate ultrasound at frequencies above 50 MHz. The surface acoustic pressure could reach over 500 MPa with the black PDMS film and over 100 MPa for the gold nanostructure film for optical powers at least a factor of three below the threshold for thermal damage to the films, respectively. The spectrum of the generated ultrasound is shown to be mainly determined by the temporal profile of the incident laser pulse, meaning that both the center frequency and the bandwidth have great potential to be improved if an appropriate laser source is used. These results strongly suggest that the thermoelastic effect in black PDMS and gold nanostructure films is a practical alternative to piezoelectricity for fabricating arrays in the 100 MHz regime.

CHAPTER 3

OPTOACOUSTIC DETECTORS - ETALONS

3.1 Optimization of thin polymer etalons

One of the simplest and most effective methods for optical detection of ultrasound utilizes a polymer etalon, which represents the best balance between transduction efficiency and system simplicity. The etalon is a polymer Fabry-Perot interferometer structure, which consists of a transparent polymer slab sandwiched between two partially reflecting mirrors. The basic ultrasound detection principle is well documented [66-80, 90], and has been briefly introduced in chapter 1. Etalons have been widely used as ultrasound hydrophones [73], array elements for photoacoustic imaging [80], and the receiving end of all-optical ultrasound transducers [85].

Up to now, most studies involving etalons have been conducted in the frequency range of 1 to 40 MHz [73-80], where conventional piezoelectric techniques and surging CMUT devices are mature and virtually irreplaceable. The current state of the art is an 11 μ m thick SU-8 etalon with a bandwidth of 40 MHz [80]. Etalons with bandwidths over 50 MHz will significantly expand the frontiers of high-frequency photoacoustic and ultrasound imaging. Using a polymer material as the etalon bulk layer, broader bandwidth can be achieved by reducing etalon thickness [74]. In this chapter, the development and characterization of 5.9 μ m thick high-frequency (>50 MHz) etalon detection arrays are

presented. These etalons are especially suitable for high-resolution ultrasound and photoacoustic imaging.

An optoacoustic etalon is fabricated by first depositing a 30 nm gold layer on top of a glass substrate using an electron beam evaporator. This gold layer will serve as the first optical reflector. Then, SU-8 photoresist (SU-8 2005, Microchem Corp., Newton, MA, USA) is spin coated on top at 2100 rpm for 40 seconds. The cured film is about 5.9 μ m thick and forms the etalon's polymer bulk layer. Another 30 nm gold layer is then deposited and will be utilized as the second reflecting mirror. As a final step, an additional 2 μ m thick SU-8 layer is spin cast over the entire device for protection. A sketch of the side view of the entire etalon structure is shown in figure 3.1 (a), and a photo in figure 3.1 (b).

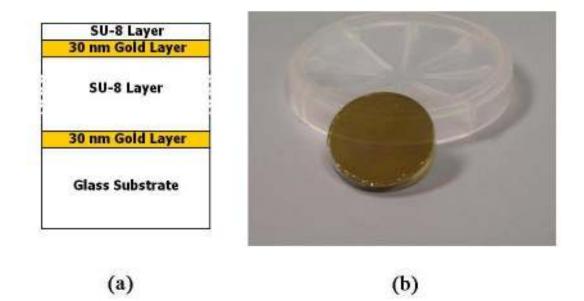


Figure 3.1: (a) A sketch of the side view of the 5.9 μ m thick etalon structure; (b) a photo of the etalon.

The polymer layer thickness can be easily changed by tuning the speed of spin coating. Choosing the proper thickness is a matter of tradeoff between sensitivity and bandwidth, as will be discussed later. In this chapter, $5.9 \mu m$ thick SU-8 etalons are investigated unless otherwise specified.

3.2 Theoretical acoustic frequency response

The frequency response of the etalon can be calculated using the method developed by P. C. Beard and his group [74]. The approach calculates the mean distribution of stress across the thickness of the etalon due to an incident acoustic wave:

$$P(f) = \frac{1}{l} \int_{l} P(f, x) dx$$
(3.1)

Here f represents the acoustic frequency, and P(f,x) is the sum of the incident acoustic wave and all subsequent reflections due to acoustic impedance mismatches at the boundaries.

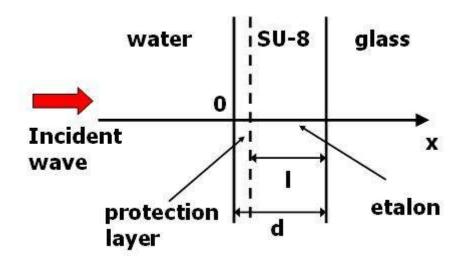


Figure 3.2: Geometry for calculating the etalon acoustic frequency response.

The geometry is shown in figure 3.2, and the incident wave is represented by $A \exp[i(\omega t - kx)]$, where $\omega = 2\pi f$ and $k = \frac{2\pi f}{c}$. The SU-8 layer consists of both the etalon bulk layer and the protection layer, and is sandwiched between water and glass. The etalon thickness and the overall SU-8 thickness are denoted as *l* and *d* respectively. Note that the gold layers are too thin to have an impact on the acoustic properties of the structure and therefore are neglected. The following can be easily derived:

$$P(f,x) = TA \exp(i\omega t) \frac{\exp(-ikx) + R_1 \exp(ikx) \exp(-ik2d)}{1 - R_1 R_0 \exp(-ik2d)}$$
(3.2)

Here *T* is the acoustic transmission coefficient from water to SU-8, R_0 is the acoustic reflection coefficient between SU-8 and water, and R_1 is between SU-8 and glass. They can be calculated as the following:

$$T = \frac{2Z_{SU8}}{Z_{SU8} + Z_{water}} = 1.32$$
(3.3)

$$R_0 = \frac{Z_{water} - Z_{SU8}}{Z_{water} + Z_{SU8}} = -0.32$$
(3.4)

$$R_1 = \frac{Z_{glass} - Z_{SU8}}{Z_{glass} + Z_{SU8}} = 0.67$$
(3.5)

Here $Z_{water} = 1.5 MRayl$, $Z_{glass} = 14.7 MRayl$, and $Z_{SU8} = 2.9 MRayl$ are acoustic impedances of water, glass, and SU-8 respectively. The following can be derived by

integrating (3.2) over the thickness of the etalon bulk:

$$P(f) = \frac{TA \exp[i(\omega t - kd)]}{ik[1 - R_0 R_1 \exp(-ik2d)]} [\exp(ikl) - 1 + R_1 - R_1 \exp(-ikl)]$$
(3.6)

Therefore, the acoustic frequency response of the etalon can be expressed as:

$$|P(f)| \propto \frac{1}{f} \frac{\left| (\exp(i\frac{2\pi f}{c}l) - 1) + R_1(1 - \exp(-i\frac{2\pi f}{c}l)) \right|}{\left| 1 - R_0 R_1 \exp(-i\frac{2\pi f}{c}2d) \right|}$$
(3.7)

Here *l* is the thickness of the etalon bulk layer, and *d* is the overall thickness of the etalon, including the protection layer. c = 2500m/s is the acoustic velocity in SU-8, and *f* is the acoustic frequency. The frequency responses of 5.9 µm and 11 µm SU-8 etalons with 2 µm thick protection layers are calculated and shown in figure 3.3. Apparently, thinner etalons produce broader bandwidth.

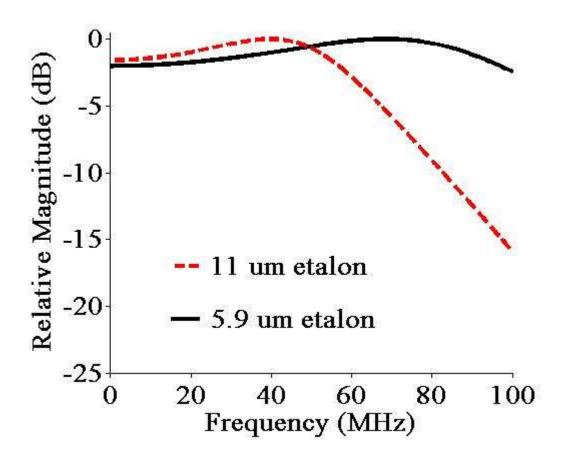


Figure 3.3: The theoretical acoustic frequency response of 5.9 μ m etalons (black solid curve) and 11 μ m etalons (red dashed curve).

3.3 Experimental characterizations

The optical resonance, acoustic frequency response, noise equivalent pressure, and acoustic angular response of the etalon must be characterized to understand device performance and determine its suitability as a high-frequency ultrasound array detector. The basic experimental setup is shown in figure 3.4.

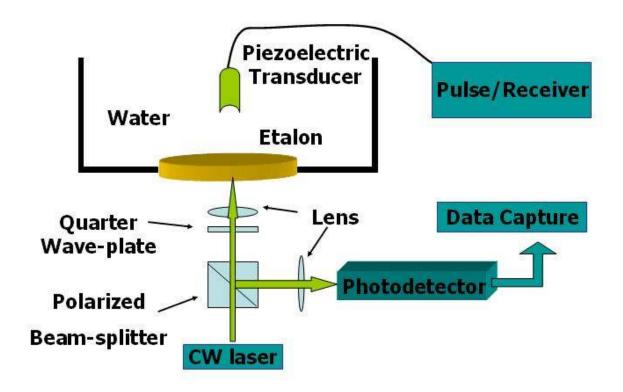


Figure 3.4: The block diagram of experimental setup to characterize the etalon.

A polarized and collimated continuous-wave (CW) laser beam with power of 4 mW and tunable wavelength from 1440 nm to 1590 nm (Agilent/HP 8168F, Agilent Technologies, Santa Clara, CA, USA) travels through a polarized beam-splitter and a quarter waveplate. It is then focused onto a 20 μ m spot on the surface of the etalon mounted at the bottom of a water tank. The 20 μ m laser focal spot defines a 20 μ m

ultrasound detection element size. The reflected beam's polarization after traveling through the quarter waveplate is perpendicular to that of the incident beam; therefore, it is reflected off the polarized beam-splitter and is then focused into an amplified InGaAs photodetector connected to the computer for data capture. A piezoelectric transducer is generally placed above the etalon as the source of ultrasound waves.

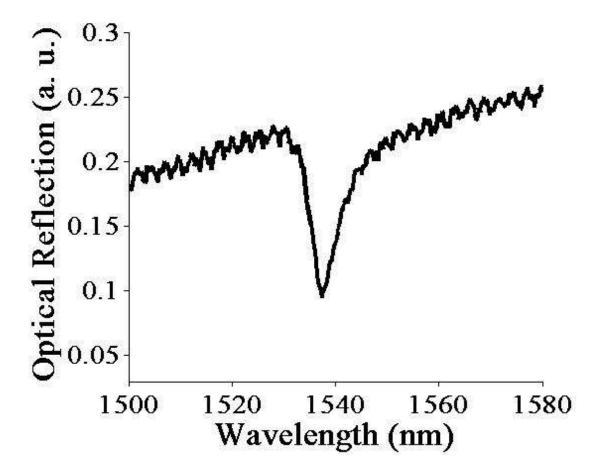


Figure 3.5: Optical resonance of the etalon structure.

The reflected optical intensity depends on the optical wavelength [90], and displays a sharp drop when the optical path of two-way travel in the etalon bulk equals an integer multiple of the wavelength. This is called the resonance condition and creates the

mechanism for ultrasound detection. The optical resonance can be measured by recording the reflected optical intensity when the wavelength of the probe CW laser is tuned. As is shown in figure 3.5, the resonance wavelength is 1538 nm with FWHM of 6.3 nm.

The quality factor of the etalon can be estimated to be [90]: $Q = \frac{2nl}{\lambda} \cdot \frac{\pi\sqrt{R}}{1-R} \approx 230$.

Here n = 1.57 is the refractive index of SU-8; $l = 5.9 \ \mu\text{m}$ is the thickness of the SU-8 layer; $\lambda = 1538 \ \text{nm}$ is the resonance wavelength; and R = 0.85 is the estimated optical reflection coefficient of the two gold layers. The theoretical FWHM of the resonance can be determined using: $\Delta \lambda_{1/2} = \frac{\lambda}{Q} \approx 6.6 \ \text{nm}$, in good agreement with experimental results. When this device is used for ultrasound detection, the wavelength of the probe laser is tuned to 1536.5 nm, the point of largest slope yielding the largest optical modulation when the resonance condition is changed by the acoustic pressure of the incident ultrasound waves.

The frequency response of the etalon was characterized with a 50 MHz piezoelectric transducer with aperture diameter of 2.5 mm and focal length of 4 mm (LiNbO₃, Resource Center for Medical Ultrasonic Transducer Technology, University of Southern California, CA, USA). First, a pulse-echo signal reflected from a glass substrate is recorded by the piezoelectric transducer. Then the transducer is placed a focal length above the etalon, as shown in the setup in figure 3.4, and the signal from the piezoelectric transducer is recorded from the etalon. An 11 μ m etalon is also used for comparison purposes. The spectrum of the transducer pulse-echo signal and spectra from both etalons are shown in figure 3.6. These results confirm that a thinner etalon has a higher frequency response and a broader bandwidth than a thicker one.

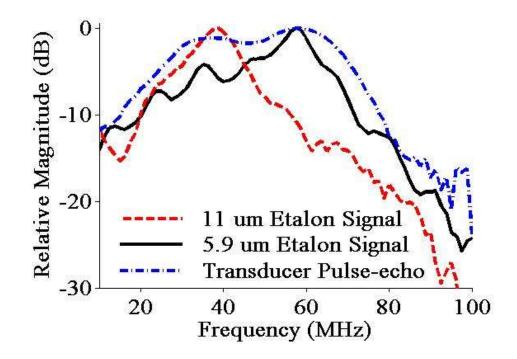


Figure 3.6: Spectra of the transducer pulse-echo signal, and signals from 11 μm and 5.9 μm etalons.

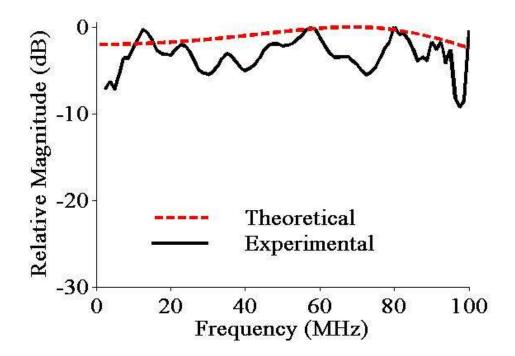


Figure 3.7: Experimental and theoretical acoustic frequency responses of a 5.9 μm etalon.

The frequency response of the etalon is derived by dividing the spectrum of the etalon signal by the square root of the spectrum of the transducer pulse echo. The derived frequency response of the 5.9 μ m thick etalon, together with the theoretical curve, is shown in figure 3.7. Clearly, the experimental frequency response is in good agreement with the theoretical model. These results suggest that the 5.9 μ m thick etalon is suitable for ultrasound detection at above 50 MHz.

The noise equivalent pressure of the current system can be measured by replacing the high frequency transducer in the setup of figure 3.4 with a calibrated 10 MHz transducer (V312, Panametrics NDT, Waltham, MA, USA). It has a diameter of 6.35 mm, focal length of 19 mm, and generates a negative peak pressure of about –2.6 MPa at focus when driven by a commercial pulser/receiver (5077PR, Panametrics NDT, Waltham, MA, USA). The etalon was put at the focal plane of the transducer. The system outputs a root mean squared noise of 6.4 mV over 25–75 MHz with a 32.5 dB gain amplifier and a peak signal of 101 mV without the amplifier. Therefore, the NEP within the specified 50 MHz bandwidth is estimated to be:

$$NEP = \frac{2600kPa}{101mV} \times \frac{6.4mV}{42} = 3.9kPa$$
(3.8)

For 20 μ m diameter optoacoustic detection element, NEP of 3.9 kPa over a 50 MHz bandwidth is at least as good as, if not significantly better than, polyvinylidene fluoride (PVDF) hydrophones of equivalent size. For example, a sensitivity of 6 nV/Pa over a bandwidth of 40 MHz was reported for a 40 μ m diameter PVDF needle hydrophone (HP 0.04 mm Interchangeable Probe, HPM04/1, Precision Acoustics LTD, Dorchester, UK) [118]. The output noise level of the pre-amplifier is 60 μ V, which yields

a NEP of 10 kPa for this 40 μ m PVDF hydrophone. Taking into account the difference in the effective element size and assuming that NEP is inversely linear with area, the NEP of a 20 μ m PVDF hydrophone should be 40 kPa, so the etalon is actually much more sensitive than a typical PVDF hydrophone of similar size.

The noise equivalent pressure of the etalon can be further improved. Theoretically, the sensitivity of the etalon can be derived by characterizing two separate aspects: how the reflected optical intensity changes with cavity length, and how cavity length changes with incident acoustic pressure. According to previous studies, the reflected optical intensity change is related to the cavity length change [79]:

$$\delta I_R = I_0 \frac{3\sqrt{3}}{2} \frac{Fn}{\lambda} \delta L = I_0 \frac{3\sqrt{3}}{4} Q \frac{\delta L}{L}$$
(3.9)

Here δI_R is the change in reflected optical intensity, I_0 is the optical intensity of the etalon probing beam, F is the finesse of the etalon, n is the refractive index of the etalon bulk material, λ is the wavelength of the etalon probing beam, δL is the change in cavity length, L is the cavity length, and Q is the quality factor of the etalon.

In simplest form, the change in cavity length induced by incident acoustic pressure is:

$$\delta L = \frac{L \cdot p}{E_{\gamma}} \tag{3.10}$$

Here p is the acoustic pressure, and E_y is the Young's modulus of the etalon bulk polymer material. Combining (3.9) and (3.10) yields:

$$\delta I_R = \frac{3\sqrt{3}}{4} I_0 \cdot Q \cdot \frac{1}{E_Y} \cdot p \tag{3.11}$$

Assuming that the optical detector sensitivity is I_s , the noise equivalent pressure

 p_{NEP} can be expressed as:

$$p_{NEP} = \frac{4}{3\sqrt{3}} \frac{I_s \cdot E_Y}{I_0 \cdot Q}$$
(3.12)

Therefore, the noise equivalent pressure of the etalon is proportional to the sensitivity of the photodetector and the Young's modulus of the etalon bulk material, and is inversely proportional to the intensity of the etalon probe beam and the quality factor of the etalon. The most straightforward methods to reduce the noise equivalent pressure are to increase the probe beam intensity and increase photodetector sensitivity. Currently, the probe beam power is 4 mW, focused onto a spot size of 20 μ m, yielding a fluence of 1.27 kW/cm². This is much lower than the damage threshold of gold films, which generally exceeds 1 MW/ cm² [119], meaning that the probing optical intensity can be increased by at least 10 to 100 times without damaging the etalon. Of course, much higher optical energy should be avoided because part of the energy can travel through the etalon and lead to an undesirable temperature rise at the boundary of the etalon and overlying samples.

Reducing the etalon thickness is the easiest way to reduce the quality factor. For example, NEP of 2 kPa was measured with 11 μ m etalons using the method described above. However, making thicker etalons to improve the quality factor is not desired because thinner etalons lead to a broader detection bandwidth. The quality factor can also be increased by improving the optical reflection of the gold layers. When the reflection is improved from 85 % to 93 %, the quality factor can be enhanced by a factor of 2. Assuming a 5 times higher probing laser intensity, which is a very conservative estimation, combined with a 2 times higher quality factor, we conclude that the noise

equivalent pressure can be reduced by at least 20 dB. This has significant implications on ultrasound and photoacoustic imaging applications because extensive averaging can be avoided with better detection sensitivity, thus ensuring real-time imaging.

The same experimental setup shown in figure 3.4 can be used to measure the angular response of the etalon. A signal generator (8647A, Agilent Technologies, Santa Clara, CA, USA) outputs a continuous wave (CWs) signal at different frequencies (30–80 MHz with a 10 MHz step). This signal was gated with a 400 ns pulses (at a pulse repetition rate of 1 kHz) from a waveform generator (33250A, Agilent Technologies) using a frequency mixer (ZFM-4, Mini-Circuits, Brooklyn, NY, USA). The gated CW burst was amplified by a home-made power amplifier (37 dB gain) and then drove a 50 MHz piezoelectric ultrasound transducer (LiNbO₃, Resource Center for Medical Ultrasonic Transducer Technology, University of Southern California, CA, USA). The transducer was mounted on a mechanical stage capable of rotation and three-dimensional translation.

After setting an angle (between the transducer axis and the normal direction of the etalon), the transducer was moved to focus on the active element defined by the optical spot on the etalon surface, and signals corresponding to different (CW) frequencies detected by the etalon were then recorded by an oscilloscope (WaveSurfer 432, LeCroy, Chestnut Ridge, NY, USA). The signal magnitudes at different frequencies and angles from 0 to 45 degrees are shown in figure 3.8. Note at frequencies up to 50 MHz, the relative attenuation at 45 degrees compared with 0 degree is only 3 dB, almost negligible. Only at frequencies above 80 MHz does the attenuation at 45 degrees exceed 10 dB.

Therefore, the etalon is suitable for high-frequency ultrasound detection even at large angles, making it ideal for photoacoustic imaging applications.

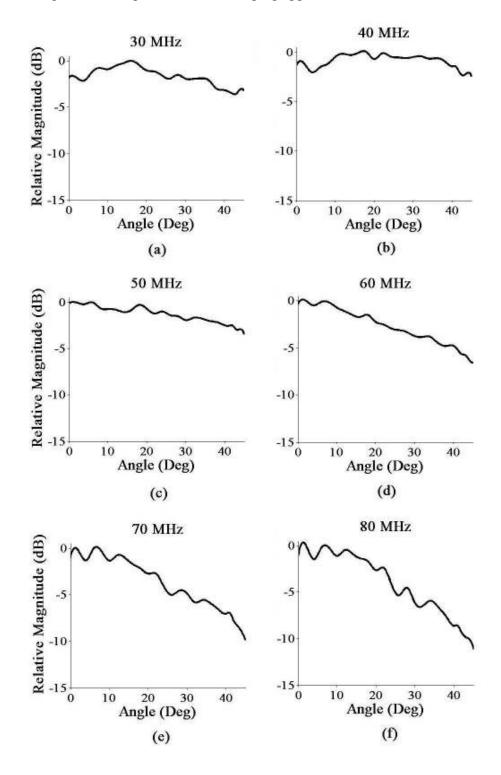


Figure 3.8: Angular response from 0 to 45 degrees at acoustic frequencies of (a) 30 MHz, (b) 40 MHz, (c) 50 MHz, (d) 60 MHz, (e) 70 MHz, and (f) 80 MHz.

3.4 Photoacoustic imaging

Among all medical needs, the ability to detect cancers at early stages [120-122] is one of the ultimate goals of virtually every biomedical imaging modality. However, most imaging techniques, including X-ray computerized tomography (CT) [123-124], ultrasound [125-126], and magnetic resonance imaging (MRI) [127], cannot image small tumors with high specificity. This limitation significantly reduces the probability of successful therapy, since current systems often cannot identify tumors before they reach lethal range. Therefore, it is strongly desired to develop new, non-invasive imaging technologies providing both high contrast and high resolution, preferably in real-time.

Pure optical imaging can produce the high contrast needed because of the vast differences in optical properties within biological tissues. Optical imaging suffers from strong light scattering in tissues, however, resulting in either poor imaging depth, as in optical coherence tomography (OCT) [128-130], or limited imaging resolution, as in diffusive optical tomography (DOT) [131-133]. On the other hand, ultrasound scattering is much weaker, which consequently yields good resolution at sufficient depth. Conventional ultrasound imaging [134] is based on detection of the compressional properties of tissue; therefore its contrast is generally weak. A hybrid approach could possibly overcome shortcomings of each by merging the contrast advantages of optical imaging with the resolution advantages of ultrasound imaging. Photoacoustic imaging represents such a hybrid technology [135-140].

Typically, a pulsed laser illuminates the imaging target, where optical energy is absorbed with a distribution corresponding to the optical absorption properties within the desired volume. Acoustic waves are launched by thermoelastic expansion, and are measured by ultrasonic transducers placed around the sample in a pre-determined geometry. Then, recorded photoacoustic signals are used to reconstruct the image representing the optical absorption distribution. Clearly, contrast is based on the optical properties of biological tissues, and resolution is scaleable with the frequency of the ultrasound waves.

Photoacoustic imaging has seen fast and aggressive growth during the past decade. Successful studies on breast cancer detection [138-139], small animal imaging [141-142], functional imaging [143-144], and reconstruction algorithms [145-148] have brought tremendous excitement to this field. Most experimental setups include a pulsed laser and an ultrasound transducer array as the two key elements. The wavelength of the pulsed laser is determined by optimizing both optical penetration depth and optical contrast between cancerous and normal tissues. In most cases, the near-infrared range gives the best trade-off between the two.

Like in ultrasound imaging, lack of 2D ultrasonic detection arrays is also the bottleneck for 3D real-time high-resolution photoacoustic imaging. Etalons can be used as an alternative to piezoelectric arrays, and have shown promising results under frequencies of 40 MHz. Clearly, the new 5.9 µm etalons will be able to significantly improve the spatial resolution of photoacoustic imaging systems.

Photoacoustic imaging experiments were conducted using the setup shown in figure 3.9. The imaging target is placed on the etalon, and a pulsed laser beam illuminates from the side. The laser source is a commercial high energy solid state laser at 532 nm (Surelite, Continuum, Inc., Santa Clara, CA, USA), which produces a 5 ns laser pulse at a

repetition rate of 20 Hz. The energy is typically above 100 mJ/pulse, and is normally reduced to 10 mJ/pulse using neutral density (ND) filters before hitting the sample. This yields an optical fluence of about 15 mJ/cm², lower than the visible light safety threshold of 20 mJ/cm² per pulse [149]. The probe CW laser beam configuration is the same as that in the characterization experiments (figure 3.4). Therefore, the detection array element size is unchanged at 20 μ m and the probe power is still 4 mW. The only difference is that all probe beam components are mounted on a 2D stage, which mechanically scans across the etalon to form a detection array.

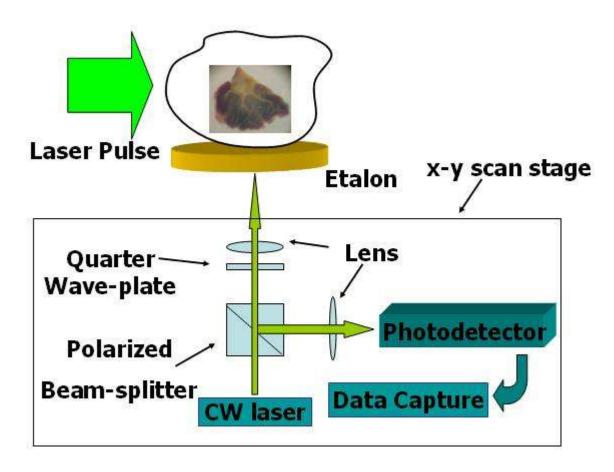


Figure 3.9: Experimental setup for photoacoustic imaging using an etalon.

Note that the probe beam is scanned, but the etalon remains fixed. This means any variation in optical resonance characteristic across the imaging aperture will affect ultrasound recordings. This scan geometry was chosen because it emulates the ultimate geometry of a fully parallel probe system. Typical scanning configurations are demonstrated in figure 3.10, with the red dots illustrating a 2D array and the blue dots representing a 1D array, used for 3D and 2D imaging respectively. At each detection position, the signal is averaged 100 times before recording and transfer to the computer for image reconstruction.

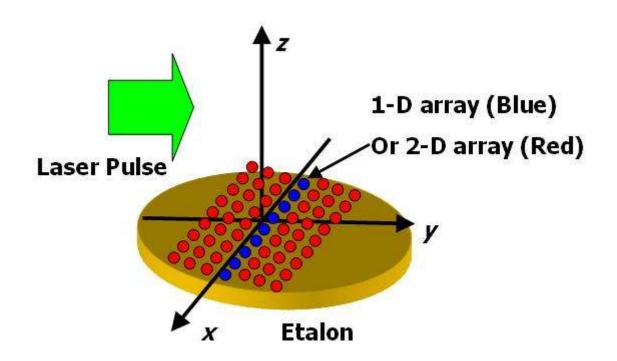


Figure 3.10: Demonstration of 1D and 2D etalon detection array configurations.

A 6 μ m diameter carbon fiber embedded in gel phantom is first used as the imaging target. The carbon fiber is positioned parallel to the etalon at a distance of about

1.25 mm from the surface. The detection optical beam is scanned 3 mm in 20 µm steps along the cross section of the carbon fiber. The spectrum of a typical signal is shown in figure 3.11 (a). Note that there are a number of interferences in this signal due to the presence of several photoacoustic sources (discussed below). Nevertheless, there is still significant acoustic power at frequencies up to 150 MHz. A band pass filter from 70 to 150 MHz is applied to the recorded signal at each position before conventional delay-sum beamforming algorithms are used for 2D image reconstruction. Note that the application of the 70-150 MHz band pass filter reduces the SNR of each signal by about 9 dB, but still leaves enough for a reasonable dynamic range. A filter in the higher frequency range produces images with better resolution, thus the specific choice of the band pass filter is based on the tradeoff between resolution and dynamic range.

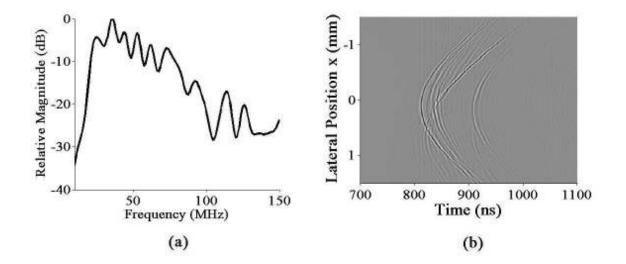


Figure 3.11: (a) Spectrum of a typical acoustic signal from a single array element. (b) Wavefield plot of the detected acoustic field along a 1D array.

The wavefield plot of the detected acoustic field along this 1D array is shown in figure 3.11 (b), and the reconstructed image is shown in figure 3.12. Note that there are

other photoacoustic sources in the phantom, as clearly seen from the wavefield plot. They represent small optically absorbing objects that were accidentally mixed in the gel. However, these objects are a few hundred microns away from the carbon fiber, and therefore do not appear in the reconstructed cross-sectional image of the carbon fiber. The lateral FWHM resolution is determined to be 20 μ m and the axial resolution is 19 μ m. These results demonstrate that thin polymer etalons can form detection arrays for photoacoustic imaging at resolutions better than 20 μ m, representing one ultrasound wavelength at a frequency of 75 MHz.

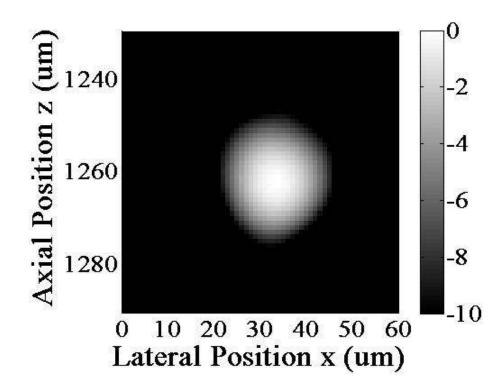


Figure 3.12: Reconstructed image of the cross section of a 6 μ m diameter carbon fiber displayed over a 10 dB dynamic range, where 0 dB represents the maximum reconstructed signal.

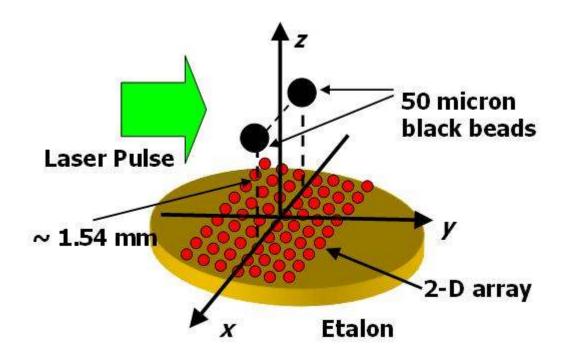


Figure 3.13: Experimental configuration with two black beads as imaging targets.

The next task is to test a 2D array for 3D photoacoustic imaging. A gel phantom containing two 50 μ m diameter black beads with centers separated by about 70 μ m was used for these studies. The geometrical configuration is shown in figure 3.13. The two beads are placed along the x direction about 1.54 mm above the etalon. The coordinates of their centers are designated as (35, 0, 1540) μ m and (-35, 0, 1540) μ m. A photo of these beads on the x-y plane at z=1540 μ m is displayed in figure 3.14 (a). The detection laser is scanned through a region of 2 mm in x by 2 mm in y, with 20 μ m steps in both directions. Band pass filtering from 35 to 105 MHz was chosen to balance desired resolution and dynamic range. The reconstructed image from the x-y plane at z=1540 μ m, which contains the centers of the beads, is shown in figure 3.14 (b), while those from the y-z plane at x=35 μ m (intersection just one bead) and x-z plane at y=0 (intersecting both beads) are shown in figure 3.14 (c), and 3.14 (d) respectively. These images accurately

depict the actual source geometry, and confirm the capability of etalons for 3D photoacoustic imaging.

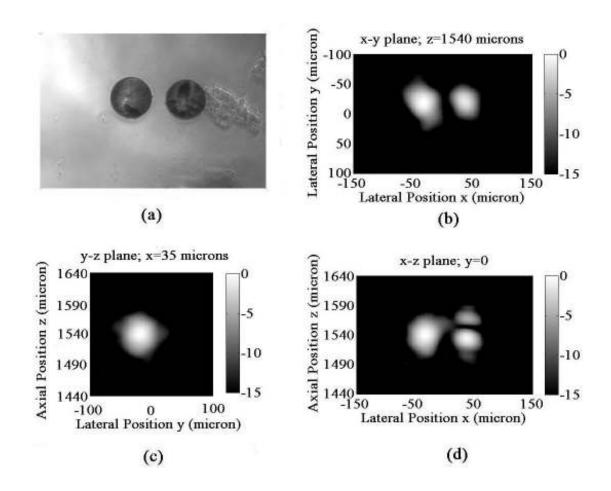


Figure 3.14: (a) Photo of two 50 μ m diameter black beads. Reconstructed images from the (b) x-y, (c) y-z, (d) x-z planes displayed over a 15 dB dynamic range, where 0 dB represents the maximum reconstructed signal.

As a very preliminary demonstration of the capabilities of thin polymer etalons for high-resolution biomedical applications, a brain slice was imaged photoacoustically using the same setup. A 0.5 mm thin slice from the cerebellum of a guinea pig was prepared, and the sample was stained with Cresyl Violet to increase optical absorption using a standard histological protocol. A photo of the brain slice under a microscope is shown in figure 3.15 (a). Dark, purple-colored regions contain large concentrations of dye, and are far more absorbing than the remaining parts of the tissue. The slice is embedded in a gel phantom, and positioned in the x-z plane, defined exactly the same as shown in figure 3.13. Because the sample is a thin slice, a 1D detection array was employed to illustrate major structures within the slice.

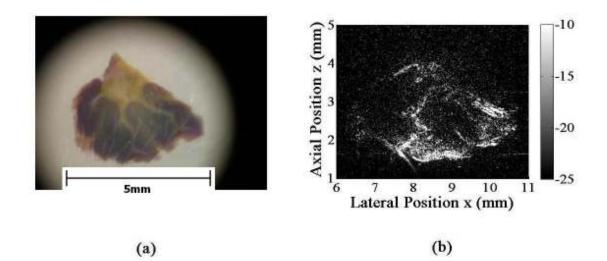


Figure 3.15: (a) Photo of a dye-injected brain slice. (b) Reconstructed photoacoustic image of the brain slice displayed over a 15 dB dynamic range, where 0 dB represents the maximum reconstructed signal.

The detection laser is scanned 6 mm in 20 μ m steps along the x direction right below the slice. A band pass filter from 35 to 105 MHz was used. The reconstructed image is shown in figure 3.15 (b). As the photoacoustic image suggests, the centerbottom part of the tissue is highly optical absorbing, thus has a high concentration of dye. This is verified by looking at the photo on the left, where an extensive purple area exists at the corresponding position in the photo. The same conclusion also holds for the area at the lower right corner of the tissue. The photo shows another area with high concentration of dye at the far left edge of the tissue, which does not show up in the photoacoustic image. One possible reason is that laser illumination was not uniform, and this area was possibly beyond the boundary of the laser beam, greatly reducing the absolute energy absorption and consequent acoustic signal generation. Overall, the outlines of the dye-injected portions in the photo of the brain slice are clearly captured in the photoacoustic image, suggesting that the etalon system described here is appropriate for high-resolution biomedical applications.

3.5 Etalon thickness and array capabilities

Choosing the appropriate thickness is a critical step in fabricating an etalon. As discussed in this chapter, thinner etalons produce broader bandwidth, while thicker etalons yield better sensitivity. Determining the optimal thickness is a tradeoff between bandwidth and sensitivity. In our current experimental setup, a 5.9 µm thick etalon provides bandwidth of well above 50 MHz and NEP of 3.9 kPa. In contrast, an 11 µm thick etalon has a better NEP of about 2 kPa, but is limited to below 40 MHz in bandwidth. Generally, thinner etalons are preferred for two reasons. First, a 2D array element with higher bandwidth, especially above 50 MHz, is not available with other transduction techniques. Consequently, the number one priority should always be to provide as large a bandwidth as possible. Second, the sensitivity can be easily improved using higher laser power for the probe beam. The results presented here were at optical fluencies far below those that could potentially damage the etalon surface.

At this stage, the focus of the detection laser probes a single array element, and an array is formed by mechanically scanning the detection laser. However, simultaneous

detection from all elements in an array system is strongly desired for real-time imaging applications. Our group has taken a big step forward by building a system with an optical end capable of parallel probing [150], where an unfocused laser beam probes a large area on the etalon surface, and a photodetector is scanned to acquire signals from all channels. A realistic imaging device can be realized by splitting the ultrasound detection beams into an array of focused spots on the surface of the device. Three main questions must be answered for this approach: first, how to deliver an array of laser beams; second, whether the device thickness is uniform enough for multi-point ultrasound detection; and third, how to detect an array of reflected CW laser beams simultaneously modulated by echo acoustic waves.

For laser beam delivery, we propose to use a graded index (GRIN) fiber bundle for simultaneous illumination and detection. Typically, a fiber bundle contains several thousand individual light guides; each has a diameter of 10 to 20 μ m, and the spacing is also about 10 to 20 μ m. The small size of each individual light guide provides an easy way to achieve smaller element size, which is critical for limiting the divergence of the radiation pattern for high-frequency imaging. Laser power should also be increased corresponding to the total number of elements used, which can be done using optical amplifiers such as Erbium doped fiber amplifiers (EDFA).

The uniformity of the device is important especially for ultrasound detection, because the thickness of the structure determines the resonance wavelength of the etalon. This correspondingly determines the optimal wavelength for the detection laser beam, which must be equal to or close to the actually used wavelength at all element spots. Figure 3.16 shows how the optimal wavelength changes over a distance of 2 mm. The largest

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difference is merely 0.1 %, which means that the 5.9 μ m SU-8 etalon is uniform enough to build a high-frequency array of thousands of elements. Meanwhile, InGaAs photodiode arrays are commercially available, which can be integrated into our system with appropriate modifications. Therefore, expanding a single element optoacoustic detector into an array system can be practically done by splitting the laser beams and focusing them onto an array of focused spots.

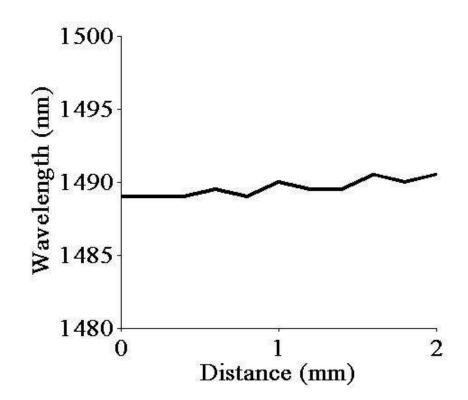


Figure 3.16: Optical detection wavelength over a distance of 2 mm.

3.6 Summary of etalons

In summary, a 2D optoacoustic detection array using thin polymer etalons has been fabricated, characterized, and tested for ultrasound and photoacoustic imaging. A 5.9 µm thick etalon possesses bandwidth of well over 50 MHz, NEP of 3.9 kPa for a 20 μ m diameter element, better than that of PVDF equivalents, and angular attenuation of barely 10 dB at 45 degrees for frequencies approaching 80 MHz. Photoacoustic imaging experiments have demonstrated the etalon's utility in 3D imaging, high-resolution (better than 20 μ m) imaging, and imaging of biological tissues. These results suggest that thin polymer etalons are attractive alternatives to piezoelectric transducers for 3D highresolution ultrasound and photoacoustic imaging.

CHAPTER 4

INTEGRATED TRANSDUCER USING GOLD NANOSTRUCTURE

4.1 Fabrication

Both optoacoustic transmitters and receivers have been significantly improved especially in terms of bandwidth, and are mature enough to be integrated into a single device for practical imaging applications. Particularly, optoacoustic transmitters using a 2D gold nanostructure as the optical absorber provide a convenient configuration for pulse-echo operation. In this chapter, we explore details of the design and fabrication of the integrated device, as well as characterization of the optical and acoustical properties. Images obtained from a 1D synthetic aperture formed by mechanically scanning the imaging target also are presented.

The most crucial part of the device is the non-liftoff 2D gold nanostructure, which is fabricated using laser interference lithography and nanoimprint lithography. The procedures were described in chapter 2, and are briefly recapped here as the following: A mold is first fabricated, which consists of a 2D array of SiO₂ pillars with thickness of 200 nm and period of 220 nm on a Si wafer. Each pillar is 128 nm by 110 nm in cross section. It is then imprinted on a 200 nm thick photoresist layer, which was spin-coated onto a glass substrate. After separation, a 200 nm thick photoresist layer with 2D arrangements of air holes spaced every 220 nm is obtained, where each air hole is 200 nm deep and 128 nm by 110 nm in cross section. A 20 nm layer of gold is then deposited on top of the structure using an electron beam evaporator, not only leaving a 2D array of gold nanoparticles at the bottom of the air holes, but also covering the top of the photoresist layer. The structure obtained here is called the non-liftoff gold nanostructure, and are shown in figure 2.13 in chapter 2.

A mixture of PDMS (Sylgard 184, Dow Corning, Midland, MI, USA) and D4 (Octamethylcyclotetrasiloxane, Gelest Inc., Philadelphia, PA, USA) is then spin-coated on top of the gold nanostructure, where the thickness is about 2.7 μ m. A 30 nm thick gold layer is then deposited using a second gold evaporation. As a final step, an additional 0.5 μ m thick PDMS layer is spin cast over the entire device for protection. A sketch of the side view of the entire structure is shown in figure 4.1.

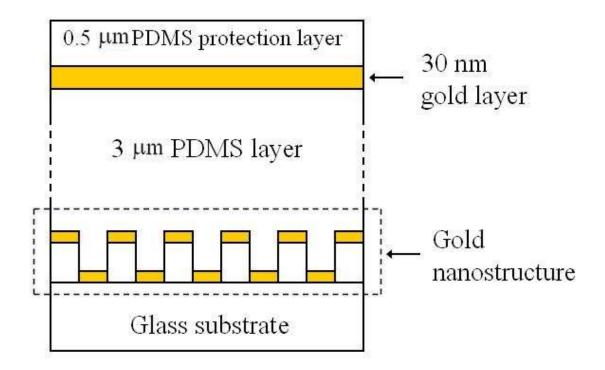


Figure 4.1: Sketch of the side view of the all-optical transducer.

4.2 Optical and acoustical properties

Optical transmission measurements were done using a Nikon TE300 Eclipse inverted microscope (20x objective; NA=0.44) with transmitted light coupled into an Ocean Optics SD2000 fiber coupled spectrometer using an achromatic lens. Figure 4.2 shows how optical transmission varies with wavelength.

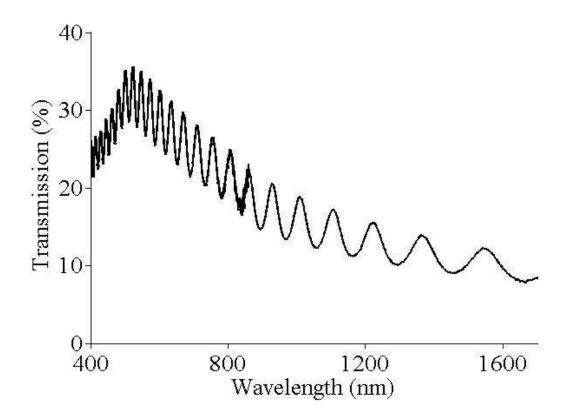


Figure 4.2: Optical transmission vs. wavelength (400 nm - 1650 nm) for the gold nanostructure.

The gold nanostructure serves as efficient optical absorber for optoacoustic transmission because surface plasmons localized around the particles strongly absorb light at a resonant wavelength that depends on the size, shape, and local dielectric environment of the gold nanoparticles. It has been previously shown in chapter 2 that the resonance wavelength for the structure used in our device is generally in the range of 700 nm to 800 nm. Optical reflection from the substrate side of the gold nanostructure has been measured at wavelengths of 780 nm (51%) and 1550 nm (75%). This means that optical extinction, mostly optical absorption, is about 30 % at 780 nm, and about 10 % at 1550 nm, consistent with the previously demonstrated resonance wavelength.

Due to the large percentage of optical absorption around the resonance wavelength, a pulsed laser beam at a wavelength of 780 nm is used for ultrasound generation. The laser pulse is focused onto the gold nanostructure, where the gold nanoparticles absorb a large portion of the optical energy. Heat absorbed is then rapidly transferred to PDMS surrounding the gold nanostructure, causing a temperature rise at the focal spot of the laser. Therefore, thermal expansion in the PDMS layer will launch an acoustic wave, which easily penetrates the thin gold layer and the additional PDMS layer into the overlying samples. High-frequency ultrasound is guaranteed using a laser pulse with duration of several nanoseconds.

An etalon is formed for optical detection of ultrasound with the gold nanostructure, the additional 30 nm gold layer, and the 2.7 μ m PDMS layer between. A high quality factor is achieved if both the gold layer and the gold nanostructure are highly reflective. Optical reflection from the polymer side of the gold nanostructure is measured and fluctuates between 85 % and 95 % in the wavelength range of 1440 nm to 1590 nm, far from the resonance wavelength, as shown in figure 4.3. Optical reflection from a 30 nm thick gold layer in the same wavelength range is about 90 %. Thus, taking both reflection coefficients to be 90 %, the quality factor is estimated to be:

$$Q = \frac{2nd}{\lambda} \cdot \frac{\pi\sqrt{R}}{1-R} \approx 150 \tag{4.1}$$

Here n = 1.43 is the refractive index of PDMS; $d = 2.7 \mu m$ is the thickness of the PDMS layer; $\lambda = 1520 \text{ nm}$ is the resonance wavelength; and R = 0.9 is the optical reflection.

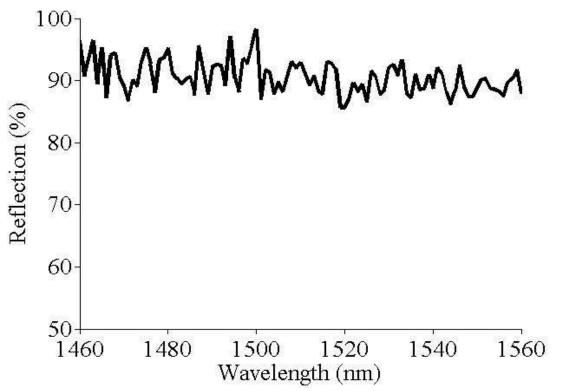


Figure 4.3: Optical reflection vs. wavelength far from the resonance wavelength (1460 nm - 1560 nm) for the gold nanostructure.

As stated in chapter 3, optical reflection from a Fabry-Perot cell shows sharp resonance when the optical path of two-way travel in the etalon bulk equals an integer multiple of the wavelength ($2nd = m\lambda$, where *m* is an integer). Figure 4.4 shows the experimentally measured optical reflection resonance, which occurs at 1520 nm with FWHM of 10.5 nm. Theoretically, the FWHM of the resonance can be determined using:

$$\Delta \lambda_{1/2} = \frac{\lambda}{Q} \approx 10 \text{ nm}$$
(4.2)

The theoretical value agrees well with experimental results.

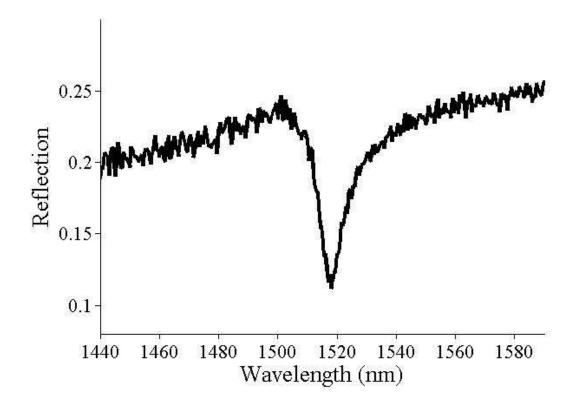


Figure 4.4: Optical resonance of the etalon structure.

The frequency response of the etalon structure is characterized using a high frequency piezoelectric transducer. First, a pulse-echo signal of the piezoelectric transducer reflected from a glass substrate is recorded. Then the glass substrate is replaced by the etalon, where the signal from the piezoelectric transducer is also recorded. The frequency response of the etalon is derived by dividing the spectrum of the etalon signal by the square root of the spectrum of the transducer pulse echo. Both spectra, together with the derived frequency response of the etalon, are shown in figure 4.5. Clearly, the etalon is suitable for ultrasound detection exceeding 50 MHz.

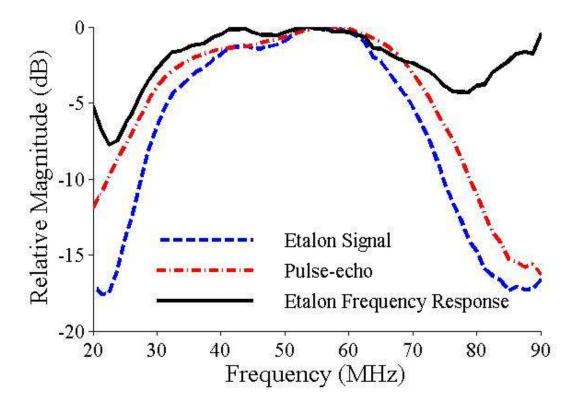


Figure 4.5: Spectrum of the etalon signal, square root of the spectrum of pulseecho signal of the piezoelectric transducer, and the derived etalon frequency response.

In summary, the working mechanism of the all-optical ultrasound transducer is defined by the unique optical properties of the gold nanostructure. Large optical absorption near the resonance wavelength of the structure enables the gold nanostructure and the PDMS layer to operate as an optoacoustic transmitter using a pulsed laser beam. Meanwhile, very high optical reflection at wavelengths far from resonance enables the gold nanostructure, the gold layer, and the interleaved PDMS layer to form an etalon for ultrasound detection, where a continuous wave laser is used as the optical probing beam.

4.3 Ultrasound pulse-echo experiment

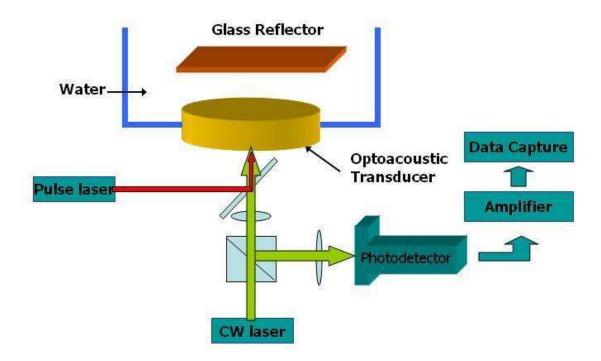


Figure 4.6: Setup of an ultrasound pulse-echo experiment.

As a preliminary evaluation of the all-optical ultrasound transducer, a simple ultrasound pulse-echo experiment is performed using the setup shown in figure 4.6. The integrated optoacoustic transducer is mounted at the bottom of a water tank. The pulsed laser excitation source is a commercial high energy solid state laser with tunable wavelength (Surelite, with OPO Plus, Continuum, Inc., Santa Clara, CA, USA), which produces a 5 ns laser pulse when tuned to the wavelength of 780 nm, roughly the resonance wavelength of the gold nanostructure. The beam is coupled into a multimode fiber with core size of 50 μ m, output through a collimator with focal length of 15 mm and a convex lens with focal length of 20 mm, and reflected from a dielectric mirror before

it's focused onto a 70 μ m spot on the gold nanostructure with energy of about 1 μ J/pulse (fluence of 26 mJ/cm²). The dielectric mirror is designed to be highly reflective at wavelengths of 750 nm to 850 nm, and highly transmittable at wavelengths of 1300 nm and higher. A 70 μ m spot is the minimum diameter for out current setup.

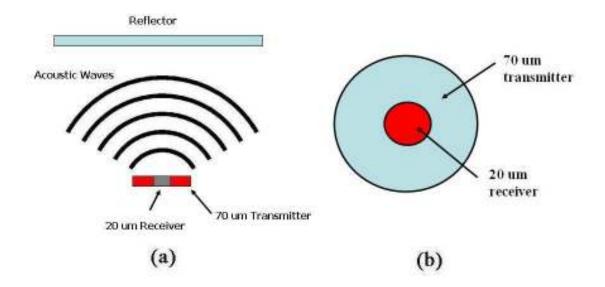


Figure 4.7: (a) Diagram of the transmit/receive elements. (b) The top view of the transmit/receive elements.

The generated acoustic waves are launched into the water, and reflected back from a glass slide aligned parallel to the etalon surface and placed about 1.5 mm away. To detect the pulse-echo ultrasound signal, a CW laser beam at 1517 nm with power of 4 mW is used. It travels through a polarizing beam splitter and a quarter-wave plate before being focused onto a concentric 20 µm spot through the dielectric mirror. A diagram of the transmit/receive elements is shown in figure 4.7. A smaller detection spot size is used to maximize the angular response of the equivalent array element for imaging applications. Reflected light, which has been modulated by the pulse-echo ultrasound waves, is collected using an amplified InGaAs photodiode. The signal is amplified by 30 dB before data capture.

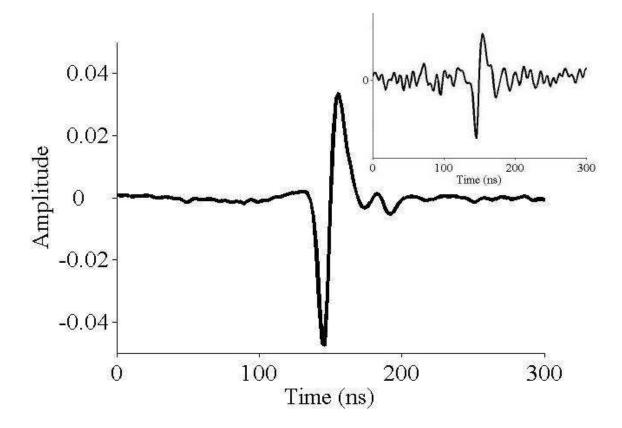


Figure 4.8: Pulse-echo signal; insert is a single shot recording and the primary signal represents an average of 1,000 recordings.

Figure 4.8 shows the pulse-echo signal with 1000 averages, and the upper right inset shows a single-shot acquisition of the pulse-echo signal. The signal to noise ratio (SNR) is measured to be over 10 dB for this experiment in which only a very small fraction of the transmitted acoustic power is captured by the 20 μ m diameter receive aperture. According to the results in chapter 2, the acoustic pressure is about 500 kPa at a distance of 10 mm away from the device surface with input optical power of 25 μ J.

Because the acoustic pressure is proportional to the input optical power over distance, and assuming that the glass substrate is a perfect acoustic reflector, we conclude that the acoustic pressure echoed back at the device surface is about 67 kPa.

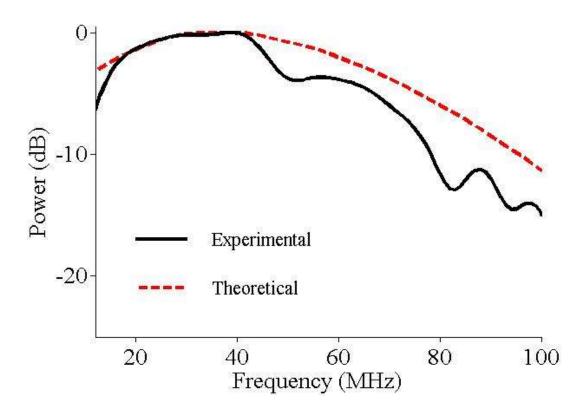


Figure 4.9: Spectrum of pulse-echo signal (solid curve) and simulation curve (dashed curve).

The spectrum of the pulse-echo signal (solid curve), averaged 1000 times, is shown in figure 4.9. The center frequency is 40 MHz, with -6 dB bandwidth of 57 MHz. The theoretical spectrum is also shown for comparison. It is derived by taking the spectrum of the time derivative of the laser pulse, multiplying by the etalon frequency response, then taking into account the attenuation in water (0.0022dB/cm/MHz²). Apparently, the two curves are in reasonably good agreement. As noted above, the temporal profile of the generated ultrasound is proportional to the derivative of the input optical pulse. Therefore, the center frequency and bandwidth of the pulse-echo signal is mainly determined by the incident laser pulse, as well as the frequency response of the etalon.

The most straightforward approach to achieve higher center frequency and broader bandwidth is to use a shorter pulse. Also, the frequency response of the etalon is directly determined by the thickness of the polymer bulk, and can be greatly improved by further reducing the overall thickness of the structure. When a 2 ns laser pulse is used instead of a 5 ns one, combined with an etalon thinner than 1.5 μ m, the bandwidth is expected to be enhanced to over 100 MHz.

The surface acoustic pressure at the moment of generation can be calculated using equation (2.15):

$$A_{near} = \sqrt{\frac{e}{8\ln 2}} \frac{2\pi cr\tau}{S} A_{far}$$

In our current experiment, the optoacoustic transmission element is about 70 μ m, the pulse duration is 5 ns, the distance is 3 mm, and the far field acoustic pressure is 67 kPa. Therefore, it's not difficult to calculate that the surface acoustic pressure is estimated to be 1.93 MPa. High surface pressures exceeding 1 MPa enable good SNR and high-quality imaging even though the transmitter emits a far-field, diverging radiation pattern where intensity falls off as the inverse square of distance.

As a side note, the optoacoustic transduction efficiency η can be conveniently calculated using the surface pressure following equation (2.17):

$$\eta = \sqrt{\frac{\pi}{8\ln 2}} \frac{S\tau A_{near}^2}{ZE_{in}}$$

In our experiment, the transduction efficiency is about 0.037 %. Note that because A_{near} is proportional to E_{in} , and is inversely proportional to S, it's easy to conclude that the transduction efficiency is proportional to E_{in}/S . Therefore, the efficiency can be greatly enhanced by increasing input optical energy and reducing the element spot size.

Both the surface and the far-field acoustic pressures increase linearly with the optical energy absorbed by the gold nanostructure, thus the SNR can be further improved with higher input laser energy. The ultimate acoustic pressure available is determined by the thermal damage threshold of the structure, measured to be 25 μ J/pulse delivered to a spot size of 25 μ m (fluence of 5.1 J/cm²), about a factor of 200 higher fluence than used in our experiment. If the transmission element remains at 70 μ m, then at this energy level, the far-field acoustic pressure could reach 1.6 MPa at 3 mm away from surface, while the surface pressure can be as high as 50 MPa. For high frequency applications, an element size of 20 μ m or less is highly desired, which limits the maximum energy input to 15 μ J/pulse, yielding a far-field acoustic pressure of 1 MPa at 3 mm away from the surface and a surface pressure of 350 MPa with enhanced transduction efficiency of 7 %. Thus, even with reduced element size of 20 μ m, the generated acoustic pressure can be easily enhanced by 20 dB without damaging the device.

The noise equivalent pressure of the etalon can also be further improved. The sensitivity of the etalon can be expressed using equation (3.12):

$$p_{NEP} = \frac{4}{3\sqrt{3}} \frac{I_s \cdot E_Y}{I_0 \cdot Q}$$

Therefore, the noise equivalent pressure of the etalon is proportional to the sensitivity of the photodetector and the Young's modulus of the etalon bulk material, and

is inversely proportional to the intensity of the etalon probing beam and the quality factor of the etalon. According to arguments in section 3.3, the noise equivalent pressure can be reduced by at least 20 dB using a 5 times higher probing laser intensity combined with a 2 times higher quality factor. Because the generated acoustic pressure can also be improved by another 20 dB, the overall SNR of a single shot pulse-echo signal from a single 20 µm transmit/receive element can easily reach as high as 50 dB.

4.4 Ultrasound imaging

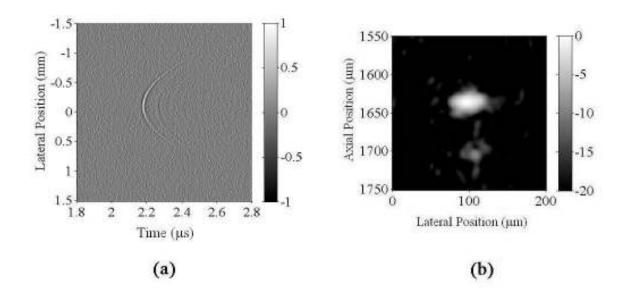


Figure 4.10: (a) Wavefield of a 70 μ m diameter metal wire; (b) reconstructed image (b) of the 70 μ m diameter metal wire.

To further evaluate this all-optical ultrasound transducer, its ultrasound imaging capabilities must be demonstrated. An imaging object replaces the glass slide in the setup shown in figure 4.6 while everything else remains the same, and a 1-D synthetic aperture is formed by mechanically scanning the imaging object. First, a 70 µm diameter metal

wire is used as the imaging target. A 2 mm long imaging aperture is scanned in 20 μ m steps. At each position, the signal is averaged 100 times before recording. Figure 4.10 (a) shows a wavefield plot of the detected acoustic field. Band-pass filtering (25 MHz to 85 MHz) and demodulation are applied, and then beam forming according to a simple synthetic aperture focusing technique (SAFT) is performed to reconstruct the image, shown in figure 4.10 (b).

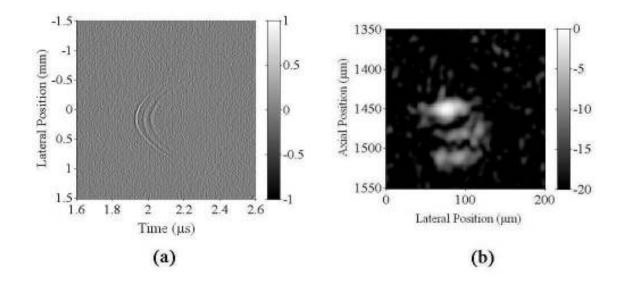


Figure 4.11: (a) Wavefield of a 70 μ m diameter hair; (b) reconstructed image (b) of the 70 μ m diameter hair.

Waves detected by the transducer are reflected from both front and back edges of the wire. Because metal is such a good acoustic reflector, most energy is reflected back from the front edge, while the signal from the back edge is quite small. As a result, two curves are observed in the wavefield plot, the first much stronger than the second. These features are captured in the reconstructed image, where the front edge is much brighter than the back. For comparison, the metal wire is replaced by a 70 µm diameter human hair, whose acoustic reflection is much lower than metal. The wavefield plot of the detected acoustic field is shown in figure 4.11 (a) and the reconstructed image in figure 4.11 (b). Clearly, both front and back edges are well represented in the wavefield and reconstructed image.

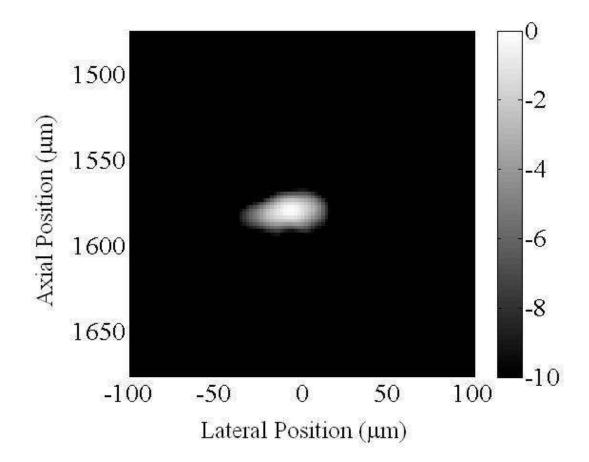


Figure 4.12: The reconstructed image of a 25 µm diameter metal wire.

To determine the imaging resolution of the current system, a 25 μ m diameter metal wire is chosen as the imaging object, while the scanning distance remains 2 mm with 20 μ m step separation. The reconstructed image is shown in figure 4.10. The -6 dB

axial resolution is determined to be 19 μ m, consistent with the bandwidth of the pulse. This also confirms that only the echo from the wire front edge contributes to the image because the axial resolution is smaller than the actual wire diameter. The -6 dB lateral resolution is 38 μ m, representing about one acoustic wavelength at the 40 MHz center frequency.

The transmit element size relative to an acoustic wavelength significantly affects the divergence of the radiation pattern [65]. A small element, especially 20 μ m or less, can emit considerable energy even at angles above 45 degrees, which is highly desired for a high-frequency array system. Due to limitations in our current illumination system, the optoacoustic transmission element size is 70 μ m, several times the acoustic wavelength at high frequencies. This means that recorded echo signals have lower bandwidths when the object is moved toward the end of the scan, thus limiting image quality. To reduce transmit element size below 20 μ m, the most straightforward method is to directly couple a pulsed laser with a multimode fiber having a core size smaller than 20 μ m, or preferably a single-mode fiber which allows more convenience and flexibility for a smaller focal spot.

4.5 Potential for real-time 3D imaging

At this stage, the imaging target is mechanically scanned to form an equivalent 1-D synthetic aperture. However, real-time imaging applications require simultaneous detection at all elements in an array system, and even simultaneous excitation in some configurations. This can be realized by splitting both the ultrasound generation and detection beams into an array of focused spots on the surface of the device. A diagram of this configuration is shown in figure 4.13 (a).

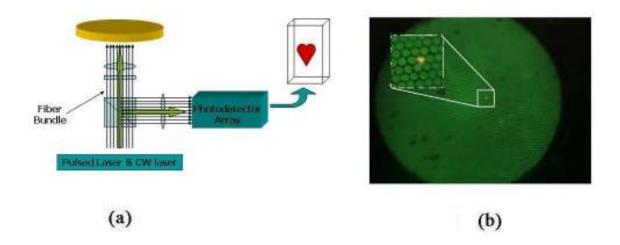


Figure 4.13: (a) Configuration of an optoacoustic array using a fiber bundle. (b) A typical cross section of a fiber bundle.

As discussed in section 3.5, a graded index (GRIN) fiber bundle can be used for optical beam delivery for simultaneous illumination and detection. A typical cross section of a fiber bundle is shown in figure 4.13 (b). The uniformity of the device is important, especially for ultrasound detection. It was previously shown in section 3.5 that in an etalon with SU-8 as bulk material, the optimal wavelength is uniform over regions of several millimeters, which is enough to build a high-frequency array of thousands of elements. Similar uniformity is expected to be achieved with PDMS etalons after further refinement of our fabrication methods. Meanwhile, InGaAs photodiode arrays are commercially available, which can be integrated into our system with appropriate modifications. Therefore, expanding a single element all-optical ultrasound transducer into an array system can be practically done by splitting the laser beams and focusing them onto an array of focused spots.

Another practical consideration for building an array system is to achieve SNR higher than 10 dB in our current single element system. This requires higher generated acoustic pressure and lower etalon noise equivalent pressure. As described in previous sections, the most efficient way to generate higher acoustic pressure is to increase input pulse laser energy, and the best methods to reduce etalon noise equivalent are to increase probing laser beam intensity, improve etalon quality factor, as well as utilize photodetectors with higher sensitivity. The acoustic pressure has the potential to be improved by 20 dB simply by increasing the input energy from 1 µJ/pulse to above 10 µJ/pulse, while it's not difficult to reduce the noise equivalent pressure by at least another 20 dB by increasing the probing laser power from 4 mW to 20 mW and the etalon quality factor from 150 to 300. Thus, the SNR of a single shot pulse-echo signal from a single element can easily exceed 50 dB without damaging the device, a value more than sufficient for a 2-D array element.

4.6 Summary of optoacoustic transducer using gold nanostructure

A broadband all-optical ultrasound transducer has been designed, fabricated, and evaluated for high-frequency ultrasound imaging. The device consists of a twodimensional gold nanostructure imprinted on top of a glass substrate, followed by a 3 μ m PDMS layer and a 30 nm gold layer. A laser pulse at the resonance wavelength of the gold nanostructure is focused onto the surface for ultrasound generation, while the gold nanostructure, together with the 30 nm thick gold layer and the PDMS layer between, forms an etalon for ultrasound detection, which utilizes a CW laser at a wavelength far from resonance as the probe beam. The center frequency of a pulse-echo signal recorded in the far field of the transducer is 40 MHz with -6 dB bandwidth of 57 MHz. The signal to noise ratio (SNR) from a 70 μ m diameter transmit element combined with a 20 μ m diameter receive element probing a near perfect reflector positioned 1.5 mm from the transducer surface is over 10 dB, and has the potential to be improved by at least another 40 dB. A high-frequency ultrasound array has been emulated using multiple measurements from the transducer while mechanically scanning an imaging target, where the -6 dB lateral spatial resolution is 38 μ m. Planned next steps include optimizing the gold nanostructure for maximal optical absorption at the resonance wavelength, modifying the experimental system to achieve higher SNR, and expanding a single transducer element to an array system for real-time imaging. Characterization of the device's optical and acoustical properties, as well as preliminary imaging results, strongly suggest that all-optical ultrasound transducers can be used to build high-frequency arrays for real-time high-resolution ultrasound imaging.

CHAPTER 5

INTEGRATED TRANSDUCER USING BLACK PDMS

5.1 Fabrication

In this chapter, we describe an integrated all-optical theta-array system combing the 11 μ m thick black PDMS film and the 5.9 μ m thick SU-8 etalon. The fabrication and configuration of the device will first be described, followed by ultrasound pulse-echo characterization. Preliminary images obtained using three metal wires as imaging targets will also be presented.

In this device, the generation and detection elements have to be spatially separate, as will be illustrated in the following texts. Therefore, a sparse array has to be used instead of a fully-sampled 2D array. The simplest array that separates transmit and receive elements is the Mills cross [151], which consists of a straight line of transmit elements and another straight line of receive elements in a perpendicular direction. However, this configuration is not feasible due to the limitation in our device structure. The first order approach utilizes a theta-array geometry, which contains a straight line of transmit elements and an annular-ring array [152-156] of detection elements surrounding the transmit elements. This configuration is the simplest and most effective 3D imaging array with our device structure.

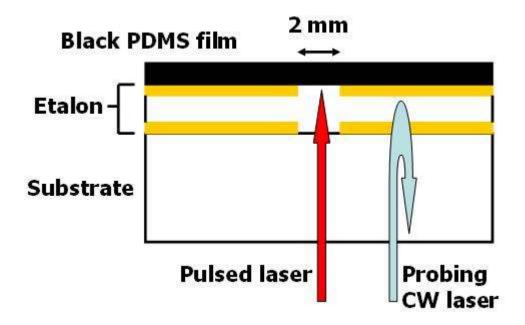


Figure 5.1: Side view of the optoacoustic device's structure

The first step is to fabricate a 5.9 μ m SU-8 etalon with a 2 mm diameter transparent window on top of a glass substrate. A 30 nm gold layer is first deposited as the first reflecting mirror using an electron beam evaporator, where a 2 mm diameter tape is masked onto glass to block the deposition of gold within the region. Then SU-8 photoresist (SU-8 2500, Microchem Corp., Newton, MA, USA) is spin coated on top at 2100 rpm for 40 seconds after the tape is pealed off, and the cured film is about 5.9 μ m thick and forms the etalon's polymer bulk layer. A 2 mm diameter tape is masked on the same region again, and another 30 nm gold layer is deposited as the second reflecting mirror. A mixture of PDMS (Sylgard 184, Dow Corning, Midland, MI, USA) and carbon black (Raven 14, Columbian Chemicals Inc., Marietta, GA, USA) with ratio of 6:1 is then spin coated into an 11 μ m black PDMS film on top. A cross-section through the structure is shown in figure 5.1. The 2 mm transparent window in the middle of the etalon

allows a pulsed laser beam to be focused onto the black PDMS film for ultrasound generation, while a CW laser beam is focused onto the remainder of the etalon for ultrasound detection.

5.2 Acoustical Properties

The 11 μ m thick black PDMS film and the 5.9 μ m thick etalon have been previously characterized for optoacoustic transmission and detection. However, the PDMS layer changes the acoustical properties of the etalon, as it attenuates ultrasound by about 1 dB/ μ m, and affects the frequency response of the etalon by changing the overall structure thickness. Therefore, the acoustic frequency response of the etalon needs to be re-investigated.

The etalon's optical resonance is first measured by recording the reflected optical intensity as the wavelength is scanned from 1480 nm to 1550 nm, as shown in figure 5.2 (a). The resonance wavelength is 1513 nm with FWHM of 6.3 nm. Therefore, the wavelength of the probing beam is tuned to 1511.5 nm for maximum detection sensitivity. The frequency response of the etalon is characterized using a 50 MHz piezoelectric transducer with a diameter of 2.5 mm and focal length of 4 mm (LiNbO₃, Resource Center for Medical Ultrasonic Transducer Technology, University of Southern California, CA, USA). First, the pulse-echo signal reflected from a glass substrate is recorded by the piezoelectric transducer. Then the transducer is placed a focal length away above the etalon, and the signal from the piezoelectric transducer is recorded from the etalon. Spectra of both signals are displayed in figure 5.2 (b). Clearly, the 5.9 µm thick etalon can detect ultrasound at 50 MHz and above even with an 11 µm black PDMS layer on top.

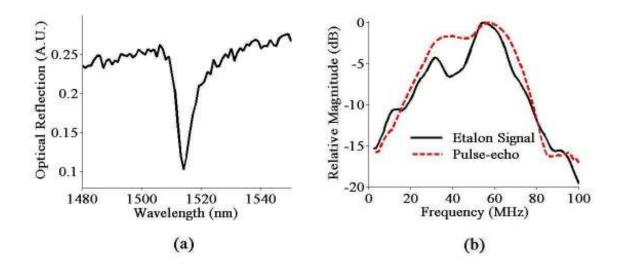


Figure 5.2: Optical resonance of the 5.9 μ m thick etalon with 11 μ m thick black PDMS on top; (b) spectrum of the etalon signal compared with the square root of the spectrum of pulse-echo signal of the piezoelectric transducer

An ultrasound pulse-echo experiment is performed using the setup shown in figure 5.3 (a). The integrated optoacoustic transducer is mounted at the bottom of a water tank. The pulsed laser excitation source is a commercial pulsed fiber laser (MIRVISION, Keopsys Inc., Lannion, France), which produces a 5 ns pulse with energy of 200 nJ/pulse at wavelength of 1064 nm and repetition rate of 5 kHz. The beam is output through a multimode fiber with core size of 18 µm, followed by a collimator with focal length of 12 mm and a convex lens with focal length of 20 mm. The beam is then reflected from a dielectric mirror before being focused onto a roughly 30 µm spot on the black PDMS film. The dielectric mirror is designed to be highly reflective at wavelengths of 1000 nm to 1100 nm, and highly transmittable at wavelengths of 1300 nm and higher. The generated ultrasound is reflected back from a glass reflector positioned parallel to the device surface and 1.7 mm away.

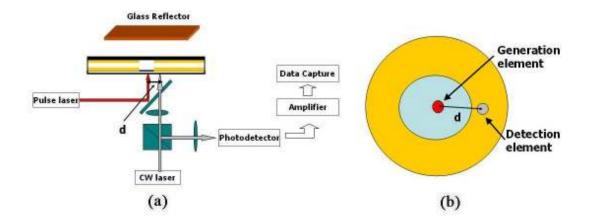


Figure 5.3: (a) Experimental setup of the pulse-echo experiment; (b) top view of generation and detection

To detect the pulse-echo ultrasound signal, a CW laser beam at 1511.5 nm with power of 4 mW is used. It travels through a polarizing beam splitter and a quarter-wave plate before being focused onto a 20 μ m spot on the etalon through the dielectric mirror. The distance between the generation and detection spots is denoted as d. Figure 5.3 (b) shows the top view of the geometry.

Figure 5.4 (a) shows the pulse-echo signal averaged 1000 times, when generation and detection elements are separated by 1 mm (d=1 mm). The SNR of this signal is 19 dB. The corresponding spectrum is shown in figure 5.4 (b), where the center frequency is 33 MHz and the -6 dB bandwidth is 41 MHz. The center frequency and bandwidth increases with shorter distance, as demonstrated in figure 5.4 (c) and 5.4 (d), where d is 0.6 mm and 0.2 mm respectively. For instance, when d=0.2 mm, the center frequency is 34 MHz while the -6 dB bandwidth is 51 MHz. The primary reason for this increase is that the frequency response of the etalon is incident-angle dependent, and always displays attenuation at higher angles [77]. Therefore, shorter distance leads to a smaller incident angle for the echo ultrasound waves, which consequently produces broader bandwidth.

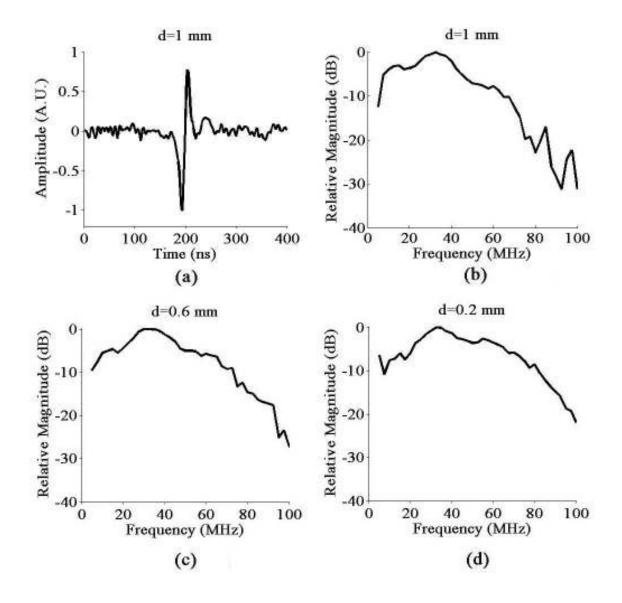


Figure 5.4: (a) Pulse-echo signal averaged 1000 times when d=1 mm; Spectrum of the pulse-echo signal averaged 1000 times when (b) d=1 mm; (c) d=0.6 mm; (d) d=0.2 mm.

5.3 Ultrasound imaging

An optoacoustic theta-array is illustrated in figure 5.5. The generation pulsed laser beam is scanned along a straight line to provide the optoacoustic transmission array, and the probe CW laser is scanned over an annulus to form the optoacoustic detection array. The setup is the same as that in the pulse-echo experiment shown in figure 5.3 (a), other than both lasers are controlled by separate motors for scanning. In our imaging experiment, the transmission array consists of eight elements with 200 μ m separation between adjacent elements, and the detection array contains 400 elements along a circle with 2.5 mm diameter (19.6 μ m separation between adjacent elements).

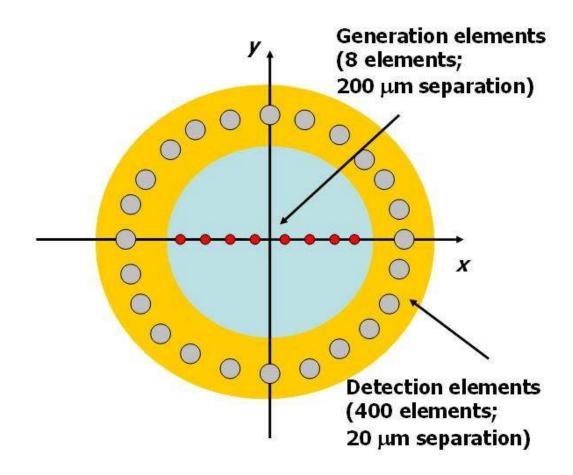


Figure 5.5: Illustration of the optoacoustic theta-array configuration

The center of the circular window is defined as the origin of the coordinate system. Because generation and detection beams are controlled by independent motor systems, it's important to first align the generation beam to the center of the annular detection array (origin of the coordinate system) so that the relative positions of all array elements can be accurately identified during imaging experiments. After alignment, pulse-echo signals are recorded at all detection elements, and the ultrasound wavefield is shown in figure 5.6. Clearly, the travel time from the generation spot to all detection spots are equal, verifying that the pulsed laser is indeed focused onto the center of the detection array.

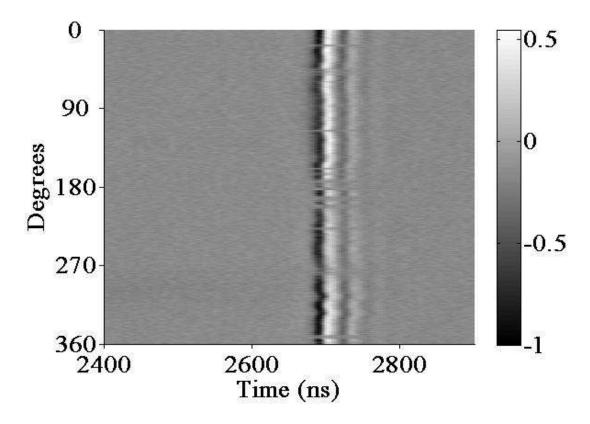


Figure 5.6: Wavefield of recorded pulse-echo signals from the annular detection array when the generation element is aligned to the center

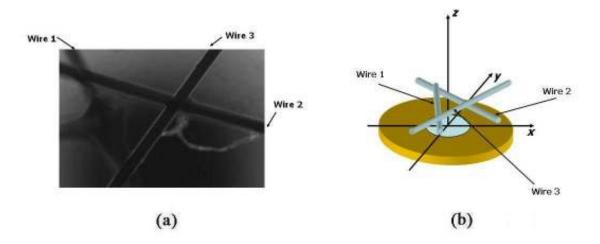


Figure 5.7: (a) Photo of the three metal wires used as imaging targets; (b) Geometry and experimental configuration used to image the three metal wires

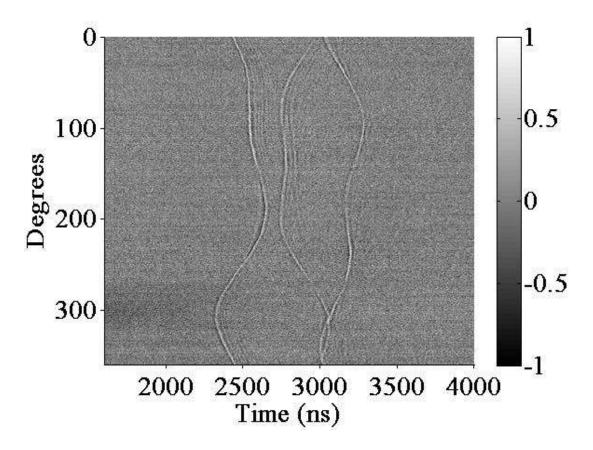


Figure 5.8: Wavefield plot of the detected acoustic field along the annular detection array at one generation spot

As a preliminary test using the theta-array, three 50 µm diameter metal wires are used as imaging targets. A photo of the arrangement is shown in figure 5.7 (a). The experimental configuration is shown in figure 5.7 (b), where the wires are placed roughly 1.8 mm from the device surface. When the generation laser is focused onto each generation element spot, the probe laser is scanned through the annular detection array, and the ultrasound signal at each position, averaged 1000 times, is recorded. The wavefield plot of the detected acoustic field along this annular array at one of the generation spots is shown in figure 5.8. Clearly, the three wires are well represented by the three distinctive curves in this wavefield.

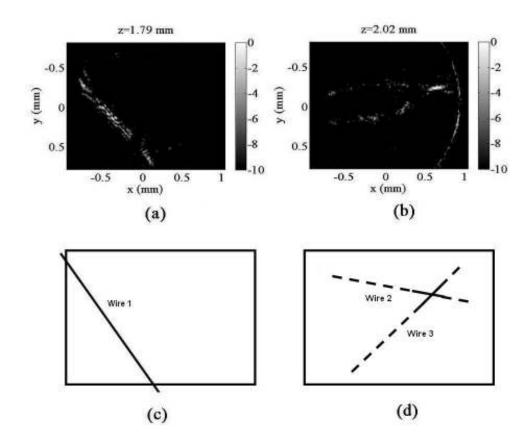


Figure 5.9: Reconstructed image from the x-y plane at (a) $z=1790 \ \mu\text{m}$; (b) $z=2020 \ \mu\text{m}$; Expected image from the x-y plane at (c) $z=1790 \ \mu\text{m}$; (d) $z=2020 \ \mu\text{m}$.

Band pass filtering from 25 to 75 MHz is chosen and applied to all signals to balance desired resolution and dynamic range. Conventional beamforming algorithms are then used for image reconstruction. The reconstructed image from the x-y plane at $z=1790 \mu m$, which intersects the first wire, is shown in figure 5.9 (a), while the reconstructed image from the x-y plane at $z=2020 \mu m$, which intersects the joint of the second and third wires, are shown in figure 5.9 (b). Correspondingly, expected images as determined by the geometry of the three wires are shown in figure 5.9 (c) and (d) respectively. The reconstructed images capture the positions of the three wires with reasonable accuracy, and the geometry of the imaging targets are well depicted overall, confirming the optoacoustic theta-array's capabilities and potential for 3D ultrasound imaging. Note that artifacts do exist, and there is a certain degree of distortion in the reconstructed images. Methods for improvements are discussed below.

5.4 Discussion and summary

At present, image quality is mainly limited by the small number of optoacoustic transmission elements. Sufficiently high pulsed laser energy is not available for our experiments, thus extensive signal averaging of 1000 times is required at each detection array element. The total time consumed at each generation element is a few hours, including mechanically scanning through the annular detection array, signal averaging, and data acquisition. Time constraints force us to use a maximum of eight generation elements with separation of 200 μ m, where of course significantly more generation elements with separation of less than 20 μ m are highly desired to reduce artifacts and greatly improve image quality.

In our experiments, optical pulses with 200 nJ/pulse are delivered to a spot size of 30 μ m for ultrasound generation, yielding an optical fluence of 0.03 J/cm², whereas the thermal damage threshold has been measured to be 30 J/cm². Commercial pulsed lasers with energy much higher than that used in our setup are available, thus the SNR of ultrasound signals can be improved by at least 40 dB using an optical energy of 20 μ J/pulse. This would enable us to record single-shot signals at each array element, enabling a significantly larger number of generation elements.

Simultaneous ultrasound detection from all array elements at each firing of an individual generation element would decrease imaging time significantly, making realtime imaging possible. A system with an optical end capable of parallel probing has previously been built by Dr. Huang from the University of Michigan, where an unfocused laser beam probes a large area on the etalon surface, and a photodetector is scanned to acquire signals from all channels. The most practical proposed solution utilizes a fiber bundle, which splits the primary laser beam into an array of separate laser beams that are then focused onto a programmable array geometry on the device surface. Immediate future work includes optimizing the optical setup involving fiber bundles, fabricating etalons with better thickness uniformity, and evaluating photodetector arrays.

The major drawback of this integrated optoacoustic device combining black PDMS films and etalon structures is that transmission elements and detection elements must be spatially separate. This means that fully-sampled 2D arrays are not available with this device, even though a theta-array system is adequate for many forward-looking imaging applications. An integrated single element capable of both ultrasound generation and detection can be fabricated using a gold nanostructure as optoacoustic transmitter.

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The advantage of using black PDMS over a gold nanostructure, however, is that its thermal damage threshold is six times higher, enabling higher ultimate acoustic pressures.

In summary, we have fabricated and tested an integrated optoacoustic device for 3D ultrasound imaging. The device consists of an 11 µm thick black PDMS film confined to a 2 mm diameter circular region acting as an optoacoustic transmitter, and surrounded by a 5.9 thick µm Fabry-Perot polymer etalon structure serving as an optoacoustic detector array. Pulse-echo signals display center frequencies above 30 MHz with bandwidths of at least 40 MHz with a 30 µm generation element and a 20 µm detection element. A theta-array is emulated by mechanically scanning the generation laser beam through eight 1D elements separated by 200 µm and the detection laser beam along an annular array of 400 elements separated by 19.6 µm. The reconstructed images capture the imaging targets with reasonable accuracy. Acoustical characterization, together with preliminary imaging results, demonstrates the device's capabilities and potential for 3D ultrasound imaging.

CHAPTER 6

CONCLUSIONS AND FUTURE WORK

6.1 Conclusions

The goal of this work is to develop broadband all-optical ultrasound transducers for high-resolution ultrasound imaging. Previously, the most efficient optoacoustic transmitter is a 25 µm thick black PDMS film, and the state of the art optoacoustic detector utilizes an 11 µm thick Fabry-Perot polymer etalon. Both devices operate below 50 MHz, and an integrated structure combining both optoacoustic transmitters and detectors has not been designed and fabricated.

This thesis first presented optoacoustic transmitters and detectors operating above 50 MHz, and then focused on integrated devices combining etalons and gold nanostructures or black PDMS films. Chapter summaries with key contributions are presented below:

In <u>CHAPTER 2</u>, two kinds of thin films have been developed for thermoelastic generation of ultrasound at above 50 MHz. The first is a thinner black PDMS film with thickness of 11 μ m, while the other uses a 4.5 μ m transparent PDMS film with a 2D gold nanostructure as optical absorber. With a 5 ns incident laser pulse, the center frequencies from both films exceed 60 MHz, and the bandwidths are over 50 MHz. Both structures

approach ideal thermoelastic sources, where the spectrum of the generated ultrasound is shown to be mainly determined by the temporal profile of the incident laser pulse, meaning that both the center frequency and the bandwidth have great potential to be improved if shorter laser pulses are used. The ultimate surface acoustic pressure available, determined by the thermal damage threshold of these films, could reach over 1500 MPa with the black PDMS film and over 300 MPa for the gold nanostructure film, more than sufficient for real-time ultrasound imaging applications.

Portions of the work contained in Chapter 2 were presented at the 2006 IEEE International Ultrasonics Symposium, and published in Applied Physics Letters and the IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control. The relevant citations are:

- Y. Hou, J. S. Kim, S. Ashkenazi, M. O'Donnell, and L. J. Guo, "Optical generation of high frequency ultrasound using two-dimensional gold nanostructure," Applied Physics Letters, 89 (9), 093901 (2006).
- Y. Hou, S. Ashkenazi, S. W. Huang, and M. O'Donnell, "Improvements in optical generation of high frequency ultrasound," IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control, 54 (3), 682-686 (2007).
- Y. Hou, J. S. Kim, S. Ashkenazi, M. O'Donnell, and L. J. Guo, "Optical generation of high frequency ultrasound using a two dimensional array of gold nanoparticles," Proceedings of the 2006 IEEE International Ultrasonics Symposium, 401-404 (2006).

The main contributions of this work are:

- Optimization of thin black PDMS films for optical generation of ultrasound at over 50 MHz.
- 2. Design and development of an innovative gold nanostructure for optoacoustic transmission at nearly 100 MHz. This structure is efficient and can also be used to create an integrated transmit/receive optoacoustic transducer
- 3. Systematic characterizations of optoacoustic transmitters' bandwidth, acoustic pressure, transduction efficiency, and thermal damage threshold.

In <u>CHAPTER 3</u>, a thinner etalon with thickness of 5.9 μ m have been fabricated and characterized as a high-frequency optoacoustic sensor. It possesses bandwidth of well over 50 MHz, NEP of 3.9 kPa for a 20 μ m diameter element, better than that of PVDF equivalents, and angular attenuation of barely 10 dB at 45 degrees for frequencies approaching 80 MHz. The detection sensitivity can be improved by increasing the probe beam intensity and reducing the etalon quality factor. Photoacoustic imaging experiments have been performed to demonstrate the etalon's utility in 3D imaging, high-resolution imaging, and imaging of biological tissues. Axial and lateral resolutions have been demonstrated to be better than 20 μ m. These results suggest that thin polymer etalon arrays can be used as ultrasound detectors for 3-D high-resolution ultrasound and photoacoustic imaging applications.

Portions of the work contained in Chapter 3 were presented at the 2007 BMES Annual Fall Meeting, and submitted to Journal of Biomedical Optics for publication. The relevant citation is: Y. Hou, S. W. Huang, S. Ashkenazi, R. S. Witte, and M. O'Donnell, "Thin polymer etalon arrays for high-resolution photoacoustic imaging," submitted to Journal of Biomedical Optics

The main contributions of this work are:

- Development and characterizations of 5.9 μm thick polymer etalons for highfrequency ultrasound detection arrays demonstrating bandwidth well above 50 MHz.
- 2. Demonstration of high-resolution photoacoustic imaging using etalon detection arrays with axial and lateral resolution better than 20 μm.

In <u>CHAPTER 4</u>, the first broadband all-optical ultrasound transducer has been designed, fabricated, and evaluated for high-frequency ultrasound imaging. The device consists of a two-dimensional gold nanostructure imprinted onto top of a glass substrate, followed by a 3 μ m PDMS layer and a 30 nm gold layer. The pulse-echo signal from a 70 μ m diameter transmit element combined with a 20 μ m diameter receive element probing a near perfect reflector positioned 1.5 mm from the transducer surface is over 10 dB, while the center frequency is 40 MHz with -6 dB bandwidth of 57 MHz. High-resolution ultrasound imaging capabilities have been demonstrated using a 1-D synthetic aperture formed by mechanically scanning the imaging target, where the -6 dB lateral resolution is 38 μ m.

Portions of the work contained in Chapter 4 were presented at the 2007 SPIE Symposium on Biomedical Optics, and the 2007 IEEE International Ultrasonics Symposium, and published in Applied Physics Letters and IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control. The relevant citations are:

- Y. Hou, J. S. Kim, S. Ashkenazi, S. W. Huang, L. J. Guo, and M. O'Donnell, "Broadband all-optical ultrasound transducers," Applied Physics Letters, 91 (7), 073507 (2007).
- Y. Hou, J. S. Kim, S. W. Huang, S. Ashkenazi, L. J. Guo and M. O'Donnell, "Characterizations of a broadband all-optical ultrasound transducer from optical and acoustic properties to imaging," accepted by IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control, In Press.
- Y. Hou, J. S. Kim, S. Ashkenazi, S. W. Huang, M. O'Donnell, and L. J. Guo, "All-optical ultrasound transducer," Proceedings of the 2007 SPIE Symposium on Biomedical Optics, 6437, 64370F (2007).
- Y. Hou, S. Ashkenazi, S. W. Huang, J. S. Kim, L. J. Guo, and M. O'Donnell, "Integrated all-optical ultrasound transducers," Proceedings of the 2007 IEEE International Ultrasonics Symposium, 715-718 (2007).

In addition, this work has been featured and highlighted in scientific magazines:

- ▶ Ultra-sound ultrasound, Nature Photonics, 1 (10), 560-561, Oct 2007.
- All-optical transducer designed for high-resolution ultrasound imaging,
 Photonics Spectra, Oct 2007.

The main contributions of this work are:

1. Designed and developed the first ever all-optical ultrasound transducer combining optoacoustic transmitters and detectors into a single element.

2. Demonstration of high-resolution ultrasound imaging using all-optical ultrasound with lateral resolution better than 40 μm.

In <u>CHAPTER 5</u>, an integrated optoacoustic device combining black PDMS and etalon structures has been designed and fabricated for 3D ultrasound imaging. The device consists of an 11 µm thick black PDMS film confined to a 2 mm diameter circular region acting as an optoacoustic transmitter, and surrounded by a 5.9 thick µm Fabry-Perot polymer etalon structure serving as an optoacoustic detector array. Pulse-echo signals display center frequencies above 30 MHz with bandwidths of at least 40 MHz with a 30 µm generation element and a 20 µm detection element. A theta-array, one of the lowest order array structures for 3-D imaging separating transmit and receive elements, is emulated by mechanically scanning the generation laser beam through eight 1D elements separated by 200 µm and the detection laser beam along an annular array of 400 elements separated by 19.6 µm. The reconstructed images capture the imaging targets with reasonable accuracy. Acoustical characterization, together with preliminary imaging results, demonstrate the device's capabilities and potential for 3D ultrasound imaging.

Portions of the work contained in Chapter 5 have been submitted to IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control for publication. The relevant citation is:

> Y. Hou, S. Ashkenazi, S. W. Huang, and M. O'Donnell, "All-optical theta-array for 3D ultrasound imaging," submitted to IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control.

The main contributions of this work are:

- Designed and developed a theta-array imaging system integrating black PDMS and etalon structures.
- 2. Obtained the first ever 3D ultrasound image using all-optical ultrasound from an integrated device.

6.2 Immediate next steps

The ultimate goal is to develop efficient optoacoustic transmitters and sensitive optoacoustic detectors, and integrate them into the same device for 2D high-frequency arrays, possibly onto the tip of optical fibers for practical biomedical imaging applications. Therefore, both optoacoustic generation and detection must be further optimized, while integration mechanisms and array expansion configurations must be carefully investigated. As immediate next steps, the following should be studied:

A. Transduction efficiency

For the gold nanostructure film, the optical absorption at resonance wavelength is merely 30 %. Optoacoustic transduction efficiency can be improved by increasing the optical absorption, already 100 % for black PDMS films. This can be done by changing the shape, thickness, and arrangements of the gold nanoparticles. For example, theoretically, doubling the thickness of each gold nanoparticle alone will significantly improve and nearly double its optical absorption. Other patterns of gold nanoparticle arrangements can also be fabricated by modifying the nanoimprint process, and the resultant new structures might yield ideal optical absorption properties. For instance, nonsquare (diamond) shaped gold nanoparticle arrays can be fabricated, as illustrated in figure 6.1 in the SEM picture of a top view. A general model, including both altered geometry and coupling between nanoparticles in a periodic array, must be developed to help optimize these structures for photoacoustic applications. Once optimal structures are defined, fabrication methods must be adjusted to approach these ideal structures.

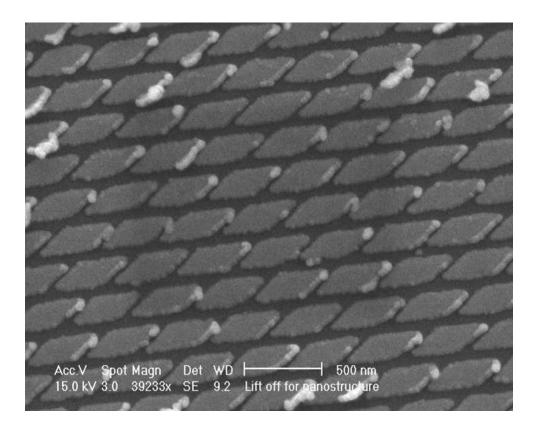


Figure 6.1: SEM photo of 2D diamond shaped gold nanoparticle arrays

Another idea is to disperse gold nanoparticles into PDMS instead of confining them within a thin layer. Figure 6.2 illustrates this approach. It will not only improve transduction efficiency, but also the thermal damage threshold, consequently increasing the ultimate acoustic power possible. Large concentrations of gold nanoparticles dissolved in solvents have been widely studied and well documented [96, 100]. These gold nanoparticles show dramatic contrast in optical absorbing capabilities at and far from resonance wavelengths, with the ratio of optical absorbance exceeding 20. To achieve the same level of optical absorbing abilities as the gold nanostructure described in chapter 2, the concentration of gold nanoparticles dispersed in PDMS should be at least $10^{13}/m^2$, which is not conveniently available yet. In addition, other noble metals should be considered. For example, silver nanoparticles are more efficient optical absorbers than gold ones.

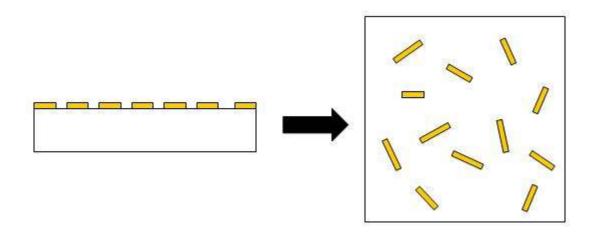


Figure 6.2: Illustration of dispersing gold nanoparticles into PDMS

On the other hand, making thinner black PDMS films is still a valid option, where reducing the viscosity of the mixture of carbon black and PDMS is the key. Further reducing the percentage of carbon black in the film is the most straightforward option, which causes non-uniformity in terms of film thickness according to preliminary tests. However, to a certain extent, the uniformity of the film can be compromised because equal ultrasound generation at different array elements is not overwhelmingly critical in imaging applications. Strongly absorbing dye materials, especially wavelength-selecting dyes, are attractive if available because of integration considerations.

B. Etalon material and uniformity

The etalon bandwidth has been improved by reducing the bulk material thickness in this thesis, and currently, a thickness of 5.9 µm represents the best balance between sensitivity and bandwidth. The etalon bulk material is currently SU-8, mainly because of its convenience for fabrication and thickness control. According to equation 3.12, the sensitivity can be improved by reducing the Young's modulus of the polymer. Therefore, exploring other polymer materials is needed to optimize performance. Plus, in the future, the etalon needs to be fabricated on fiber tips, and spin-coating might not be suitable in that case. This means that evaporative deposition is likely to replace spin-coating, making SU-8 an unfavorable material. The quest for new polymer materials will focus on smaller Young's modulus and compatibility with thermal evaporative deposition techniques. Meanwhile, PDMS etalons need to be optimized for the integrated device. Sensitivity and bandwidth changes with bulk layer thickness and gold layer thickness should be investigated for optimal performance of the integrated transducer described in chapter 4.

As discussed in section 3.5, etalon uniformity is extremely important. Currently, the etalon is uniform enough over a 1D distance of 2 mm, but unfortunately not over 2D regions of millimeters. The bottleneck might be the spin-coating fabrication process, which is not the most desirable method if uniformity is the primary concern; again, thermal evaporation might be the solution.

C. Crank up the power

In our experiments, the SNR of the pulse-echo signal from the all-optical transducer element is mainly limited by the incident optical power. We claimed that the SNR can be increased by at least 20 dB, but were not able to verify due to limited laser power. High power fiber lasers are commercially available, and having access to one will be a huge boost toward demonstrating the real capabilities of all-optical ultrasound transducer. Clearly, a higher SNR must be demonstrated before optoacoustics can really out-muscle piezoelectrics and CMUTS for practical biomedical imaging applications.

D. Theta-array considerations

The limited number of optoacoustic generation elements severely reduces image quality in our theta-array experiments, and again, this is due to the unavailability of higher laser power. Currently, 1000 averages are needed for each detection element. Higher laser power would enable single-shot recording at each array element, enabling a significantly larger number of generation elements with less than 20 µm separation for our theta-array configuration, which undoubtedly will significantly reduce artifacts and greatly improve image quality. This approach is illustrated in figure 6.3. On the other hand, simultaneous ultrasound detection from all array elements at each firing of an individual generation element would decrease imaging time significantly, making real-time imaging possible. For example, when a 5 kHz laser is used with 100 transmit elements, and at each firing, 400 receive elements record single-shot signals simultaneously, acquiring full 3D volume data only takes 30 milliseconds, yielding volume frame rate of 50 Hz.

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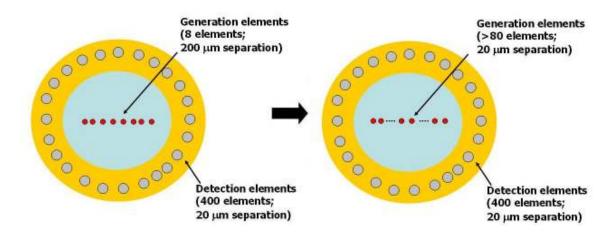


Figure 6.3: Illustration of increasing the number of generation elements in the theta-array system

The position control system needs to be upgraded as well. Right now, the position of the generation element is controlled by a manual motor, which makes the exact position somewhat unpredictable. This has a huge impact during image reconstruction, as accurate beamforming requires precise knowledge of the relative coordinates of all generation and detection elements. As a side note, the laser pulse is reflected from a dielectric mirror, thus the angle of the mirror is also critical and needs to be calibrated. Overall, direct observation and control over the exact position of all elements is essential to achieve high image quality.

E. Expansion to arrays

The original motivation for exploring optoacoustic transducers is to build 2D high-frequency arrays, which have not been achieved with other technologies. At this stage, a single element all-optical ultrasound transducer has been developed, and array capabilities have been demonstrated by mechanically scanning the optical beams or the imaging target. As discussed in sections 3.5 and 4.5, simultaneous generation and

detection are strongly desired for real-time imaging applications, and 2D optoacoustic arrays must be built to achieve the ultimate goals. Three specific tasks lie ahead: first, appropriate fiber bundles, as well as convenient laser coupling techniques, must be studied as the optical delivery system; second, optoacoustic devices, especially the etalons, must be uniform enough over 2D regions of millimeters, and preferably can be fabricated on fiber tips; third, photodetector arrays must be designed and fabricated for simultaneous acquisition of reflected probing optical beams.

F. Stability issues

The thermal damage threshold has been measured for all devices described in this work. However, it's worthy to note that the threshold is highly dependent on the repetition rate, wavelength, and exposure time. Plus, occasionally a big chunk of absorbing particles, or even some dust, can cause quick damage.

Another concern is that the optical wavelength of the etalon can shift by a few nanometers after being placed in water. And in some rare cases, the structure was damaged after being immersed in water for a few hours, which causes problems even though the device is unlikely to make contact with water for more than a few minutes in real-time applications. Therefore, further studies of stability are needed to move toward clinical environments

G. Another integration configuration

Another convenient and realistic method for an integrated optoacoustic transducer is presented here. The key part is a PDMS layer containing wavelength-sensitive dye, or high concentrations of gold nanoparticles. This PDMS layer will absorb optical energy within a specific wavelength range, and transmit optical beams at other wavelengths. The structure consists of a dielectric mirror on top of a glass substrate, where this mirror transmits the PDMS layer's absorption wavelength, and reflects other wavelengths. The PDMS layer and a conventional gold mirror will be fabricated on top. The generation beam will transmit through the dielectric mirror, and will be absorbed by the PDMS layer for ultrasound generation, while the detection beam is used to probe the etalon formed by the gold mirror, the dielectric mirror and the PDMS layer. This is feasible due to the PDMS film's high contrast in optical absorbing abilities at different wavelengths. The most urgent step for fabricating this device is to find wavelength-sensitive dye materials, or disperse large concentrations of gold nanoparticles into PDMS.

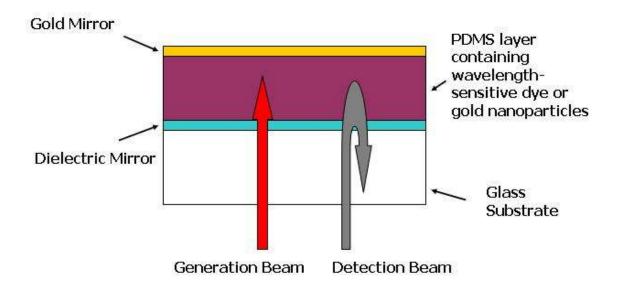


Figure 6.4: Proposed structure of another type of optoacoustic transducer

6.3 Outlook for all-optical ultrasound transducers

The ultimate vision of the all-optical ultrasound transducers is to develop 2D high-frequency arrays that can fit onto the tip of a catheter, guidewire or needle probe. Major components include one high-power CW laser for ultrasound detection, one pulsed laser for ultrasound generation, a photodetector array system for simultaneous data acquisition, a fiber bundle system for optical beam delivery, and the all-optical transducer at the front-end. The transducer is a thin film containing multiple layers of gold and polymers, and is simple, cheap and disposable, which makes it convenient for clinical applications. A demonstration of the scheme is shown in figure 6.5. This approach will enable real-time ultrasound imaging with resolution better than 50 µm.

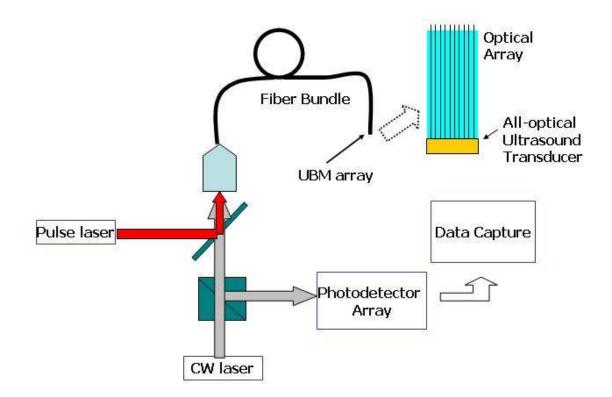


Figure 6.5: Demonstration of an ultrasound imaging system using all-optical ultrasound transducers

Another appealing prospect is to combine this high-resolution imaging array with therapeutic systems. Either laser therapy or ultrasound therapy using high-intensity focused ultrasound (HIFU) can be conveniently embedded in this system. The combined system will probe targeted tissues within the human body, provide real-time images, and then utilize either optical energy or ultrasonic energy to destroy undesired targets like tumors or even individual cancer cells. Optical energy can be extracted from the pulsed laser beam used for ultrasound generation. Meanwhile, an acoustic lens can be added to focus the generated acoustic waves for therapeutic purposes. These approaches are demonstrated in figure 6.6.

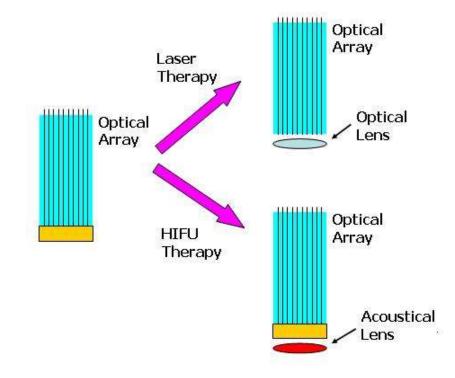


Figure 6.6: Scheme of combining the optoacoustic imaging array with either laser therapy or HIFU therapy

In summary, the ultimate all-optical ultrasound imaging system will contain a 2D high-frequency optoacoustic array for real-time 3D ultrasound imaging, as well as therapeutic functions utilizing either laser or HIFU therapy. This device will significantly expand the frontiers of medical ultrasonics, and see extensive useful applications in clinical environments.

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