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A PMUT Based Photoacoustic System as a Microfluidic Concentration Detector

Kaustav Roy^a, Vijayendra Shastri^b, Akshay Kumar^a, Jagi Rout^a, Isha^c, Kritank Kalyan^d, Jaya Prakash^{*c}, Rudra Pratap^{*a}

^aCentre for Nano Science and Engineering, Indian Institute of Science, Bangalore – 560012, KA, India; ^bDepartment of Precision and Microsystems Engineering, TU Delft, Mekelweg 5, 2628 CD Delft, Netherlands; ^cInstrumentation and Applied Physics, Indian Institute of Science, Bangalore – 560012, KA, India; ^dSingh Center for Nanotechnology, University of Pennsylvania, Philadelphia – 19104, PA, U.S.

*All correspondence should be addressed to jayap@iisc.ac.in and pratap@iisc.ac.in

ABSTRACT

We report on the development of a novel piezo-MEMS based optofluidic platform to detect the concentration of various species dissolved in a fluid. This platform employs piezoelectric micromachined ultrasound transducers (PMUTs) to work as a photoacoustic receiver, receiving ultrasound from fluid targets present in microfluidic channels while illuminated with a nanosecond pulsed laser. We fabricate both the PMUTs and the microfluidic channels and subsequently use them for the experiment. We also show the capability of PMUTs as a general photoacoustic receiver and demonstrate its signal-to-noise characteristics (~31) and its wide fractional bandwidth (~73%).

Keywords: PMUTs, microfluidic, photoacoustic, microfabrication, piezo-MEMS, concentration detection

1. INTRODUCTION

Liquids are ubiquitous and are regularly used by various industries such as pharmaceuticals, oil and gas, petrochemical, automobiles, process industries, etc., for different utilitarian purposes. The dye industry is one of the important industries that manufacture and uses liquids, which makes dye for various applications such as in food, textile, photography, leather, etc. and uses the solvent in which the dye is dissolved in order to tune the concentration of dye. Also, it is reported that the concentration of the colorant in the dye-solvent mixture decreases with time¹. Due to such a problem, tracking the dye concentration becomes vital and there exists few literature that addresses such problems².

The development of nanofabrication techniques has led to the creation of several microscale electron devices. Nanotechnology has also enabled compact fluid devices such as microvalves³, micropumps⁴, microfluidic flow sensors⁵, microneedles⁶, micronozzles⁷, etc.

The coupling of light with microfluidic devices has led to the creation of a new research domain called optofluidics⁸. The advantages of the fluidic side such as – controlled diffusion of miscible fluids, the creation of smooth immiscible fluid interfaces, ability to work as an excellent transport medium and the advantages of the optical side such as the availability of highly sensitive optical sensing techniques, localization of light to spot at the sub-micron scales, ability to manipulate objects has proven to be fascinating enough to create state of the art devices such as fluid lenses⁹, optofluidic microscopes¹⁰, resonators¹¹, tweezers¹² and much more. With the advent of pulsed/modulated light sources, the field has further evolved, acting on the fluid contained in the microfluidic channel, producing sound^{13–21}. This phenomenon of making sound from any object by illuminating it with pulsating light is called photoacoustics, which has several applications in diverse fields^{22,23}. The electromagnetic properties of light in conjunction with the penetrability of sound have made it possible to penetrate deeper and obtain information that is functional to the target thus probed. Pulsed LEDs and MUTs^{24–37} have revolutionized the field further, thereby creating wearable photoacoustic devices.

In this work, we have fabricated piezoelectric micromachined ultrasound transducers (PMUTs) using functional thin-film piezoelectric material and the nanofabrication tools and have demonstrated their use as a photoacoustic receiver. Further, we have also fabricated PDMS-glass-based microfluidic channels and have used them as soft conduits to contain fluid targets. We have subsequently built an experimental setup using a pulsed laser, a microfluidic channel, and PMUTs, to

Photons Plus Ultrasound: Imaging and Sensing 2022, edited by Alexander A. Oraevsky, Lihong V. Wang Proc. of SPIE Vol. 11960, 1196013 · © 2022 SPIE · 1605-7422 · doi: 10.1117/12.2608934 demonstrate the capability of PMUTs as photoacoustic receivers. PMUTs were thoroughly characterized and were found to be suitable candidates for photoacoustic applications. Next, we have used several mixtures of blue ink with water as fluid photoacoustic targets and demonstrated the capability of the PMUT to detect the change in concentration as the levels of ink in the DI water was varied, thereby concluding that PMUTs can be successfully used as concentration detectors.

2. MATERIALS AND METHODS

The following section describes the PMUT in detail, the microfluidic channel that has been used in work and the photoacoustic experimental setup that have been built for conducting the experiments.

2.1 PMUTs

Piezoelectric Micromachined Ultrasound Transducers (PMUTs) are piezoelectrically driven microelectromechanical acoustic devices which are fabricated following the micro/nanofabrication approaches. Structurally, PMUTs are micro-plates/membranes composite made up of five different layers stacked on top of each other and backed by an acoustic cavity as shown in the schematic (Fig. 1.(a)). The PMUTs used for the photoacoustic experiments were designed and fabricated at the National Nanofabrication Centre (NNFC), Centre for Nano Science and Engineering (CeNSE), Indian Institute of Science (IISc), Bangalore. These devices comprise thin film lead zirconate titanate (PZT), which serves as the active piezoelectric layer and either drives the diaphragm or gets dynamically charged when the diaphragm vibrates. Thus, a PMUT can work as a transmitter, a receiver, or even a transceiver. The PZT thin film was deposited on a silicon-on-insulator (SOI) substrate having a constant device layer thickness of 10 µm. PMUTs can be engineered by varying the diaphragm size, changing the stack thickness, and varying the interlayer fabrication stresses. The PMUTs fabricated for



Figure 1. Depiction of the piezoelectric micromachined ultrasound transducers fabricated at NNFC, CeNSE. (a) A 3d, three-quarter schematic of a typical single cell PMUT. (b) The PMUT die used for this work. (c) Optical Microscopic image of the PMUT die consisting of twenty-five single cell PMUTs shorted together to form an element. Each one of the white outlined circles represents a single cell PMUT. (d) The PMUT die packaged to a ceramic package.

this work were circular in shape, having a diameter of 250 µm, and were fabricated on a silicon-on-insulator (SOI) substrate having a device layer of 10 µm. Fig. 1.(b),(c) demonstrates the PMUT die used for the work, which consists of twenty-

five single-cell PMUTs shorted together. The PMUT after fabrication was wire-bonded to a pin grid array ceramic package and was coated with parylene-c for enhancing the noise immunity, as shown in Fig. 1.(d).

2.2 Microfluidic Channels

The microfluidic channel was fabricated at the NNFC, CeNSE the using the following steps as described below. The process starts with a silicon mold which is RCA cleaned (Fig. 2.(a)) followed by the deposition of silicon dioxide, which serves as a hard mask in the subsequent etching steps (Fig. 2.(b)). The resulting substrate was patterned with a thick AZ4562 photoresist (Fig. 2.(c)) followed by the etching of oxide in the non-patterned areas (Fig. 2.(d)) using the reactive ion etching. This was followed by the ashing of the photoresist (Fig. 2.(e)). The resulting substrate was then timed etched using deep reactive ion etching to obtain a certain desired depth (Fig. 2.(f)) to obtain the microfluidic mold. PDMS mixed with the curing agent (10:1) was subsequently poured on the mold (Fig. 2.(g)) and was cast at 120 °C for half an hour. The



Figure 2. Depiction of the elaborate details about the microfluidic channel used for the work (a) The fabrication process starts with a silicon wafer (DSP) (b) Oxide grown on the wafer (c) Patterning of photoresist on the resultant substrate (d) RIE etch of the exposed oxide (e) Photoresist ashing to leave a hard mask of oxide on silicon (f) DRIE etch (timed) to level down silicon for a thickness of 50 μ m thereby creating the silicon mold (g) PDMS mixed with the curing agent (10:1) being poured on the mold (h) Casting of PDMS by heating it at 120°C for half an hour (i) Stripping off the hardened PDMS from the mold and bonding it to the glass slide by the help of plasma (j) The microfluidic channel filled with blue ink used for the experiment.

casted solid PDMS was carefully stripped from the mold (Fig. 2.(h)) and was bonded to a glass slide using a plasma etcher from Harrick plasma Inc (Fig. 2.(i)). The bonded microfluidic device was baked at 120 °C for an enhancement in the bonding strength. Fig. 2.(j) demonstrates the typical bonded microfluidic device (cross-section dimensions: width – 1000 μ m, depth 50 μ m) used for the experiment.

2.3 The Photoacoustic experimental setup

A schematic diagram of the photoacoustic experimental setup is demonstrated in Fig. 3. (a). It consists of a tunable OPO nanosecond pulsed laser (SpitLight 1000 from InnoLas Lasers GmbH) capable of working in the wavelength range – 660 nm to 2500 nm, having a pulse width of 7 ns and a repetition rate of 30 Hz. The light from the laser illuminates the microfluidic target attached to the wall of a custom-made glass fish tank at a wavelength of 660 nm. The target absorbs the time-variant light and emits acoustics which is received by the PMUT. The voltage signal from the PMUT was amplified using a low noise voltage-controlled amplifier (VCA 2615, Texas Inc.) before feeding it to the oscilloscope (Mixed Signal Oscilloscope, Tektronix Inc.), which was in turn fed to the computer for real-time digital signal conditioned display. The PMUT was mounted with an x-y linear stage (CONEX-TRA25CC, Newport Inc.) for precision control of its position. The stage was controlled using dedicated software from the computer. The computer also controls the pulsed laser for accurate trigger generation, which is subsequently synchronized to the oscilloscope for obtaining accurate

dynamic measurements. Fig. 3. (b) demonstrates a zoomed-in view of the PMUT while it was receiving the photoacoustic signal from a microfluidic target when illuminated with a 660 nm laser pulse.



Figure 3. The photoacoustic experimental setup. (a) A two-dimensional schematic of the setup used for the work (b) The working experimental setup demonstrating the PMUT receiving signal and passing it on to the low noise amplifier (LNA), the laser light, the microfluidic channel with its inlet (I) and outlet (O).

3. RESULTS

This section contains the result of the photoacoustic characterization of the PMUT used for the work as well the demonstration of the PMUT as a concentration detector. For all the following experiments, the fluence was kept within the ANSI safety limits (half of the ANSI limit, which is 22 mJ/cm²)

3.1 Photoacoustic characterization of the PMUT

The PMUT used for this work after being packaged was characterized as an acoustic detector by using the above-mentioned experimental setup. A pencil lead (circular cross-section) having a diameter of 500 µm was used as the test target for



Figure 4. Photoacoustic characterization of PMUT, showing the A-line photoacoustic signal and its FFT received from a pencil target at a distance of 5 cm

photoacoustic emission, while the laser-illuminated it with pulsed nanosecond light and was kept 5 cm away from the PMUT's surface. The data obtained was plotted and reported in the Fig. 4. The peak-to-peak voltage received from the PMUT as observed in the oscilloscope after a gain of 45 dB is found to be 155 mV. This corresponds to a raw signal of 2.76 mV from the PMUT. The signal-to-noise ratio (SNR) is found to be 31. A Fast Fourier Transform (FFT) was applied on the resulting A-line data and is plotted in the Fig. 4. The central frequency is found to be 1.06 MHz in water. The fractional -3 dB bandwidth is also calculated and is found to be 72.8%.

3.2 Concentration detection using the PMUT

The PMUT was subsequently used to receive the photoacoustic signal emitted by the microfluid target containing test solutions. The test solutions were prepared by dissolving various volumes of ink in water to make solutions of several ink concentrations -10% to 80% in the intervals of 10%. The test solutions were injected into the channel one after the other,



Figure 5. Working of PMUT as a fluid concentration detector. Graph showing the variation in the received voltage magnitude with the variation in the ink-in-water concentration.

and the peak-to-peak voltage signal as observed from the PMUT situated at 2 cm after a 45 dB of gain is plotted and is reported in the Fig. 5. The signal strength was observed to be increasing linearly from 10% to 50%, after which the trend saturates. This might be because of the fact that at a higher concentration of the ink, any fresh addition in the number of ink species does not necessarily create a proportional increase in light absorption, thereby limiting the magnitude of the acoustic pressure, making the peak received voltage to saturate. Alternatively, it might also be due to nonlinear effects due to saturation of absorption coefficient or usage of large fluence values^{38,39}. The observations obtained from Fig. 5 indicate that PMUTs can be used as fluid concentration detectors. Earlier works have shown photoacoustic spectroscopy with bulk transducers ⁴⁰, which could also be explored using the PMUTs.

4. **DISCUSSION**

Thus, in this work, we have reported on the development of a new platform that employs PMUTs as photoacoustic receivers, to detect ultrasound generated from microfluidic targets while illuminated with pulsed light. The PMUTs were photoacoustically characterized, and their performance was found quite impressive, having a signal-to-noise ratio (SNR) of 31 and a fractional bandwidth of \sim 73%. It was also found that PMUTs were capable of detecting the change in concentration of ink in water successfully, indicating that PMUTs can be one of the ideal candidates for photoacoustic imaging and sensing applications.

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