Picolitre acoustic droplet ejection by femtosecond laser micromachined multiple-orifice membrane-based 2D ejector arrays

U. Demirci and A. Ozcan

Described is a new method of generating droplets by using the higher-order resonance mode shape displacement of a two-dimensional (2D) silicon nitride membrane array with multiple orifices, fabricated by a femtosecond laser. This higher-order resonance ejection method increases the flow rate per membrane on a 2D ejector array and introduces the capability to eject from various locations on a membrane. Combined with the simplicity and the functionality of femtosecond laser micromachining, the novel devices are especially well suited for continuously generating picolitre droplets for various applications in biotechnology and the semiconductor industry.

Introduction: A reliable, rapid method of dispensing picolitres of various fluids has emerged as an attractive technology. Droplet generation techniques have been employed in fields such as inkjet printing [1], semiconductors [2–5] and biotechnology [4–7]. For example, bio-arrays for drug testing could easily utilise controlled droplet generation techniques; writing DNA arrays by drop-on-demand ejection is another example; small droplets could also be used for drug delivery [7].

There are various methods for generating droplets by using acoustic waves [2–4, 6, 7]. In this Letter we describe a new generation method using the higher resonance mode shape displacement of a 2D silicon nitride membrane array with multiple orifices, fabricated conveniently by a femtosecond laser. This method also increases flow rate per membrane on a 2D ejector array. Moreover, the orifices could be placed at various distances from the centre of the membrane and could be selectively actuated by changing the mode shape of the membrane via changing the frequency of actuation. This feature would allow flexibility in the design of practical devices utilising this technology.

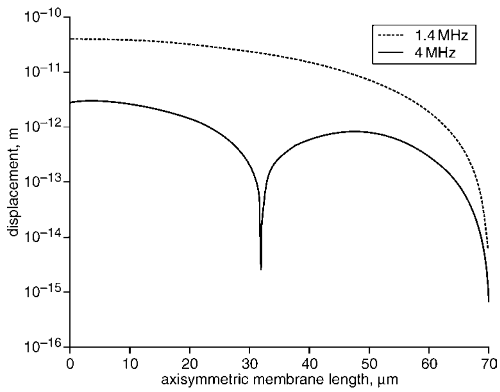
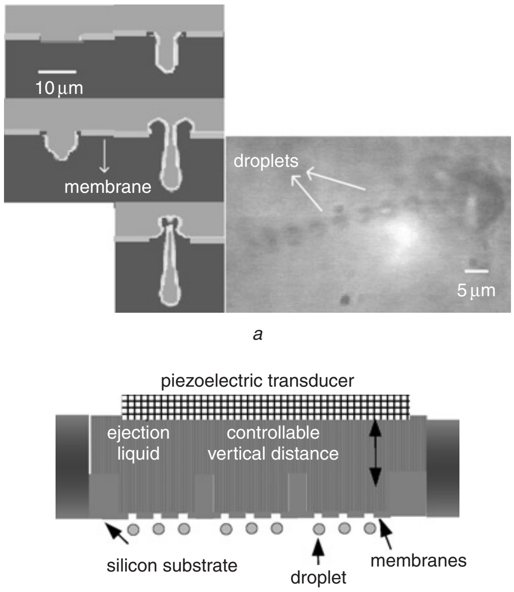


Fig. 1 *3D finite element simulation demonstrating resonant mode shapes of 140 µm-diameter clamped circular membrane*

Theory: In the design of the single orifice devices, the location of the orifice is placed at the centre of the membrane, where the displacement on the membrane is maximal. As the membrane resonates at higher modes, there are other high displacement locations on the membrane surface. The location of the multiple side orifices located around the central orifice has to coincide with these secondary maximum displacement locations for ejection at low acoustic power. The displacement profiles at two resonance modes are shown in Fig. 1 for 140 µm-diameter 2.1 µm-thick Si_xN_y membranes using a 3D finite element method (ANSYS 5.6). The simulations revealed that, as expected, the displacement is highest at the centre of the membrane for resonance mode shapes of 1.4 and 4 MHz. Also, displacement was large at 45 µm away from the centre of the membrane at 4 MHz. The theoretical locations of the orifices on the membrane for acoustically actuated displacement-facilitated ejection were determined from these simulations, i.e. secondary orifices were fabricated 45 µm away from the central orifice. The simulations assume that the presence of orifices on the membrane during resonance calculations is negligible. This assumption is a reasonable one since for a device that has five orifices, the total area of the orifices constitutes only ~2.5% of the total area of the membrane, which suggests that the resonant frequency of structure should not change significantly.

The droplet formation from an orifice as a result of membrane displacement is simulated by using the finite element method as shown in Fig. 2*a*. The same membrane displacement aided droplet generation process takes place at each of the multiple orifices. The simulation result of a 5 µm-diameter droplet generated from a 10 µm-diameter orifice as a result of membrane displacement at 1.2 MHz is demonstrated to agree with experimental ejection from a 10 µm-diameter orifice obtained by stroboscopic imaging, as shown in Fig. 2*a*. This simulation assumed a membrane displacement activated by a uniform pressure field on the membrane and the droplet was ejected as a result of the membrane displacement.



b

Fig. 2 *Finite element simulation of 5 µm-diameter droplets at 1.2 MHz generated from 10 µm-diameter orifices, and illustration of generation of droplets by acoustic plane waves from multiple orifice ejector array*

a Finite element simulation of 5 µm-diameter droplets at 1.2 MHz generated from 10 µm-diameter orifices
Simulation agrees with 5 µm-diameter droplets ejected at 1.24 MHz from 10 µm-diameter orifice on Si_xN_y membrane based ejector array. Results of stroboscopic imaging of droplet ejection also shown
b Illustration of generation of droplets by acoustic plane waves from multiple orifice ejector array

Design and fabrication: A flexurally vibrating circular membrane clamped at the periphery on one of the faces of a cylindrical fluid reservoir constitutes a single element of an ejector array. This is replicated in a 2D geometry in order to form an array of ejectors as shown in Fig. 2*b*. Orifices are etched into the circular membrane so that droplets are ejected when the membrane is actuated by acoustic waves from an ultrasonic immersion transducer (Panametrics, MA) that travel through the fluid reservoir as shown in Fig. 2*b*.

The devices were built using standard micromachining methods [3]. The multiple orifices could be etched into the membranes using lithographical methods. However, we took a simpler approach: the orifices on the membrane were built using femtosecond laser micromachining [8, 9]. In the femtosecond laser fabrication process, the samples were positioned on a computer-controlled 3D rotation stage, with a movement precision of ~1 µm. The femtosecond laser (Ti:Sapphire, 800 nm, full-width-half-maximum of ~300 fs, energy ~1 µJ, repetition rate ~10 kHz) was focused onto the membrane surface using microscope lenses (N.A. ~ 0.2–0.6) down to various spot sizes ranging from 2 to 10 µm. In general, a larger orifice size than the spot size can easily be fabricated by moving the sample plane laterally in and out of the focus of the femtosecond laser. For fabricating holes with sizes less than the spot size of the laser beam, a single shot of the femtosecond laser (with reduced energy levels) is used by means of a fast electrical shutter. A membrane with four symmetric orifices and a central orifice is demonstrated in Fig. 3. Secondary orifices were fabricated at 35, 45 and 60 µm away (for three different membranes) from the central orifice in order to explore the effect of the mode shape on the ejection.

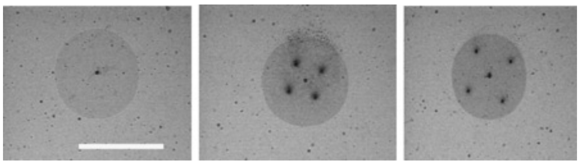


Fig. 3 *Micromachined 10 µm-diameter centre orifice surrounded by laser micromachined 10 µm-diameter multiple side orifices on 160 µm-diameter 2.1 µm-thick Si_xN_y membranes (side orifices placed 35 and 45 µm away from centre of membrane)*

Results and discussion: We observed multiple simultaneous ejections from all orifices of the membrane at the geometry where the multiple orifices were placed 45 µm from the central orifice without increasing the acoustic wave amplitude off the transducer at 3.96 MHz for isopropanol ejection *at higher resonance modes*. These locations were predicted by the theory (Fig. 1) where the membrane displacement is second highest. We also observed ejection from the multiple orifices that are placed 35 and 60 µm from the central orifice by increasing the acoustic radiation power from the transducer to higher levels, tilting the transducer to actuate asymmetric resonance modes of the membrane. These kinds of ejection mechanisms were mostly dominated by the acoustic radiation pressure breaking the surface tension forces and may be unstable [3].

The theory also predicts a membrane resonance at 1.4 MHz, whereas we observed ejection at 1.08 MHz *for the first resonance of the membrane*. This difference may be due to fabrication errors during the deposition of the Si_xN_y thin film, which later forms the membranes. We observed the membranes to suffer from bubble formation in the Si_xN_y layer resulting in a non-uniform membrane material. This issue can be resolved by using a silicon-on-insulator wafer to silicon wafer bonding and by creating uniform single crystal silicon membranes that are 1 mm thick with a standard deviation variation of 0.024 mm over an array [3].

The increase in the number of the orifices is expected to increase the droplet generated per second per membrane. The volume of a single 5 µm-diameter droplet is 65.5 fL. A membrane with a single orifice operating at 1.08 MHz generating 5 µm-diameter droplets has a flow rate of 71 nL/s. A 20 × 20 2D ejector array ejecting from all array elements would have a flow rate of 28.3 µL/s. A multiple orifice ejector array would eject at a flow rate that multiplies the above values with the number of orifices.

Conclusion: A new method of droplet ejection utilising the higher-order resonance mode shapes of a thin membrane is demonstrated using femtosecond laser micromachined 2D ejector arrays. The method increases flow rate per membrane on a 2D ejector array, are introduces the capability to eject from various locations on the membrane, which are highly desirable in most applications utilising microdroplet technologies. We believe these devices will find novel application in fields such as drug testing and delivery in biotechnology.

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