Flow-through piezo-electric microdispenser

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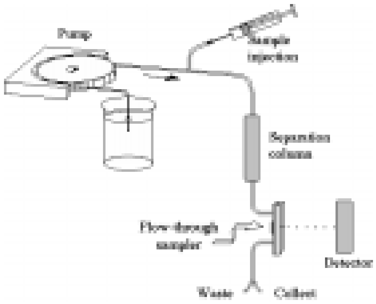
Introduction

The use of ink-jet-type droplet generators for sample handling in chemistry has increased over the last years following the demand for devices capable of handling minute liquid volumes. The piezo-electric devices have become the most commonly used since they introduce no heating of the liquid as in the bubble-jet types.

Common to all the previously used devices is that they are of a single-end type meaning that they have one liquid inlet from a reservoir, and one outlet, the droplet nozzle. This implies that they are impossible to use in a flow-through set-up. To circumvent this drawback we are developing a flow-through piezo-electric dispenser that easily can be inserted in the flow-line of a system for on-line sampling. The flow-through option also enables rapid switching of liquids in a single dispenser.

Dispenser inserted in a flow-through system for on-line sampling & detection



Examples of application areas where a flow-through microdispenser is of interest

The major areas today are found in the field of biochemistry:

Sample injection in capillary electrophoresis

Reagent supply in high-throughput screening for drug discovery

Sample supply for mass spectrometry

Depositing of samples for enrichment

Sampling in a liquid chromatography system

Fabrication and operation

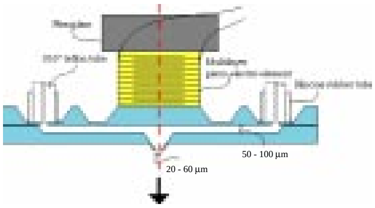
The flow-through microdispenser is micro-machined in two silicon dice. The bottom piece has a flow channel with a pyramid shaped cavity etched in the center. After n-doping this side of the wafer, the opposite side of the wafer is etched, removing material until the pyramid cavity breakes through. The etching proceeds until the desired hole size is accomplished. A passivation voltage (pn-etch stop) is then applied to the doped side (channel side) of the wafer and thus only the undoped silicon is further removed from the structure leaving a pyramid shaped nozzle extending from the wafer surface.

The top piece has flow inlet and outlet etched through the wafer and a silicon push-bar integrated on a thin silicon membrane that is defined by pn-etchstop. The two pieces are subsequently either glued or bonded together to form the flow-through cell.

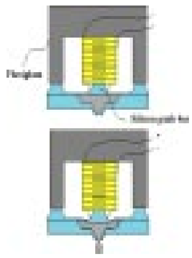
Silicone tubes are glued to the inlet and outlet, allowing standard teflon tubing to be inserted to the vicinity of the silicon surface leaving a minimum of dead space.

The displacement of a piezo-ceramic multilayer element, when a voltage pulse is applied, is coupled to the push-bar. A pressure pulse is created in the flow-through channel and a droplet is ejected through the tiny nozzle. The piezo-element is being developed within the project by the group at the Department of Material Science, Uppsala University, Sweden.

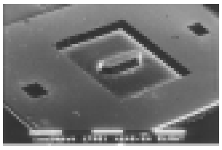
Lengthwise cross-section view of the dispenser



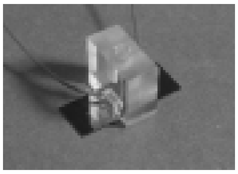
Principle for the actuation of the dispenser



SEM-image of the push-bar



Assembled actuator side of the dispenser



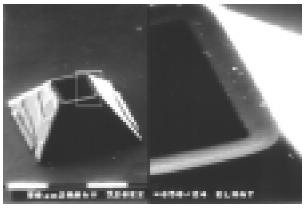
The piezo-electric element is glued (epoxy) between the plexiglass backing and the actuator silicon die. The dimensions of the silicon plate are 13 mm x 6 mm.

Nozzle

The pyramid shaped nozzle extending 70 µm from the silicon surface greatly improves the dispenser performance.

The thin walls (≈ 8 µm) minimize the nozzle front surface wetting. This eliminates problems with misdirected droplets commonly caused by liquid or particle deposits at this surface.

Pyramid-shaped nozzle defined by 8 µm silicon walls



The size of the orifice is 40 µm x 40 µm.

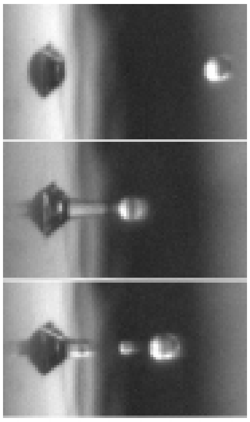
Typical specifications

Flow-through channel dimensions:
8 mm x 1 mm x 50 µm (L x W x H)
Channel volume:
400 nanoliter
Orifice size:
40 µm x 40 µm
Droplet volume:
65 picoliter
Max droplet frequency:
< 10 kHz
Voltage pulse parameters:
< 10 V, 100 µs

Control of droplet formation

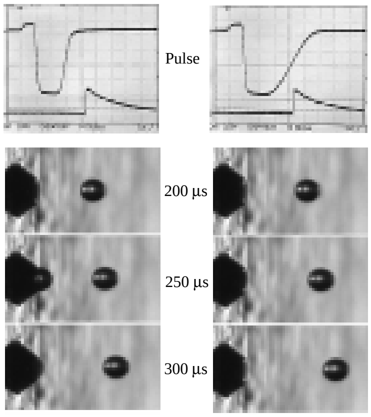
The stable formation of droplets is highly dependent on the electrical pulse that drives the piezo-electrical actuator. To be able to handle a wide range of liquids, with respect to properties as viscosity and surface tension, one must firmly know how to adjust the drive pulse. The pulse must also be adjusted to harmonise with the design of the dispenser channel, which affects the drop formation through resonance wave phenomena. A short “fill before fire” pulse, with inverse amplitude, has proved effectiveness in reducing a tendency to generate small unwanted satellite droplets. The resonance wave can be compensated by slowing down the pulse return.

Satellite droplet formation



Too high pulse amplitude causes unwanted small satellite droplets to be generated following the main droplet.

Pulse shape control

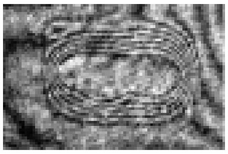


The images illustrate how an unwanted resonance in the system (left images), appearing at the nozzle 250 µs after actuation, may be compensated by slowing down the pulse return and extending the pulse length.

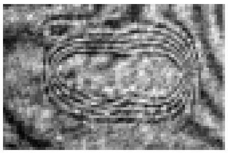
Interferometry

A common Michelson interferometer set-up is used to retrieve motion data from the membrane of the dispenser. Since the silicon surface of the membrane is a good reflector, it can be used directly as one of the mirrors in the optical arrangement. The slightest unevenness in the surface will lead to a interference pattern of fringes, due to the relative difference in optical pathlength, when observed through the interferometer. A perfectly flat surface will also generate such fringes when at an angle not perpendicular to the optical axis. By studying the change in location of these fringes, during actuation, one can resolve the magnitude of the movement of the membrane. Each shift from one fringe to another with the same intensity corresponds to a half light wavelength. The data obtained is then used when evaluating the quality of piezo-electric actuators and as input for finite element analysis of dynamic properties of the dispenser.

Movement of the actuator wall

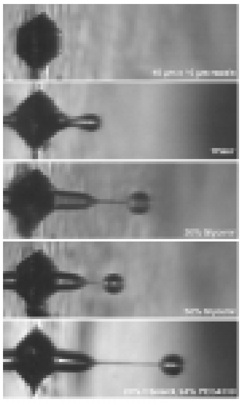


The interference pattern shows a static displacement of the push-bar of around 1.5 µm due to contraction of the epoxi resin.



The resulting movement when 10 V is applied to the piezo-element is around 350 nm, compared to the previous image.

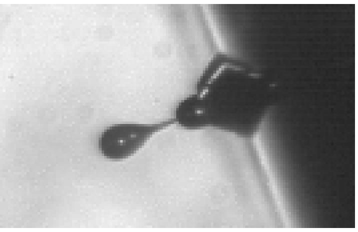
High viscosity dispensing



The sequence shows dispensing of liquids with different viscosities, ranging from water (1 mPas) to Glycerol/PEG (approx 50 mPas).

Nanolite

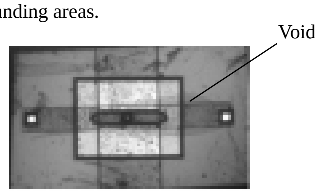
To freeze the motion of a single 100-picoliter droplet, propagating with several metres per second, you will need a camera with an exposure time of less than a tenth of a microsecond to acquire acceptable spatial resolution. Such a camera will be very expensive. Another way to accomplish this task would be by using an extremely short flash to enlighten the droplet. An electrical discharge is used to generate a flash with duration of only 10 nanoseconds, sufficiently short to correctly portray the droplet on a CCD-video camera.

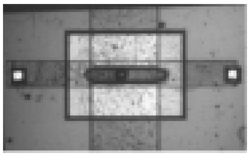


Droplet generation captured by 10 nanoseconds exposure time.

IR-imaging

By directing infrared light through a dispenser structure and using an infrared sensitive CCD-camera for detection, one has a simple but functional set-up for evaluating the quality of the assembling operation. Air cavities (voids) are easily discovered as they appear slightly darker than surrounding areas.





Future work

Silicon direct bonding of dispenser dice

Dynamic imaging of interference patterns

Finite element analysis of dispenser structure

Optimization of pulse shape

Optimization of mounting of piezo-electric element

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