# Design of a normally closed piezoelectric micro valve

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## ABSTRACT

Based on a previously developed piezoelectric membrane actuator a normally closed piezoelectric micro valve is designed. The principle of this novel actuator is explained in this paper as well as the possible design of a normally closed piezoelectric micro valve. The actuator operates with surface electrodes. The thus generated electric field causes an inhomogeneous mechanical stress distribution within the piezoelectric material. Because of this the piezoelectric material is forced to deflect without any supporting passive membrane. If this actuator is placed smartly in a micro valve device a normally closed piezoelectric micro valve can be created, as will be shown in this paper.

***Keywords***: piezoelectric, membrane actuator, normally closed micro valve, piezoelectric micro valve

## 1 INTRODUCTION

To control the flow of media in micro fluidic applications micro pumps and valves are necessary. The integration of those components is necessary if the whole fluidic system is to stay on micro scale. There are several publications on active piezoelectric membrane pumps and valves. Actively controlled piezoelectric micro valve principles usually show a normally open behavior. A possible design of a micro valve may consist of a flexible membrane which opens or closes a valve seat (Fig. 1).

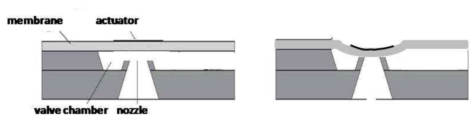


Figure 1: normally open micro valve, left: not actuated, right: actuated

Piezoelectric actuation is well suited for micro systems because of its high energy density and a comparable high membrane deviation. Piezoelectric micro fluidic devices are mainly based on one of two principles. Those are first a passive membrane with a piezoelectric layer for actuation, second a passive membrane actuated by a piezo-stack element. Both principles cause a downward movement of the membrane. Both principles work with piezoelectric layers, which are electrically connected on either side of the layer. The first uses the transversal piezoelectric effect, i.e. the piezoelectric layer will contract laterally if actuated causing the passive membrane underneath to deflect downwards [1,2]. The second principle uses the longitudinal effect, i.e. a perpendicular expansion on top of the membrane. Usually the stack is placed between the membrane and a stiff support, so that the expansion also causes the membrane to deflect downwards [1,2]. Thus considering the general valve design shown in fig. 1 a normally closed piezoelectric micro valve is not easily to design.

In a previously published paper a novel piezoelectric micro actuator was developed and investigated [1,3]. The principle of this actuator is based on actuation by surface electrodes on only one side of the piezoelectric material. The piezoelectric material used for those actuators is lead-zirconate-titanate (PZT). So far a macro-microscopic approach has shown that different electrode designs, cause different deflections [1,3].

The main advantage of this actuation principle is the new flexibility it provides, regarding novel micro pump or micro valve designs [1]. Now it is possible to design a "relatively simple" normally closed piezoelectric micro valve. Another advantage of this novel actuation principle is the possible omitting of the passive membrane. Since the inhomogeneous distributed electric field established by the surface electrodes always causes a deflection of the piezoelectric material. The deflection is directly dependent on the distribution of the electric field.

Based on those results the principle of a normally closed piezoelectric micro valve will be introduced in this paper.

## 2 DESIGN OF THE MICRO VALVE

### 2.1 Working principle of the valve

The valve is designed to open and close to a media under pressure. The valve does not have to qualify for pumping function, it only interrupts a media stream. When actuated the membrane opens the nozzle and the media is able to flow through (Fig. 2).

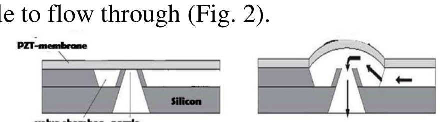


Figure 2: Principle of a normally closed microvalve
left: not actuated; right: actuated

The amount of media flowing through depends mostly on two factors. First is the deviation of the membrane, which is directly proportional to the actuation voltage. Second is the pressure of the media.

## 2.2 Basic Design

The micro valve is a hybrid construction of a silicon substrate containing the micro fluidic structures and a PZT substrate with the actuators. Figure 3 shows the basic design of the micro valve. The silicon wafer also supports the PZT substrate mechanically. For the electrical connection of the actuators, there are non-plated holes through the silicon wafer. The connection itself is realized with contact pins. The fluidic connection to the main channel is realized via a laser ablated hole through the PZT substrate. The bonding of the silicon and PZT substrate is realized by adhesive bonding with epoxy resin [4].

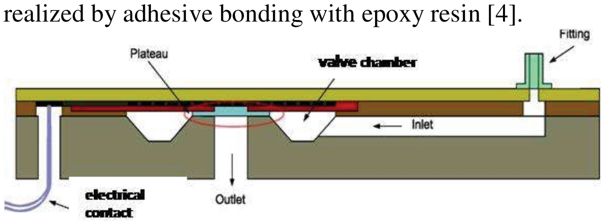


Figure 3: Basic Design of the micro valve

## 2.3 Actuator

The actuator consists of a PZT disc with a diameter of 56mm and a thickness of 250µm, piezoelectric coefficients $d_{33}$=400exp(-12)m/V and $d_{31}$=-180exp(-12)m/V. For actuation surface electrodes are used. This actuation principle is different compared to the commonly known piezoelectric actuators, since the electric flux distribution is completely different (Fig. 4) [1].

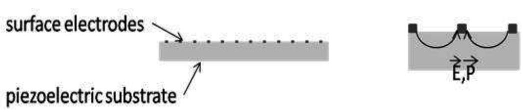


Figure 4: Surface electrodes and electric field distribution

The induced mechanic stress is directly proportional to the electric field, thus the stress distribution will also follow an inhomogeneous pattern (Fig. 5) [1]. Field peeks and therefore mechanical stress peeks are at the electrode edges, whereas into the bulk material and toward the center in between the electrodes the field and stress distribution decreases. Underneath the electrodes field strength is negligible. Hence, a passive membrane is not necessary to generate a deflection, since the piezoelectric bulk material is forced to move in any case. The electrode layout chosen for the micro valve actuation is the double concentric ring layout (Fig. 6). In previous works this layout proved to reliably deflect [3].

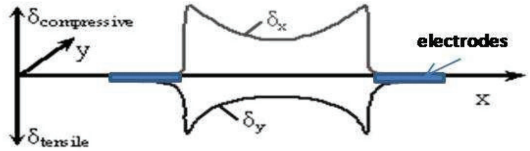


Figure 5: Stress distribution between one electrode pair

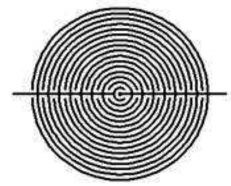

Figure 6: double concentric ring electrode layout

# 3 EXPERIMENTAL SET UP

## 3.1. Process technology

The fluidic structures, channels and valve seats are fabricated in an anisotropic silicon wet etch process. The masking layer is a 2µm thick $SiO_2$ layer, which was previously structured in BHF (buffered hydrofluoric acid). For the wet etch process KOH (caustic potash) solution is used. The desired channel depth is 100µm. Channel width varies from 70µm to 500µm. The well established wet etch process will be optimized as to the concentration of the caustic potash in relation to etching depth and etched surface.
The 65µm wide nozzles are fabricated by DRIE process. The same process is used for the connection holes with a diameter of 2mm. Therefore a suitable mask layout is designed to generate them in one DRIE process, in spite of the extremely differing diameters. This is realized as the 2mm holes are not designed as circles on the mask layouts, but as rings with a line width of 70µm. After the DRIE process the center of those rings is removed by removing the residual $SiO_2$ layer.
Thermal evaporation and a lift off process are used to deposit copper electrodes on the PZT surface. This process also is optimized. The optimal photolithographic process is developed. The pretreatment of the substrate with solution of BHF and HCL for surface smoothing is investigated. The metal layers are characterized visually as well as concerning their conductivity and the functionality of the membrane actuators. Adhesive bonding is used to assemble the PZT-substrate with the silicon substrate (Fig. 7), which is the most critical process step. Since another stiff layer beneath the PZT-substrate is not wanted a spin on process for the epoxy resin is not possible. Therefore micro contact printing methods are investigated. These micro contact printing methods are characterized as to which epoxy resins are best suited for the printing. Further important are the adhesive characteristics of the material.

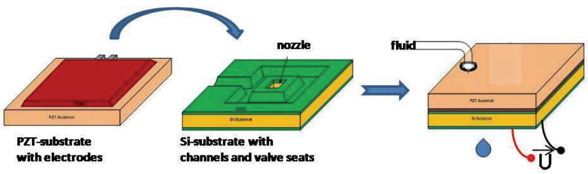


Figure 7: Adhesive bonding of the two substrates

### 3.3. Characterization of the micro valves

36 different dimensioned valves are designed. Fig. 8 shows the basic layout of the valves. The top 36 valves mirror the bottom 36 valves for redundancy reasons. The valve seats (white) and the valve chambers (green/dark) are both varied in size. The outer dimensions of the valve chambers vary from 0.75mm to 2.5mm.The outer dimensions of the valve seats vary between 470µm, 670µm and 870µm.

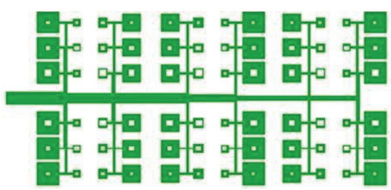

Figure 8: Channel layout for test valves [5]

Additionally to the valves the electrode dimensions of the actuators are also varied, concerning electrode width, either 10µm or 5µm and electrode distance, 10µm, 15µm or 30µm.

The test valves shall be characterized as to the best valve seat to valve chamber ratio. Relevant factors are tightness to media pressure regarding different sizes of the valve seats, the deviation and thus the area available for media throughput. The deviation is a direct function of the applied actuation voltage. A threshold voltage has to be characterized as to what voltage is needed to generate sufficient mechanical stress in the membrane to cause the deflection. Those factors combined with the opening time are directly responsible for the volume of media passing the valve.

The actuators are connected via spring contact from the bottom side. All 72 valves can be actuated individually with a voltage up to 200V. The fluid contact is only pressed on, so that an easy exchange of the sample wafer is given.

## 4 RESULTS

So far the process technologies, processing the fluidic structures and metallization of the PZT substrates have been thoroughly investigated and optimized.

The optimal process for wet etching the channels and valve chambers is to submerge the masked silicon substrates in a 40% KOH for 110 min at 80°C (Fig. 9), which causes very smooth channel surfaces. Channel depth is exactly 100µm and the masking oxide layer loses 700nm. So the masking layer of oxide, which originally measured 2µm can be reduced to less than 1µm, which is extremely important for reducing production time and costs.

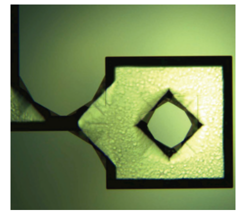

Figure 9: Valve chamber and seat etched in KOH

The left two pictures in figure 10 show the nozzle fabricated with the DRIE process, 10 times magnified. The left picture shows the nozzle, regarded from the valve side, which is the backside in the DRIE process. There is some silicon left standing, which still has to be optimized in the process, but is not impeding the functionality of the valve and nozzle. The middle picture shows the nozzle outlet on the process front side. Here a widening of the diameter by 10µm occurred. The right picture shows part of the 2mm contact hole, which has been exactly etched.

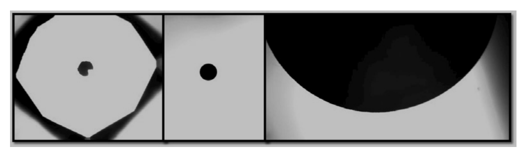

Figure 10: Left to right: nozzle valve seat, nozzle backside, part of a contact hole

For the lift off process used for the metallization of the PZT substrates first the photolithographic process was optimized. This is necessary as the PZT surface is quite rough (roughness of up to 4µm). To get a smoother surface the PZT substrates have been pretreated for various amounts of time (10min, 20min, 30min, 40min) with a BHF – HCl – solution. Visual inspection shows the pretreated substrates show the smoothest surface when treated for 20min. Treatment for a longer time causes irregularities in the surface. All metalized PZT substrates (pretreated and not) provided conducting metal paths. Not pretreated substrates show more defects if regarded underneath the microscope but not so that the conductivity is interrupted.

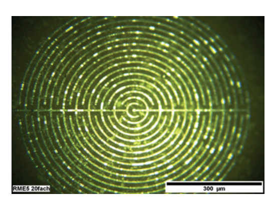

Figure 11: actuator structure on untreated PZT

The functionality of the membrane actuators is far


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better of the untreated samples (Fig. 12). As the bottom diagram in fig. 12 shows no reliable function of the actuators on pretreated PZT-substrates is achieved. The diagrams show 12 arbitrary chosen actuators.

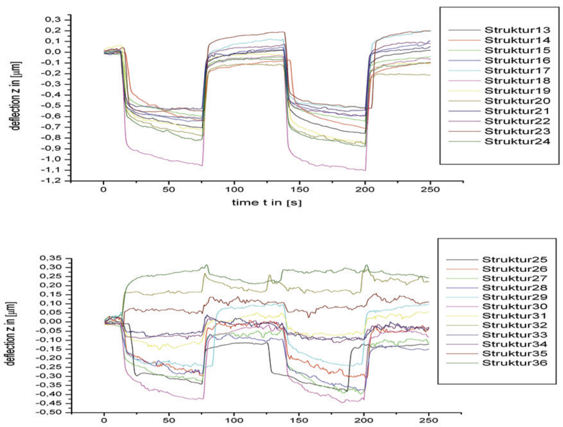


Figure 12: Top: functionality of actuators on untreated PZT, bottom, functionality of actuators on treated PZT

For the structuring of the metal paths on the PZT substrate the pretreatment is improving the process, but heavily impedes the functionality of the actuators. So it will be omitted. For the lift off process a positive resist was spun on with 500rpm and further formatted with 2500 rpm. Metallization layer is 300nm thick. This process provides for conducting paths down to a path width of 5µm and a distance between electrodes of 10µm.

For adhesive bonding a two component epoxy resin with low viscosity and a long can-time was used. The long can-time is necessary since the resin has to be stamped on the silicon substrate. As stamp a PDMS-mold is used, which has been fabricated by replica molding technique [6]. The resin is spun on a dummy wafer then the PDMS-stamp is pressed into the resin. Afterwards the PDMS-stamp is aligned to the HMDS-treated silicon wafer by a mask aligner and pressed to the silicon substrate using the bonding feature of the mask aligner. After the stamp is removed the PZT-substrate is aligned to the silicon substrate also using the mask aligner and then it is pressed to the silicon substrate again using the bonding feature of the mask aligner. Several problems occurred during the process. First a removal of the PDMS-stamp without causing a misalignment is practically impossible. So a light filling of the channels and valve chambers occurs, while removing the stamps. Visual inspection by light microscope showed that the epoxy resin in the fluidic structures is not impeding their functionality. Secondly, the can-time of 150 min does not apply to the thin films used for the micro contact printing process. So that the time needed for the alignment of the stamp, its removal and then the actual bonding of the two substrates must not exceed 10min.

In first experiments a dummy PZT-substrate without actuators was bonded to the silicon-substrate to determine if all nozzles and channels are open and have not been clogged with epoxy resin. This was first tested with methanol and afterwards with water.

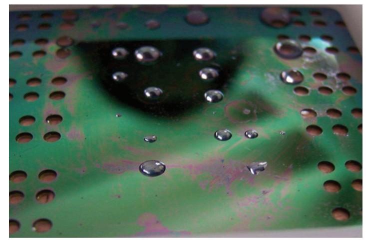

Figure 13: First results with the bonded valve

Methanol went through all nozzles, which proves that the nozzles have not been clogged with epoxy resin during the manufacturing process. Water also passed all nozzles. As fig. 15 shows on the right hand side, water leaks also out of the contact holes. This implies that the adhesive bonding is not tight in all areas and the process has to be optimized further. After this optimization of the bonding process first tests on the functionality of the micro valves will be executed.

## 5 CONCLUSIONS

A normally closed piezoelectric micro valve has been designed based on a previously developed novel piezoelectric actuator. Manufacturing processes have been optimized for the silicon and the PZT substrates. First micro valves samples have been assembled and the nozzles and channels have been proven to be passable for methanol and water so far. The adhesive bonding process still has to be optimized, since a leakage toward the contact holes occurs. The newly developed micro valve offers a simple design, thanks to the application of the novel membrane actuator. This offers totally new possibilities, since there are a lot of advantages to normally closed valves. One great advantage is that no energy is needed for the closed valve, especially with regard to security sensitive matters, if the media is toxic or highly inflammable.

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