

A MINIATURIZED CHEMICAL ANALYSIS SYSTEM USING TWO SILICON MICRO PUMPS

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Abstract

A hybrid miniature chemical analysis system is realized using two piezo electrically driven silicon micro pumps and a separate glass flow through cell with a potassium sensitive ISFET. The measurement protocol is such that the sample solution does enter the detector but does not pass the sensitive pump valves, thus improving the practical applicability of the system. During its operation the sensor is continuously calibrated with a very low consumption of calibrating solution. With a measurement rate of 4 samples per minute the use of calibrant is only 2 ml / hour.

Introduction

In the development of analytical chemical instrumentation, a clear trend exists toward miniaturization. Such miniaturized systems offer an improved efficiency with respect to sample size, response time and reagent consumption. Recent developments in chemical sensors have, for instance, shown multi-ion detectors for Flow Injection Analysis (FIA) systems [1]. Next to the miniaturized detectors, however, the FIA application still makes use of conventional (peristaltic) pumps and bulky injection valves and thus an overall reduction in size of the system is not yet attained. A further step in the development of micro instrumentation is to apply, next to the silicon based sensors, micromachined structures in silicon for the liquid handling. In that way, the internal volume of the analysis system can again be drastically reduced, thus more fully exploiting the above mentioned advantages of miniaturization.

Silicon micro pumps, actuated by a piezo-electric disc, have been shown to yield flow rates up to several tens of microliters per minute [2]. Detector cells in which ISFETs are incorporated by means of anodic glass bonding can be realized with a cell volume of less than one microliter per sensor [1]. Thus, if these micro pumps are used to introduce samples in the order of a few microliters into the detector cells, response times in the order of some seconds are attainable.

A practical limitation in the application of micro-machined pumps for fluid handling is the small displacement of the valves. Therefore, proper pump operation is very susceptible to foreign particles in the applied solutions. Thus, sample solutions have to be carefully filtered or the construction of the system has to be such that these solutions do not pass the pump valves. We propose a system comprising two pumps in connection with a sensor cell. That way, the first pump ('wash pump') can be used to flush the detector cell with filtered calibrant solution while the second one ('sample pump') is intermittently used to draw a small sample from the outside world just big enough to fill the system up to the detector but not to reach the sensitive pump valves. A schematic drawing of the measurement set-up is given in Fig. 1.

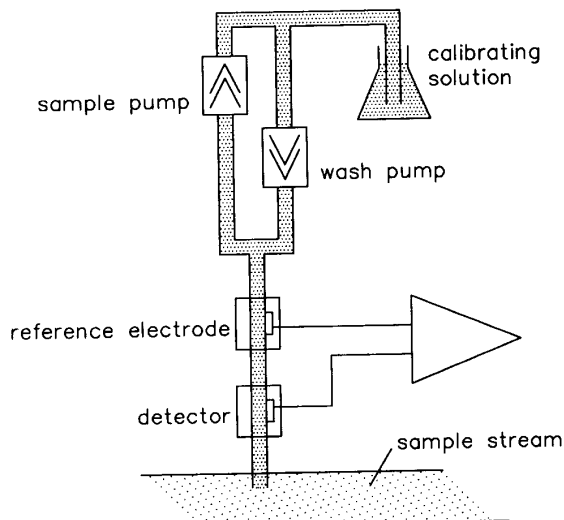


Fig. 1 General set-up for integrated analysis system

To obtain a minimal dead volume and thus an optimal response time from the miniaturized analysis system, integration of pumps, valves and detectors on the same silicon chip seems attractive. However, considering the relatively large surface area required for a pump, this would result in only a small amount of sensors per wafer. Thus, for somewhat more complex sensor fabrication processes, a hybrid version of the analysis system with separate pumps and detectors may be a better approach.

Currently a hybrid version of the micro analysis system is realized where a separate detector cell and two micro pumps are connected using stainless steel capillaries and silicone rubber tubing. The detector cell, used before as the base element for a multi-ion sensor [1], contains four ISFETs. It uses a conventional saturated calomel reference electrode, connected via a capillary between the cell and the micro pumps. The separate elements of the miniature analysis system will be described below.

Micropump

The design of the silicon micro pump is similar to that of Van Lintel et al. [2]. It consists of a micro-machined silicon part anodically bonded between two pyrex glass plates. The thicker of the glass plates (1.5 mm) serves as the base plate for the fluid connections which are made by 0.9 mm OD stainless steel tubes. The thinner glass plate (0.3 mm) forms

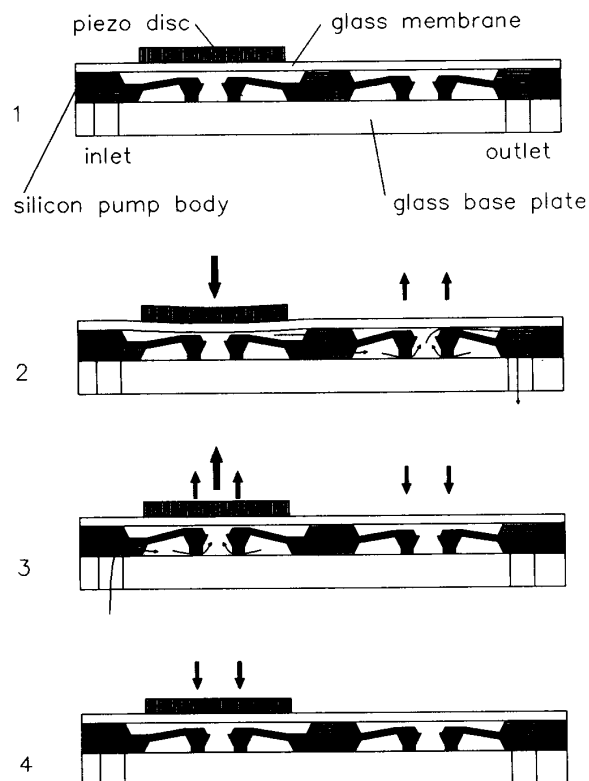


Fig. 2 Cross section of the piezo electrically driven micro pump. The operation of the pump is explained in the text.

the pump membrane that is driven by a piezoelectric disc (Philips PXE 5, 10 mm diameter, 0.2 mm thickness). The actuator disk is glued onto the glass membrane using conductive epoxy (EPO-TEK H20E). The silicon chip forms two passive valves on the glass base plate, a 1 μ m layer of silicon dioxide left on the valve rims prevents the bonding of the valves to the glass when the pump is assembled.

Figure 2 shows a cross section of the pump and explains its operation. In the first drawing (2.1) both the inlet and outlet valve are closed and no liquid is flowing. Then (2.2) a voltage is applied to the piezo disc and the glass membrane bends down. As a result the pressure in the pump chamber mounts and liquid is forced through the outlet valve which is now open. The voltage on the piezo disc is released (2.3) and the pressure in the pump chamber drops, closing the outlet valve and opening the inlet valve thus filling the pump chamber. Finally (2.4) the pressure difference across the inlet valve equals out and the original position is regained.

The actuator disc is driven with a square wave voltage with an amplitude up to 180 V. The maximum pump rate is obtained at frequencies around 30 Hz and is in the order of 200 μ l per minute. The maximum output pressure (at which the pump rate drops to zero) is approximately 200 cm H₂O.

Detector Cell

The ISFETs used as the detector in our hybrid miniaturized analysis system are incorporated in a glass flow through cell by means of anodic bonding [1]. The original cell volume of 12 μ l is reduced to 2 μ l by the use of a flat cover plate to close the channel. Fluid connections are made via stainless steel tubes of 0.5 mm OD, glued to the cover with epoxy.

The cell contains four ISFETs, all of which can be modified with different ion sensitive membranes to create a multi-ion sensor. For the results presented here only one ISFET is used, provided with a standard potassium sensitive valinomycin-PVC membrane.

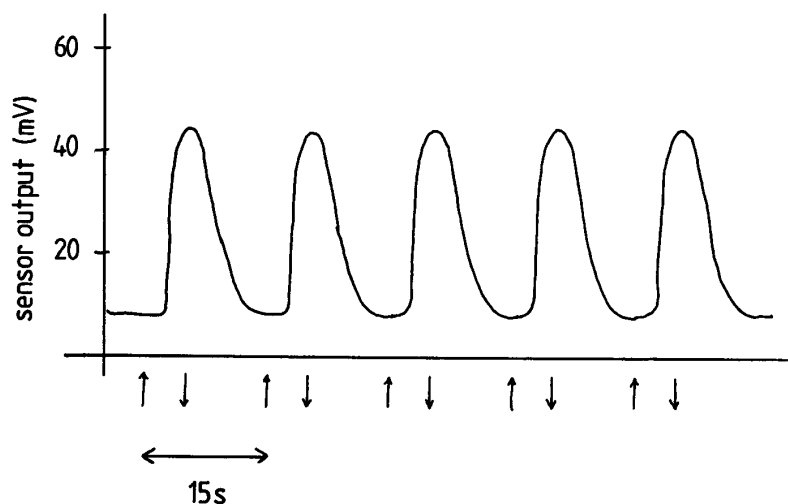


Fig. 3 Response of the micro hybrid analysis system. The system switches continuously between sampling (up going arrow) and rinsing (down going arrow). The cycle time is 15 seconds.

Results

As discussed in the introduction, the system comprises two pumps which are operated in a way that the sample solution does enter the detector cell but does not pass the pump valves. The measurement protocol is such that a sample pump is operated for 5 seconds so that a sample is drawn and subsequently the wash pump is operated for 10 seconds so that the detector cell is flushed with calibrating solution and the baseline level is regained. Figure 3 shows a measurement result where a 10^{-2}M K^+ baseline solution is used and $5 \times 10^{-2}\text{M}$ samples are taken. The dead volume of the system before the detector is approximately $5 \mu\text{l}$. The flow rate of both pumps is set to $100 \mu\text{l} / \text{minute}$, with the applied measurement protocol the consumption of calibrating solution is one third of this amount or only $2 \text{ ml} / \text{hour}$.

Discussion

It is shown that a micro hybrid analysis system is practically applicable with a very modest consumption of calibrating solution. If the dead volume before the detector can be made smaller, the response time and reagent consumption can again be considerably reduced. In our present system this dead volume is about $5 \mu\text{l}$ resulting in a lag time of several seconds between the switching on of the sample pump and the response of the sensor. At this moment the dead volume is mainly due to the internal volume of the tubing between sample stream and detector. With an alternative design for the flow-through cell this inlet tube can possibly be omitted completely and the dead volume is drastically reduced. Then also the flow rate of the pumps can be diminished resulting in a still lower consumption of calibrating solution.

Acknowledgement

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References

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