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# Paraffin Actuators in Microfluidic Systems

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#### **Abstract**

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There is a need for reliable valves and pumps in microfluidics. A good microactuator is the key for low cost and high performance of these components.

Paraffin wax is a promising material to be used as actuator material as it can produce large forces and large strokes. Further, the material is inexpensive and, none the less, the thermal heating of the material can be made with low voltages. All these properties are of interest in flow control components in microfluidics, and especially for disposables and in potable systems.

In this work, paraffin wax has been used in devices and concepts. A valve for high-pressures, a peristaltic pump, a multi-stable actuator, and injector has been shown. A material study was performed on binary mixtures of pure paraffin (n-alkanes), and a concept for loading fluid into a sealed reservoir was shown as well. Several injectors were demonstrated in a Lab-on-a-chip system with other microfluidic components.

High pressure applications in microfluidics along with the multi-stable actuator show good potential. However, the drive and control has to be further developed.

*Keywords:* Paraffin, Actuators, Valve, Micropump

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*To Maria, My and Kajsa*



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*Journal of Microelectromechanical Systems*, vol. 16, no. 3, 2007, 728-733
- II     Rapid prototyping of a polymeric paraffin microactuator  
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*Submitted to Journal of Microelectromechanical Systems*
- III    A multi-stable paraffin microactuator with positioning capabilities  
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- IV    A polymeric paraffin actuated high-pressure micropump  
R. Bodén, M. Lehto, U. Simu, G. Thornell, K. Hjort, and J.-Å. Schweitz  
*Sensors and Actuators A* 127 2006, 88-93
- V     Valve with septum for loading of fluid to on-chip reservoir  
M. Lehto, R. Bodén, K. Hjort, J.-Å. Schweitz, and G. Thornell  
*Submitted to Journal of Micromechanics and Microengineering*
- VI    Towards a self contained lab-on-a-chip concept with sequential drive for point-of-care testing  
R. Bodén, M. Lehto, J. Margell, K. Hjort, and J.-Å. Schweitz  
*Submitted to Lab on a Chip*

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# The Author's Contribution to the Papers

- I Major part of everything
- II Part of everything
- III Major part of planning, part of everything else
- IV Part of everything
- V Major part of everything
- VI Part of experimental, evaluation, and writing

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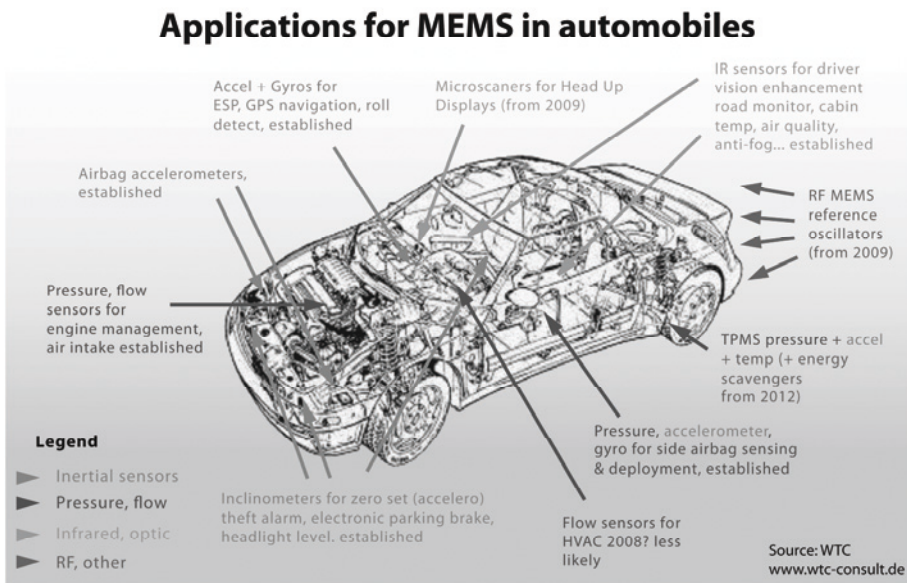
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# Introduction

Microelectromechanical systems (MEMS) or Microsystem technology (MST) are a part of everyday life and can be found in e.g. digital micromirror devices (DMD) for HDTVs or projectors [1], ink-jet heads in printers [2], pressure sensors for intravascular use [3], and in various sensors in automobiles (Fig. 1) [4]. Common for MEMS are their characteristic physical size from typically some micrometers to few millimeters, and their micromachining techniques. Reduction of cost is one of the major driving forces for technological development. And, by choosing components that are made by micromachining, processing in batches can lower the production costs. In some cases the miniaturization has resulted in better performances such as faster response times and better sensitivity for accelerometers, or by making it possible to monitor pressure inside blood-vessels.



*Figure 1.* The growing use for MEMS applications in automobiles (with permission from WTC [4]).

MEMS have their origin in the IC industry where silicon is used to fabricate electronic components. It was soon found that silicon has excellent me-

mechanical material properties as well. As mechanical structures had to be developed, the advancements in the field of MEMS was concentrated on process development. This was carried out mainly by researchers, where parts and components are built using a bottom-up approach in which primarily in-house processes were used to investigate their abilities to create devices.

With the interest of miniaturization from other disciplines such as biotechnology, there is an increased need for other materials and simpler processes. A tickling thought is to create a self contained microfluidic chip that can sample and detect diseases, pollution, or drugs by using samples such as blood, urine, saliva, or water. The believed solution is Lab-on-a-chip (LOC) or micro-total-analysis systems ( $\mu$ TAS). This is for the moment a challenge for researchers and companies all over the world. Imagine the possibilities to reduce the cost and availability for e.g. HIV tests in Third World countries, instant detection of water pollution, or monitoring of your own health without the need of a central laboratory or visit to the doctor's office. But to succeed with these systems, co-operation over different disciplines is a necessity. System interfacing, assembly, and optimizing will become more important than maximizing the performance of each part. Still, new concepts for MEMS components and parts have to be found in order to provide the designer with more choices when designing a  $\mu$ TAS. For instance, on-chip drive and control of flows are still somewhat awkward, e.g. it is still a challenge to provide valves that are leak tight and pumps that are easily integrated and controlled.

As actuators are the basic part in many valves and pumps, the chosen actuation technique will highly influence the performance and fabrication techniques of the component. Some of the problems with microactuators are the small strokes and forces. Paraffin waxes are interesting to use as a phase-change material (PCM) in microactuators, as the volume expansion is strong as well as can be large. The melting of the material can be done quite easily with thermal heaters at low voltages.

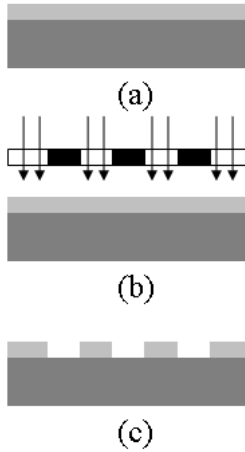
This summary will describe the exploration of paraffin wax in different microfluidic components and systems. The second chapter will briefly describe different fabrication techniques used in MEMS and the philosophy of rapid prototyping. The third chapter describes some material properties of paraffin wax and how they can be changed. The material studies made in Paper I and Paper II are summarized. Chapter four gives a survey on earlier published microactuators using paraffin wax, and a summary of the actuator presented in Paper II and III. The work in Paper V and VI are summarized in chapter five that deals with some aspects of microfluidics. In paper IV a micro pump with active valves is presented. In Paper V a concept for fluid handling on chip is presented and in paper VI this concept are further developed to handle several fluids in a LOC platform.

# MEMS fabrication and rapid prototyping

Fabrication is central in MEMS. At macro scale several fabrication techniques that can easily produce 3-D structures are available, and the structures are built one by one. In MEMS on the other hand, the structures are usually built layer by layer, including repeated patterning, deposition, and etching of material. Here, several devices can be processed simultaneously side by side on the same substrate in batches. As a result, a MEMS device can be produced at a low cost. The fabrication techniques available for MEMS depend on which material that has to be processed. To understand the diversity a brief description of different fabrication techniques and methods are presented. A more comprehensive description can be found in textbooks on the subject [2, 5, 6].

## Lithography

Patterning is usually made with lithography. Here a substrate is spin coated with a special polymer sensitive to light (photoresist). Then by selective exposure with UV light through a mask, a desired pattern is transferred to the photoresist that becomes more or less sensitive and can be developed (Fig. 2). The patterned photoresist can now serve as protection of the surface below from being deposited or etched. The photoresist is removed with a solvent before the next layer of material can be deposited. The lateral resolution is normally sub-micrometer in thin resist with a thickness of about 1  $\mu\text{m}$ . Layer thicknesses of up to 2 mm can be done using special polymers and multiple coatings [2].



*Figure 2. (a) Substrate with photoresist, (b) UV exposure through mask, and (c) developed pattern.*

## Silicon processing

The most common material in MEMS is silicon due to, in many cases, its superior material properties. High-temperature and vacuum equipment located in clean rooms are frequently used to process silicon. As these processes are time consuming, high volume production is appropriate. Less expensive processes can also be used but with lower resolution as a result. However, with the development in the IC industry there exists many ways to create complex electro mechanical structures. Silicon processing can be divided into two fields; surface and bulk machining.

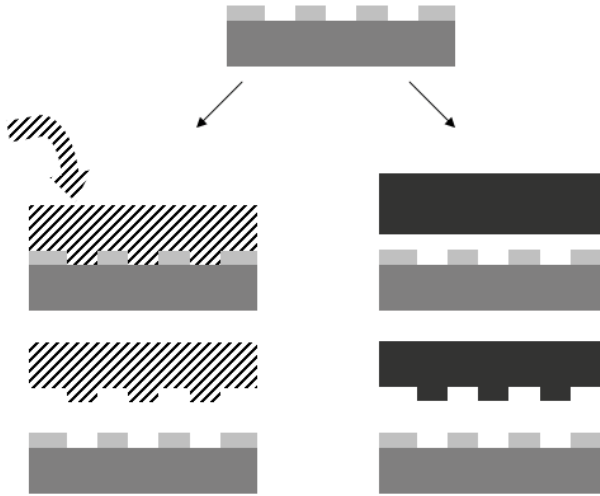
In surface machining thin layers of for instance silicon oxides, silicon nitrides, metals, and polycrystalline silicon are deposited, masked, and etched. Popular substrate materials are silicon or glass. The thickness of these layers ranges from about a hundred nanometers to a few micrometers.

Bulk micromachining is similar to surface machining, but with the difference that e.g. grooves or trenches, usually tens of microns deep, can be formed in the substrate material.

## Polymer replication processes

Non-photolithographic techniques such as embossing, injection molding, cast molding, nanoimprinting, and microreplication have become more interesting with the introduction of polymers into MEMS [7]. Here, patterns

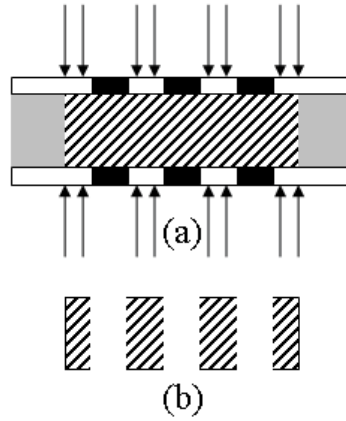
are transferred to polymers from a master by physically deforming the polymer or by casting (Fig. 3). These techniques do not require the same equipment used in silicon processing. However, a master can be fabricated by means of surface or bulk micromachining. The expensive and time consuming part is the fabrication of the master, but once having this, the production cost can be low as one single master can be used repeatedly.



*Figure 3.* A master (above) is for instance used for casting of a liquid polymer (left), or for physically deforming a substrate by embossing (right).

## Liquid polymerization

A way to create millimeter thick structures is to use e.g. liquid resin of UV curable epoxy. Instead of spin coating photoresist as in ordinary lithography, a one-component epoxy that is poured into a mould can be used. Solidification, patterning, and curing takes place in one single step (Fig. 4) and the structures are complete with a short post-cure after the uncured epoxy is rinsed. This method does not require as long curing steps compared to thick photoresist.



*Figure 4.* Solidification, patterning, and curing with UV-light from two sides (a), removed, rinsed, and post-cured structure (b).

Liquid photopolymerization has been used as a rapid prototyping technique in Paper II and IV, using overhead transparencies with resolution of 1200 dpi as masks and a 400 W metal halide lamp as the UV source.

## Milling and drilling

As polymers are becoming more frequently used in MEMS, milling and drilling are interesting. For example, a commercial prototype milling machine for printed circuit boards (PCB) [8] is sufficient in many cases. The resolution is a few micrometers and commercial tool diameters of down to 100  $\mu\text{m}$  are available. Milling is useful for fabrication of few components, drilling of holes for fluidic connection, or to create masters that can be used with polymer replication techniques. Milling is also useful for rapid prototyping.

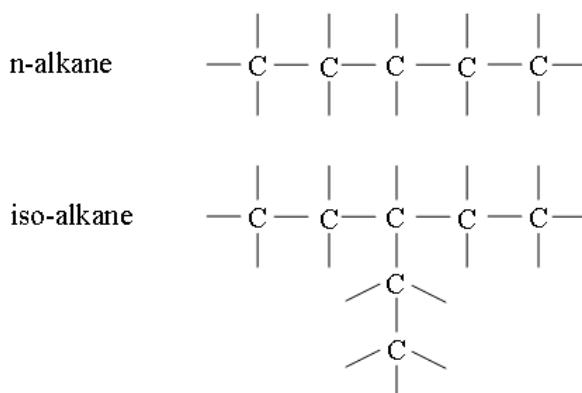
## Rapid prototyping and rapid fabrication

Prototyping includes development techniques where preliminary versions (prototypes) of a system, or part of a system, are built to investigate its feasibility, and reducing the risk. Rapid prototyping can be used to test unknown characteristics of components or systems by fabrication techniques where the time from idea to realized device is short. Milling and soft lithography can be considered as rapid fabrication techniques.

# Paraffin wax

## What is paraffin wax?

The word paraffin (para-, -finum) means lacking affinity or lacking reactivity, and paraffin does not easily mix with polar fluids as water. Paraffin wax is a mixture of saturated alkanes, hydrocarbons, with the general formula  $C_nH_{2n+2}$ . Alkanes can either be straight or branched, i.e. normal or isomeric (Fig. 5). The simplest alkane is the gas methane,  $CH_4$ , and the first isomeric alkane is isobutane  $(CH_3)_3CH$ . The isomeric possibilities increase with the number of carbon atoms and there exists about 4 billion combinations of  $C_{30}H_{72}$  [9].



*Figure 5.* An illustration of linear normal (n-) and branched isomeric (iso-) alkanes, where each free end-site is represented by a hydrogen atom.

Paraffin is commonly used in products such as skin lotion, protective surfaces for food, window openers in green houses, and for thermostats in cars.

Depending on the refinement process, some paraffin waxes usually contain few wt-% of oil. However, refined paraffin waxes that can be used in the food industry, contain only 0.4-0.8 wt-% oil, are colorless, and do not contain any substances that are harmful to health [10].

## Material properties

Paraffin waxes change their volume at phase transitions and are by definition solid at room temperature. The phase change of paraffin is reversible, and the heat that is needed to change phase, e.g. melting, is stored in the material as latent heat and is returned when the material crystallizes. The melt temperature for n-alkanes, and thus paraffin waxes, are connected to the chain length of the n-alkane. However, the highest melt temperature converge to about 140°C for very long chains e.g. polyethylene (Fig. 6). A fluid can, however, be supercooled and will then not start to crystallize at the same temperature as where the melting ends. The critical limit for supercooling for alkanes between  $C_{16}$ - $C_{34}$  is about 95% of their coagulating point, meaning that they will spontaneously start to crystallize 10-15°C below that point [11]. But in practice this temperature is only a few degrees below the coagulation point.

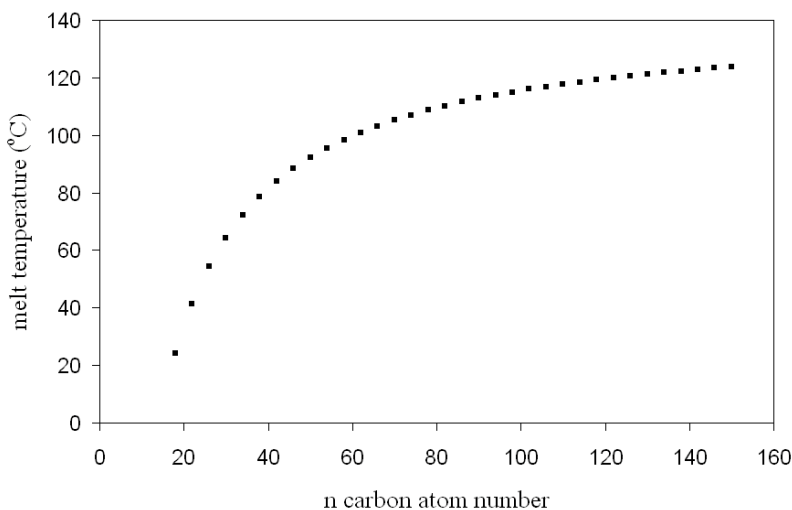
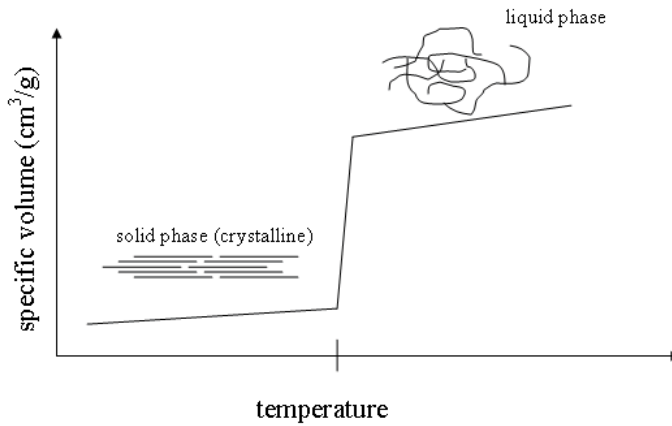


Figure 6. Melting temperature as a function of number of carbon atoms.

When a melt of paraffin starts to coagulate, the molecules will pack close together in ordered form, i.e. they will start to crystallize. Consequently the more ordered solid phase has a higher density and a decrease in volume is seen (Fig. 7). Similar to metals, n-alkanes crystallize in different configurations (lattices). There are four known ways for n-alkanes to crystallize, namely (hexagonal ( $\alpha_H$ ), orthorhombic ( $\beta_O$ ), monoclinic ( $\beta_M$ ), and triclinic ( $\beta_T$ ); each with their own shape and size. Thus, volume changes can also be observed for solid-to-solid transitions as well.





*Figure 7.* The volume changes with phase change such as melting when the material goes from crystalline to amorphous.

It is the thermo mechanical properties that make paraffin interesting in MEMS.  $C_{36}$ , for instance, has a free volume expansion of about 22% when heating it from 30 to 80°C, and its compressibility at liquid phase is less than 1% per 10 MPa [12]. Further, the coefficient of thermal expansion (CTE) is roughly 500 ppm/K for solid  $C_{36}$ , which is few times more compared to many polymers, and more than 10 times greater compared to metals.

If two alkanes are mixed at different ratios, numerous combinations of these configurations can be achieved. Consequently, by choosing suitable alkanes and mix ratio, more or less stepwise expansions could be utilized including solid-to-solid transitions. It is even possible to achieve an eutectic point, where the melting temperature is lower than the melting point for the two components (Fig. 8).

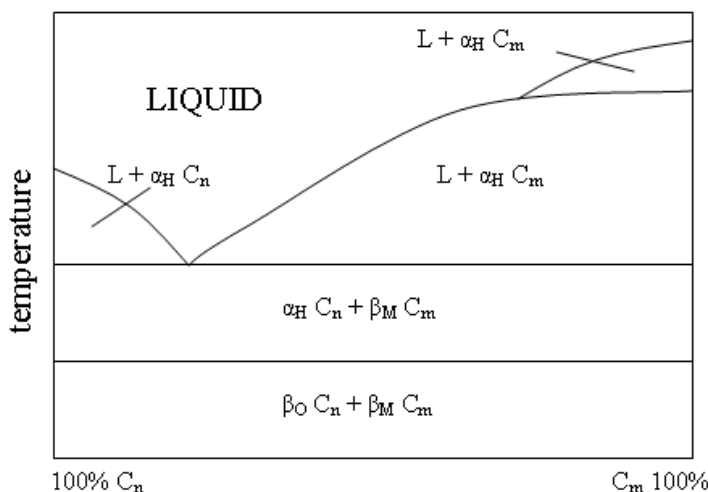


Figure 8. Example of a phase diagram of binary mixtures,  $C_n$  and  $C_m$ .

## Analysis methods

Most studies on paraffin wax and n-alkanes have been made for better understanding of the crystallization behavior of other polymers [11, 13], or for studying their thermal heat-storage properties.

Differential scanning calorimetry (DSC) can be used to attain information such as transition temperatures and enthalpies for a material. However, this method does not provide any thermo mechanical information. In addition, a dilatometer that records the linear expansion as function of temperature and pressure can be used. With this, so called pressure-volume-temperature (pVT), equipment information about for the coefficient of thermal expansion (CTE) and volume expansions at phase changes can be attained.

pVT are somewhat time consuming in the sample preparation step, and the sample sizes are about 100 times larger than for DSC. The DSC can also be loaded with several samples and can be run automatically.

## Binary mixtures of n-alkanes (Paper I)

In Paper I binary mixtures of n-alkanes are investigated by means of pVT and DSC. The mixing behavior is studied and a comparison between the two methods is made.

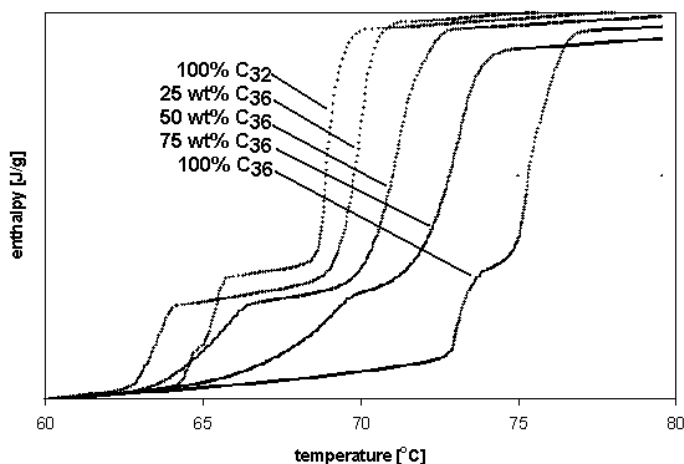


Figure 9. Enthalpy as a function of temperature as obtained with DSC (Paper I).

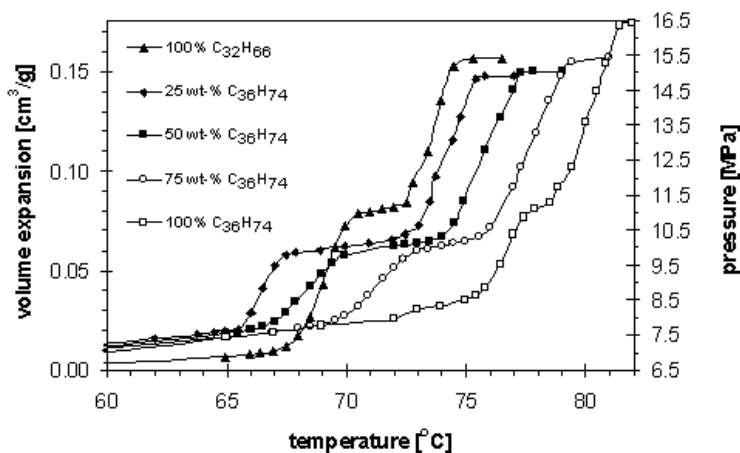


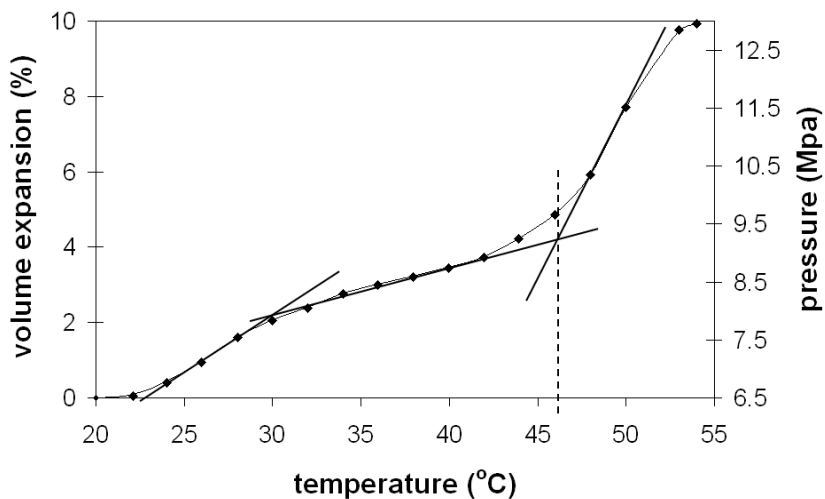
Figure 10. Volume expansion and applied pressure as a function of temperature as obtained with pVT (Paper I).

The result from pVT and DSC can be seen in Figs. 9 and 10. A good prediction of the volume expansion at phase transitions can be made with DSC, but to attain a more exact volume expansion, a pVT measurement is needed. The pressure dependency on the melting temperature is found to be about 0.3 KMPa<sup>-1</sup>.

It was found that the temperature width of the intermediate phase indeed could indeed be manipulated by mixing the alkanes.

## pVT dilatometry on a paraffin wax (Paper II)

In Paper II, the volume expansion of the paraffin wax (Sigma-Aldrich, 76228) is measured with pVT. The wax is also used in Paper III-VI. It is found that a solid-to-solid transition with volume expansion of more than 2% is present between 20-30°C, and an expansion of less than 6% was observed for the solid-to-liquid transition (Fig. 11). The linear volume expansion between 30-46°C is about 2%, and the overall volume expansion from ambient temperature of 20°C to melt is 10%.



*Figure 11.* Volume expansion of paraffin wax (Sigma-Aldrich, 76228) as obtained with pVT. The wax has a melting point at 46°C and solid phase transition between 20-30°C, (Paper II).

# Paraffin wax actuators

An actuator can be described as a transformer that can convert e.g. an electrical signal into mechanical work. There are several existing microactuators in which different techniques as magnetic, piezoelectric, thermal expansion, phase change, and electrostatic actuation are used. A simple way to compare these is by their physical properties such as force, displacement, and speed [14].

But compared to microsensors, microactuators have not yet easily been adopted to commercial products. One reason for this is their disadvantage when scaled down and most actuators can simply not produce sufficient force or displacements at microscale. For example an actuator that uses the popular piezoelectric effect can create large forces but not large displacements. Shape memory alloys (SMA) that use solid phase transitions can produce both large expansions and large forces. However, these are commonly formed to springs and wires and are less suited for microfabrication techniques. On the other hand, there are successful examples too. The electrostatic force is well suited in RF-MEMS and optical MEMS as in the DMD chip [1].

Thermopneumatics and thermohydraulics are attractive methods to use because they can produce large displacements, high forces, and can be miniaturized. These are well suited for e.g. microfluidics. Wax actuators are especially interesting as they can produce large forces along with large volume expansion. Many fabrication techniques, designs, and materials can be used as well.

## A survey on paraffin actuators

In MEMS, no commercial device is yet available with paraffin wax as the actuator material. However, in research the term microactuator used for paraffin actuators is mentioned in work from 1997 [15, 16]. These actuators are several centimeters long and the word micro refers to the possible displacements. One sub-centimeter long piston actuator capable of producing forces of 10 N is presented in [17].

A microactuator built with surface micromachining on silicon and glass substrates [18] uses a 9  $\mu\text{m}$  thick paraffin layer with diameter of 400-800  $\mu\text{m}$  and is covered with parylene as diaphragm. The deflections are about 3  $\mu\text{m}$ ,

and the glass substrate actuator has a power consumption of 100-150 mW with response times of 30-50 ms. The power consumption for the silicon substrates is up to 3 W, but with response times 10 times smaller than for the glass substrate.

A Braille cell with paraffin wax is presented in [19] using three layers of bulk micromachined silicon and a silicone rubber diaphragm. The device is filled with paraffin by placing it on a hot plate and molten paraffin is poured into the wax containers. The diaphragm rubber is applied by brushing it over the device. The approximate diaphragm thickness is 80  $\mu\text{m}$  and the diameter is 1.5 mm. The maximum dot height is 654  $\mu\text{m}$  for 8 V input voltage.

A thermal switch for thermal control meant to be used in a nanosatellite [20] use the excess heat in the satellite to melt paraffin wax to close a switch enabling heat transfer. The device is made with bulk micromachining in silicon and at the last step the paraffin is filled into the cavity using a syringe. The device is able of producing movements of about 75  $\mu\text{m}$  with 3 N load.

Another paraffin based actuator to be used in a nanosatellite propulsion system is presented in [21]. Here the paraffin was filled in the same fashion as in injection molding. The actuators are heated with attached heaters. The filling is seen as a major problem, as it is difficult to control.

Polymeric actuators with hot-embossed structures and thermally bonded or glued membrane are presented in [22, 23]. The general approach here is to enhance the bonding properties by surface treatments, or developing the glue process. Extensive rupture load tests are made to study the joining properties of the membranes to the bulk. Other paraffin actuators can also be found in [24-26].

## Rapid prototyping of a paraffin actuator (Paper II)

In Paper II a membrane paraffin actuator is made by means of rapid prototyping using liquid photopolymerization. The aim is to investigate both the usefulness of the process and the performance of the actuator by changing the input potential and frequency of a square wave over several cycles. The ability to integrate a heater into the paraffin volume is studied as well as with the use of an adhesive tape as a membrane. Deflection measurements are made using a contact probe with a high load of 0.5 N in contact pressure.

The tests indicate a degeneration of the actuator that is run over 1,050 cycles. This degeneration is a result of paraffin leakage under the tape membrane. The actuator had a maximum deflection of 90  $\mu\text{m}$  measured with a white light interferometer. It is found that a steady state behavior is present when the actuator is run at higher frequencies, and an offset of the curve can

be seen (Fig. 12). The reason for this is that the cooling is not efficient enough.

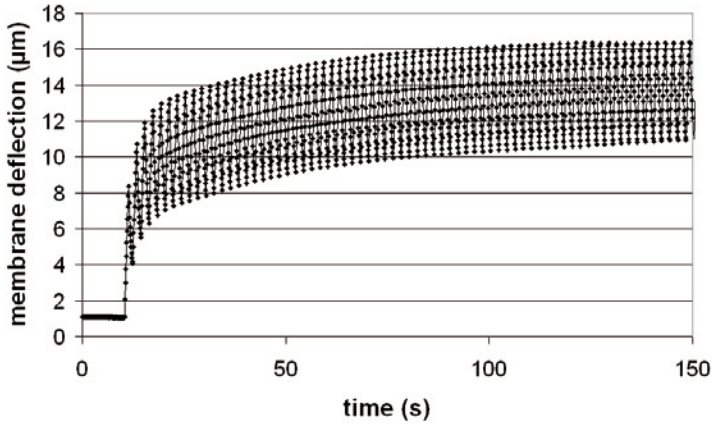


Figure 12. Deflection behavior for an actuator at 0.5 Hz and 0.71 V.

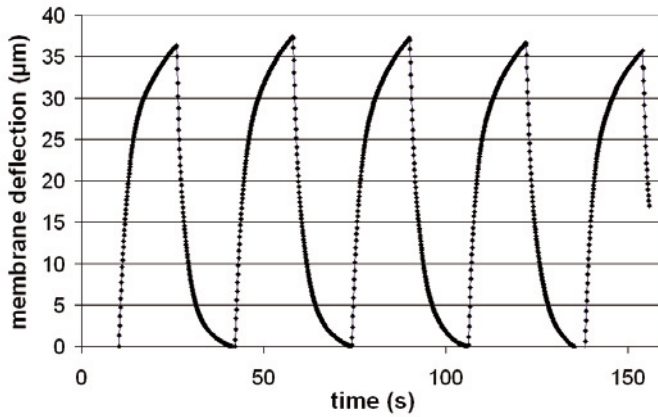
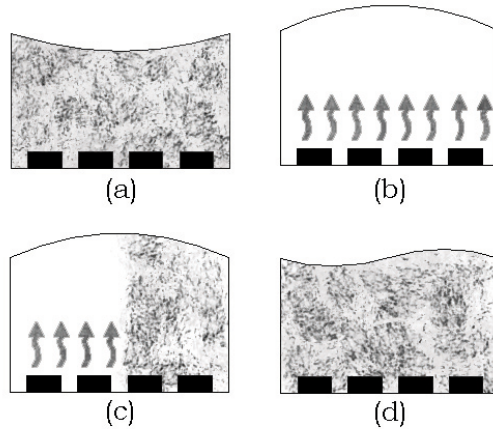


Figure 13. The deflection curve at 1/32 Hz and 0.71 V.

It is also seen that the beginning of the deflection curve (Fig. 13), traveling from zero to full deflection, has a higher slope than near full deflection. Also at higher frequencies the curves are more saw-tooth shaped but have a smaller maximum deflection. It is also found that the deflection velocities ( $\mu\text{m/s}$ ) are higher for higher frequencies.

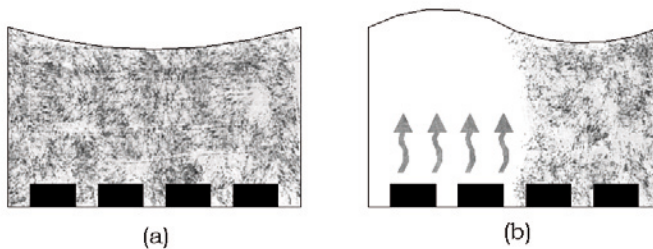
## A multi-stable paraffin actuator with local active deflection (Paper III)

The aim in Paper III is to investigate the possibility to create multi-stable positions and local deformation for a membrane paraffin actuator by using multiple heaters. Milling and gluing are used to build the actuators.



*Figure 14.* Schematic description of multi-stable positioning. (a) solid paraffin, (b) liquid paraffin, (c) the paraffin is allowed to crystallize in part of the cavity by selective heating, and (d) solid paraffin with deformed membrane.

The poor thermal heat transfer between liquid and solid state is utilized in such a way that both solid and liquid phases can co-exist within the same cavity. This is made by the use of several heaters that are individually controlled to create thermal gradients in the paraffin (Figs. 14 and 15).

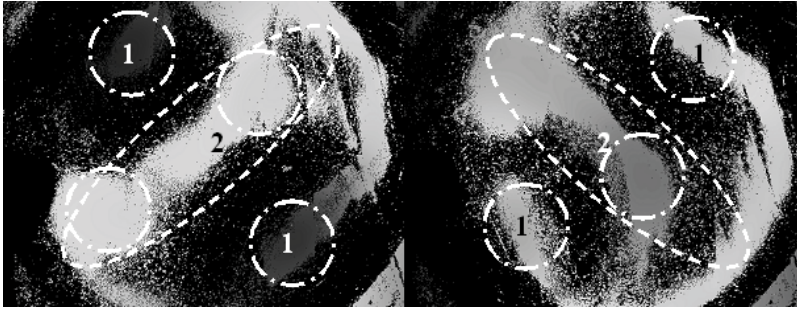


*Figure 15.* An actuator used in active mode where part of the paraffin is melted and creates a local deflection of the membrane (b), (a) is a non-activated actuator.

The actuators, run in their simplest way, are capable of multi-stable positioning with the number of stable positions depending on how many heater



elements that are used. The difference between the lowest and highest points on a membrane is typically 40-100  $\mu\text{m}$ , and the slope between these peaks and pits have angles of up to  $5^\circ$  (Fig. 16). In addition, the paraffin could also be melted locally over a heater as earlier described.



*Figure 16.* Actuator that is run in diagonal mode with an elevated ridge (2) diagonal across the membrane and lower pits (1).



# Microfluidics

The analytical performance of chemical sensing can be improved by miniaturizing fluidic systems as diffusion lengths decrease. Scaling is also beneficial in the sense that less sample volumes and reagents are needed. This has led to an impressive development of the field of microfluidics, also called Lab-on-a-chip, Bio-MEMS, or  $\mu$ TAS, over the last decade [27].

The miniaturization towards  $\mu$ TAS has open new possibilities for portable systems that can e.g. be used for point-of-care tests (POCT) where the patient their self introduces samples as saliva, blood, or urine to the microfluidic chip. In remote settings, maybe diseases as HIV can be detected only by introducing a drop of blood on site without the need of a central laboratory or skilled personnel [28].

The further away from a central laboratory with skilled personnel, the more functions have to be integrated to the chip. For instance, depending on sample, different pre-treatments such as filtration, washing, and rinsing are needed [29]. If a  $\mu$ TAS should be able of carrying this out, several reservoirs containing reagents and fluids are needed on the chip. These fluids have to be pumped one by one, and valves have to be used to control the direction of flow. The fluids have to be loaded and stored on-chip, and cross contamination has to be avoided. Moreover, it is an advantage that the valves and pumps are small, inexpensive, easy to drive, and perhaps disposable if they are located on the chip.

Miniaturised valves and pumps are associated with many difficulties, and commercialization has been delayed by the lack of reliable components [30]. When downsizing a fluidic channel, the pressure needed to drive a flow in it increases due to an increased surface-to-volume ratio. As a consequence, the pumps and valves are not capable of producing sufficient pressures. However, there are many pumps that are able of producing sufficient flow rates of ml/min, but for low flow rates of less then fractions of  $\mu$ l/min many difficulties arise [31, 32].

## Valves

Valves are mainly divided into active and passive ones. Active valves utilize an actuator for operation. The actuator can move parts to seal an orifice or be used as throttle in a channel. Passive valves have structures that

are designed to have a higher flow resistance in one direction than the other. A passive valve can also be done by means of capillary valves by drastically changing the geometry, or by increasing the flow resistance locally within the channel using hydrophobic zones. The performance of a valve is restricted by (1) the sealing properties against the valve seat, and (2) the ability to generate a force from the actuator or the passive structure (i.e. geometry or material properties). A good seal can be achieved by a large sealing area, and can be enhanced by choosing a soft material in one part of the valve [32]. For example, when used in a pump, the leakage and maximum pressure that a valve can work against will often define the performance of the pump.

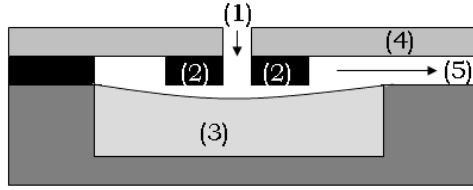
Paraffin wax is interesting in valves as it has the ability of producing high forces. For that reason paraffin wax has been used in active valves [33-34]. Even if one could expect high pressures of these valves, higher pressures than 160 kPa [35] have not been reported before. However, where paraffin has been used as latch in combination with external actuators [36-38], pressures between 275 kPa and 1,725 kPa are shown.

## A normally open active valve (Paper IV)

As part of the work presented in Paper IV where a polymeric paraffin pump is made, an active valve is evaluated using different types of mechanical clamping.

The valve was built using a paraffin actuator (Paper II). A channel structure built on a glass slide with valve seat and fluidic channels is glued to the actuator and fluidic connectors are then glued onto the chip. By activating the actuator, the membrane rises and seals against an inlet, and the valve opens when the actuator is switched off (Fig. 17).

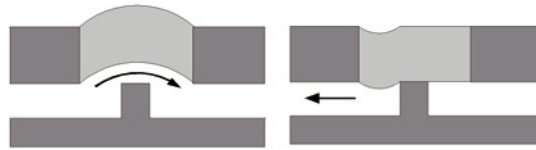
Rubber tubes and silicone glue did not give enough support for the glass substrate that was ruptured at pressures of 200 kPa. A stiff port enhanced the robustness, and the valve sustained pressures up to 350 kPa. Finally, the port is clamped with a spring clamp, and impressive pressures of 0.9 MPa were achieved at an input power of about 0.3 W without any visible leakage.



*Figure 17.* Schematic of the valve with the inlet (1) and valve seat (2) perpendicular to the membrane of the actuator (3), channel substrate (4), and the outlet (5).

## Passive valves

In Paper V and VI, passive valves are described to be used in contact with reservoirs. The valves should be able to hold pressures greater than the flow resistance in the outside channels to prevent contamination. They should also prevent backflow of fluid into the reservoirs after the reservoirs have been emptied. The valves are built of PDMS plugs that are cast or glued into drilled holes in polystyrene. The lid is glued against a channel substrate with valve seat. The function of the valves is described in Fig. 18.



*Figure 18.* Function of the passive valve that opens for an applied pressure (left) and seals against the valve seat for a negative pressure (right).

Different valve designs are tested with respect to opening pressure (Fig. 19). Designs A-C (Paper V) are glued to the valve seat and Design D (Paper VI) are mechanically clamped.

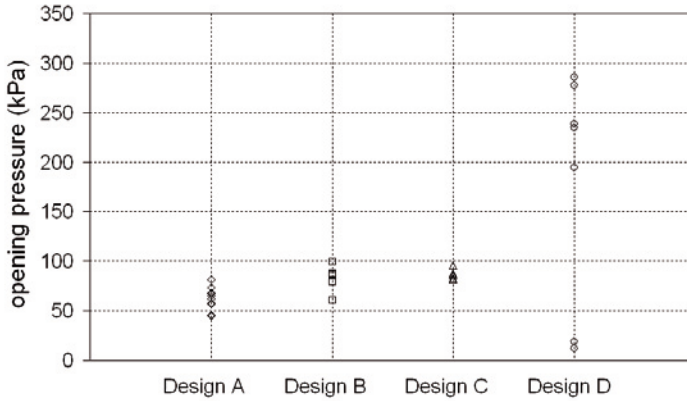


Figure 19. Comparison between valves with adhesive boning in interface design A-C (Paper V) and mechanically clamped valves, design D (Paper VI).

## Micropumps and injectors

Basically a micropump can be designed from one pumping chamber and two valves. By choosing passive valves and one pumping chamber, a reciprocating pump is achieved. The performance of such a pump is associated with the ability to continuously provide a transfer of energy to the continuously flowing fluid. These pumps are usually fitted with a piezoelectric driver that is driven with frequencies up to kHz. Even though flow rates of up to ml/min are reported, the maximum pressures of these pumps are normally about tens of kPa up to few hundred kPa. Small flow rates and non-continuous flow can be somewhat difficult to achieve due to leakage in the valves [31, 32].

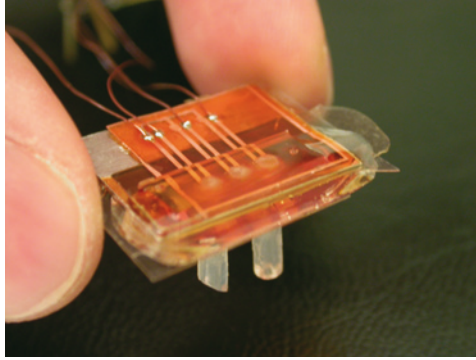
Using at least three or more active valves, a peristaltic pump can be realized. The peristaltic pump squeezes the fluid forward, and the performance of the pump is highly associated with the performance of its valves [31].

In some cases it can be more useful to use a single stroke to inject a fluid into a channel or chamber. Basically, if the peristaltic pump is dimensioned with a large pumping chamber, it can be used as injector and the whole, or part, of a stroke volume can be used.

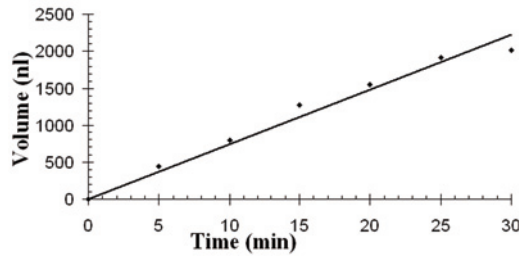
## A peristaltic pump (Paper IV)

A peristaltic pump (Fig. 20) is fabricated and tested in Paper IV. The pump is made in the same way as the active valve earlier described. The

overall size of the pump, excluding the out and inlets is  $25 \times 10 \times 2 \text{ mm}^3$ . Excluding the flexible printed circuits, the size is even smaller ( $12 \times 6 \times 2 \text{ mm}^3$ ).



*Figure 20.* A paraffin micropump with rubber tubing located over the inlet and outlet valves at the backside of chip.



*Figure 21.* Pumped volume as function of time for the micropump.

The pump was tested using a square wave of period 28.8 s and heater effect of 0.2 W gave 74 nl/min in average flow rate (Fig. 21). The pump was tested for 30 minutes that corresponds to about 60 cycles. The valves, as described earlier, are capable of sustaining pressures up to 0.9 MPa.

## Injection pump and a fluid interface (Paper V)

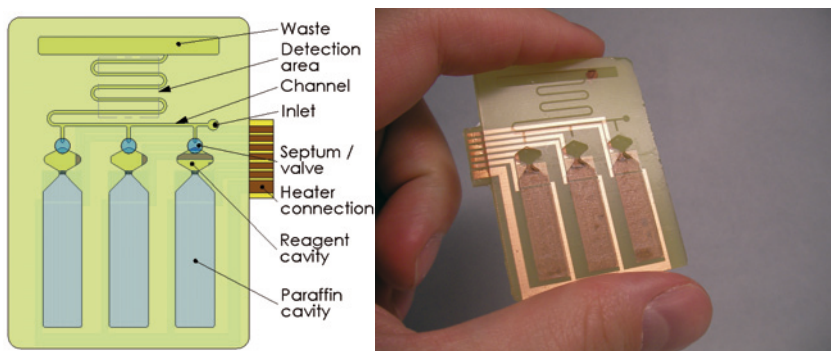
In Paper V a concept for using paraffin wax in direct contact with a fluid for injection is presented. The pump has a reservoir partly filled with paraffin and the rest with a fluid. The paraffin is filled during fabrication, and the fluid is filled via the rubber part of the passive valve (Fig. 22) using a syringe needle. The passive valve opens when the pressure is high enough, and the fluid is moved from the reservoir to a nearby channel. The paraffin will create a negative pressure in the reservoir as it starts to crystallize and the valve will close. A developed concept of this injector is described in Paper VI.



*Figure 22.* Schematic description of a septum for filling a fluid into a reservoir using a syringe needle that penetrates the soft rubber material.

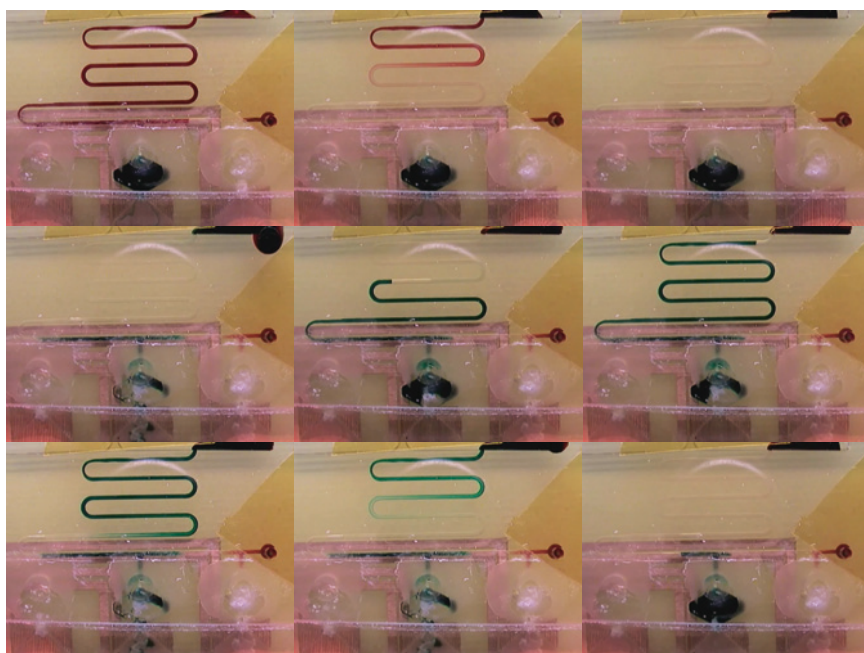
## Lab-on-a-chip (Paper V and VI)

In Paper VI the concept presented in Paper V is taken a step further and is integrated to a microfluidic chip. This  $55 \times 40 \times 4 \text{ mm}^3$  sized chip contains three individual fluidic reservoirs, paraffin actuators with integrated heaters, a fluidic channel that can serve as reaction chamber, passive valves, fluidic interface (septum) for loading of fluids to reservoirs, a sample inlet, and a waste (Fig. 23). The chip is designed to carry out sequential flow in less than 5 minutes, with a reaction chamber volume of  $10 \text{ }\mu\text{l}$ , total channel volume of  $15 \text{ }\mu\text{l}$  and, reagent volumes of  $20 \text{ }\mu\text{l}$ . Air bubbles are introduced to improve the rinsing function. Sequential flow is shown to work with three different coloured fluids and an air bubbles (Fig. 24).



*Figure 23.* A concept layout for the chip (left) and a real device seen without cover lid (right).





*Figure 24.* From above (left to right) a channel filled with colored fluid is rinsed with colorless fluid. A green fluid is then rinsed through the channel, and in the last row, a colorless fluid rinse the channel.



## Future perspectives

Without doubt, paraffin has the ability to be used in many MEMS applications in the future. On the other hand, how long it will take to reach a commercial MEMS market is an open question. However, a commercial product would be important for the research community to increase the focus on paraffin wax.

Rapid prototyping techniques were used throughout this thesis. This approach has often allowed fabrication of complex devices within a week and has given useful information in the development process of these components. For instance, the tape membrane can be used over a thousand cycles with a useful deflection. Also the valve performance can be enhanced by mechanical clamping instead of a stronger adhesive bond. However, these rapid prototyping techniques should be taken for what they are, namely a tool to quickly investigate properties and performances of a device but not for production precise structures or of large series at low costs. Hence, those paraffin actuator concepts that have potential of a larger societal interest should be transferred into devices produced with techniques more commonly used in MST or fine mechanics. Still, these devices made by rapid prototyping techniques quickly give answers that can be used for further development of a device or concept at several levels.

The most important factor to enhance the speed of these actuators is further miniaturization. But does size matter? Paraffin wax actuators have already many other advantages that compensate for speed and therefore finding applications where speed is not critical is maybe more important than maximizing speed. Nevertheless, it would be interesting to examine the limit for miniaturizing a paraffin actuator that is made by means of rapid fabrication using milling in PCB.

For the engineer who designs these actuators in future it would perhaps be useful to know how to choose the right paraffin. Moreover a simple model for how the paraffin actuator will respond to different inputs by choosing different heaters, dimensions of paraffin cavity, materials, and heat-sinks, is important. This could be provided by researchers. The devices need to be tested in more severe environment conditions. How well does a paraffin wax component work after being cycled in the military standard temperature range of -40 to 120°C? Of course, it is needed to test paraffin actuators to the standards of the application fields of interest. Finally, my opinion is that now these actuators have reached a point when they would gain more if effort

was put in other areas than process development and proof-of-concepts. For example, the drive and control have to be developed and integrated, and the need of closed-loops has to be investigated.

# Conclusion

Beside the large forces and large volume expansions of paraffin wax in miniaturized actuators, the low cost of paraffin in combination with a simple activation principle, is attractive. Further, the low voltage drive is very interesting for portable systems. The possibilities to modify paraffin to have more or less solid transitions are interesting, and the possibility to choose different melt temperatures for a paraffin wax are also attractive.

As shown in this work, paraffin wax can be fitted into many different components and systems, exploring it in different ways. Paraffin is well suited for high pressure microfluidics, sustaining pressures of almost 1 MPa in an active valve, and is not the limiting factor in achieving larger pressures. Paraffin can also be used in peristaltic pumps and in direct contact with a fluid in injectors. A multi-stable actuator is also made without extra mechanical parts, using several heaters to control the thermal gradients within the paraffin cavity. This actuator can be interesting to use in many systems where energy can be saved by using a latched actuator. In addition, active control added new dimension to the usefulness of paraffin actuators.



## Summary in Swedish

MEMS (mikroelektromekaniska system), från några mikrometer till centimeterstora, börjar bli en del av vår vardag och används exempelvis som sensorer i bilar, mikrospeglar i HDTV och som skrivhuvuden i bläckstråleskrivare, för att nämna några. Fördelen med MEMS och miniatyrisering är flera. Dels så finns möjligheten att sänka produktionskostnader för enskilda komponenter med satsvis tillverkning, eller att sensorer blir känsligare och effektivare i mikroskala. Inom bioteknik har miniatyrisering av vätskekanaler visat sig öka effektiviteten vid analyser, men andra effekter är att mindre mängder av vätskor behövs för att göra en analys. Man pratar då om *Lab-on-a-chip* och *micro-total-analysis systems* eller microfluidik.

Miniatyriseringen inom området microfluidik ger även möjligheter till att utföra diagnostiska hälsotester nära patienten, *point-of-care*. Med dessa tester kan man upptäcka sjukdomar eller sjukdomstillstånd som indikeras av till exempel höga halter av vissa ämnen i blod, urin eller saliv. Vinsten är att patienten själv skulle kunna utföra testet på liknande sätt som diabetessjuka idag testar sina blodvärden.

Tillförlitliga komponenter som ventiler och pumpar behövs i dessa chip men på mikroskala är detta svårt av flera anledningar. Ofta behöver man en motor (aktuator) som driver dessa pumpar och ventiler, men när man minskar skalan kommer förflyttningar och krafter att minska. I värsta fall orkar aktuatorerna inte av att hålla emot de tryck som uppkommer i miniatyriserade system. Utöver de fysiska aspekterna så spelar kostnaden för material och tillverkning stor roll för vilken aktuator som kommer att väljas. Idag finns det inget entydigt svar på vilken typ av aktuator som är bäst lämpad. Detta skiljer sig från fall till fall. Mer forskning krävs alltså för att hitta lämpliga aktuatorer som är effektiva men också relativt billiga.

Detta arbete fokuserar på paraffin som aktuatormaterial i miniatyriserade aktuatorer. Paraffin har visat sig vara intressant i mikrosystem på grund av dess stora volymexpansioner och förmåga att producera stora krafter. Etersom man traditionellt sett arbetar med kisel i MEMS, för att bygga sina strukturer, har tidigare paraffinaktuatorer främst varit kiselbaserade. Men, inom området microfluidik har man insett att det finns många fördelar med polymera strukturer. En anledning är att tillverkningsmetoderna för dessa är enklare och snabbare jämfört med traditionell kiselprocessning. Komponenterna i detta arbete är därför framtagna med tillverkningsmetoder som är

lämpade för plaster. Dessa metoder är dock mest lämpade för att snabbt ta fram komponenter (prototyper) för att visa fungerande koncept.

I paper I studeras hur man kan blanda två rena paraffiner (n-alkaner) och erhålla en stegvis expansion med varierande storlek beroende på grad av blandning. Utvärderingen är gjord med två olika metoder; en där man studerar entalpi (värmeupptagning) i materialet och den andra där man studerar volymexpansionen.

I paper II tillverkas en polymerbaserad paraffinaktuator med integrerade värmare i paraffinet och som membran användes en tejp av polyimid. Resultatet visade att aktuatoren trots kunde köras över tusen cykler med användbara förflyttningar mot en i sammanhanget hög punktlast.

Tidigare presenterade paraffinaktuatorer kräver kontinuerlig tillförsel av energi för att hålla en given utböjning. I paper III undersöks om paraffinet självt går att använda till att låsa utböjningar i olika positioner. Genom att använda sig av flera lateralt placerade värmare kan man lokalt smälta paraffin och styra stelning varvid flera stabila positioner går att uppnå. Denna funktion är viktig i system där man vill bibehålla en position men samtidigt spara på energi.

I paper IV byggs en peristaltisk pump som klämmer fram vätska med hjälp av två ventiler och en pumpkammare. Aktuatorerna som används är identiska med de presenterade i paper II. Denna för tillfället unika mikropump med paraffin som aktivt material har ventiler som klarar upp till 0.9 MPa mottryck utan synligt läckage, vilket är mycket högt.

Papper V och VI visar koncept som är tänkta att kunna användas i portabla *Lab-on-chip*. Här behandlas frågeställningar och koncept visas för förvaring av reagenser på chip, fyllning av dessa vätskor genom ett interface, förflyttning av vätskorna från dess behållare till intilliggande kanalsystem med hjälp av integrerade paraffinpumpar och passiva ventiler.

Slutsatsen är att paraffin är ett aktuatormaterial som har flera fördelar lämpliga i mikrosystem. Inte minst har detta visats för tillämpningar inom mikrofluidik med höga tryck och innovativa lösningar med *Lab-on-chip* som tillämpningsexempel. Paraffin är dessutom väl anpassat till att användas med tillverkningstekniker ämnade för *rapid prototyping* och *rapid fabrication* i polymera material. Den slutgiltiga bedömningen är att paraffin mycket väl kommer att passa i kommersiella produkter med MEMS-komponenter i framtiden. Kanske som en låsbar aktuator då det börjar bli allt viktigare med energibesparing i många produkter.



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Frank Zappa and Mike Keneally

To all my friends and families...

Luv!

Marcus Lehto  
Uppsala 2007-08-13



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