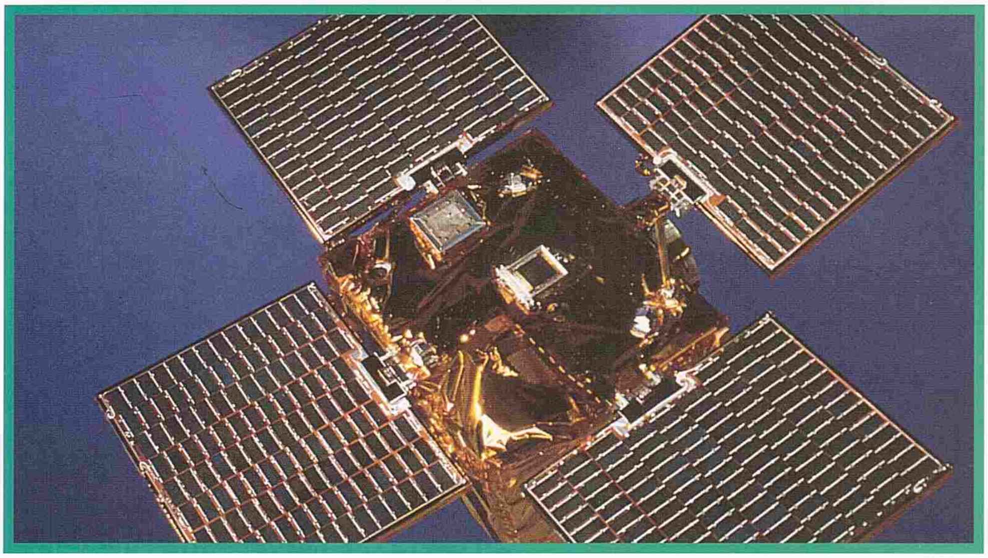
MICRO STRUCTURE BULLETIN

Newsletter for Scandinavian Micro Structure Technology No.2 May 1995

MST in Space



The Swedish microsatellite, Astrid, was launched in January with the purpose to investigate near-field plasma. Astrid represents a new philosophy for satellites with its 0.5 meter diameter, 28 kg weight, and roughly one year of development time.

Microsystems will play an important role in future space activities. How fast and how extensive will this development be?

To explore these questions, ESTEC (the European Space Agency Technology Research Center) organized a "Round Table on Micro/Nano-Technologies for Space". During the two day meeting, views were shared, ideas discussed, and problems to be solved identified.

MST are useful for space applications for several reasons, including a reduction in cost and increased performance within the available mass, volume and power.

One critical aspect is the ability to reliably withstand harsh environmental conditions. The systems *must* function under all circumstances. Protection against, vibration, radiation and temperature changes can necessitate large and heavy components for which only a minor part of the developmental effort involves the MST-based part itself.

Several space missions have already flown units containing micro-devices. MST-technology made possible a bioreactor which formed part of a microgravity laboratory onboard a shuttle flight in 1994. Limited space was a critical design restraint, and space-specific problems, such as the zero gravity-induced accumulation of gas bubbles in the pumps, had to be addressed.

A more futuristic, but still realistic, possibility is a nano-satellite. Stacking wafers with different functions, for example solar cells, electronics, and mirrors, make it possible to create communication and observation satellites. It is also feasible to have micro-propulsion systems with micromachined thrusters.

Hundreds of nano-satellites (each 10 cm in diameter) can be used to provide a complete coverage of the surface of the earth. Removing the satellites from orbit at the end of their lifetime becomes essential. Satellites as small as 2×2×2 cm^3 cube were also discussed. Compared to larger satellites, nano-satellites are better developed by smaller and more close-knit multi-disciplinary engineering teams.

Jan Söderkvist

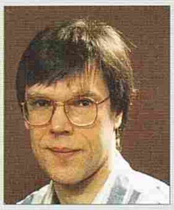
CONTENTS

EDITOR'S NOTE

This issue, devoted to liquid/gas handling and analysis systems, features the first article in a series describing MST applications. This series will continue in the last issue this year with medical applications. *Micromachining Basics* and *Micromachining Applications* will alternate.

The work of the editors will now concentrate on the *Transducers • Eurosensors* conference in Stockholm. Selecting highlights from the about 500 presentations is likely to cause some headache. By attending *Transducers • Eurosensors*, you can fully appreciate the excellent work of the Organizing Committee, headed by Prof. Lundström from Linköping University. Do not forget to register also for the European workshop *Silicon Accelerometers* that will be hosted by Uppsala University just prior to *Transducers • Eurosensors*.

The driving force of industrial development is: "It's human nature to stretch, to go, to see, to understand. Exploration is not a choice, really, it's an imperative", as the Apollo astronaut, Michael Collins, summarized. New products will be the results. The European network NEXUS (*MSB 95:1*) is therefore likely to soon have industrial members also from Sweden and Finland.



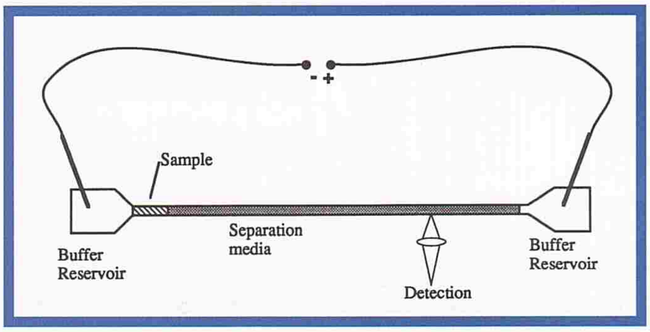
Jan Söderkvist

Micromachining Applications Part 1:

Electrophoresis in Plastic Channels

Nature has provided living organisms with a vast quantity of different molecules for the survival. When studying these molecules, the need often arises to separate them from one another.

One widely used separation technique is gel electrophoresis. The sample containing the molecules to be separated is placed in a gel located in an electric field. The field forces the electrically charged molecules to move with an electrophoretic mobility determined by factors such as charge, size, shape and characteristics of the surrounding media.



Electrophoretic setup

Shape of Gel

Electrophoresis performance is defined by different parameters, such as size of the sample plug, the separation power of the gel and the spatial resolution of the detector. Another important factor is the heat dissipation in the gel. If the temperature gradient across the cross-section of the gel becomes too large, the separation band will be distorted due to the temperature dependence of the mobility.

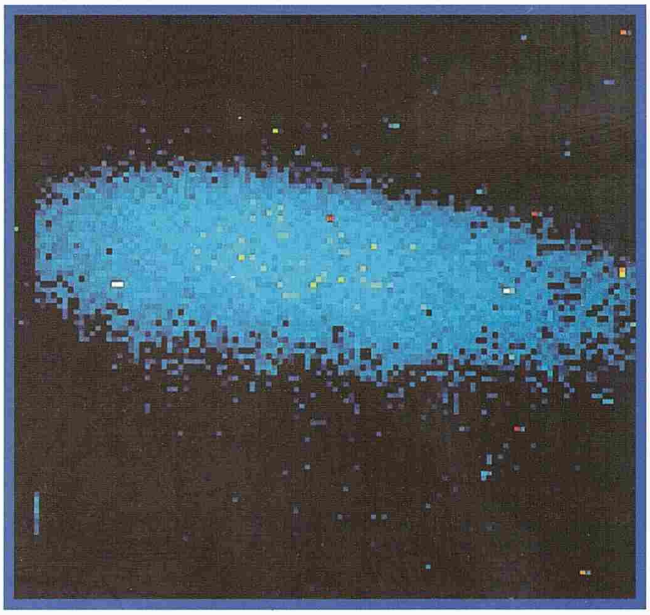
A finite element analysis program, ANSYS, was used to determine the heat gradient for both the continuous 0.35 mm thick standard gel and for a gel formed as a channel 0.35 mm high and approximately 1.5 mm wide. The analysis shows that with an identical heat load, the temperature gradient across the channel-shaped gel is only 50% of that of a continuous gel. This margin could be used to increase the separation speed by doubling the electric field strength.

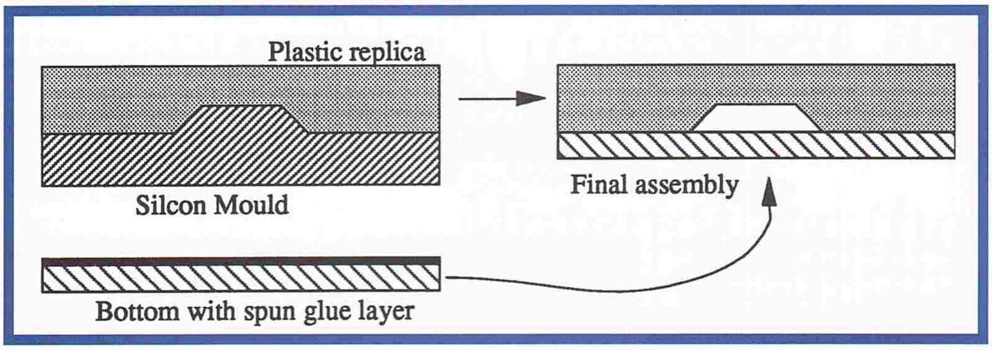
Plastic Replicas

In order to decrease the separation time, shorter separation channels have been manufactured with replica techniques. A negative mold is etched out from a silicon substrate using standard micromechanical techniques. Channel dimensions in this example are 0.35×1.5 mm in cross-sectional area and with a separation length of 50 mm.

The mold is then used to emboss the channel into a fluoroplastic material, Hostaflon TFB7100. Inlet and outlet holes to the channel are punched. To complete the channel, a top layer of Hostaflon is glued to the channel part by spinning out a thin layer of the base polymer diluted in solvent. After the solvent is evaporated, the two parts are pressed together at high temperature, forming a tight seal.

The high electric field that is required during the electrophoresis is the most important reason for making the channels in plastics instead of directly using the silicon originals. Shielding the semiconductor material is in principle possible, however, pinholes in the



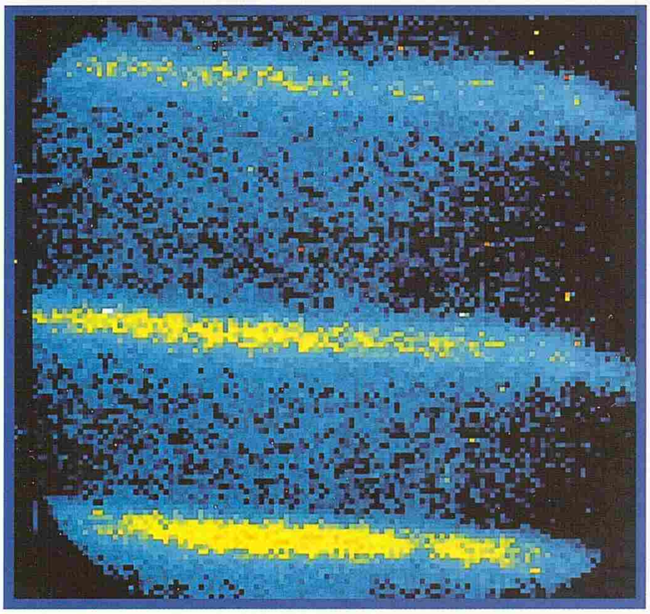


Formation of a plastic channel

passivation layer will cause a fatal breakthrough. Plastic materials do not have these problems. In addition, very good dimensional stability can be achieved at a low cost with replica processes.

Electrophoretic Method

The separation media is introduced into the channel with an injection needle. It is then polymerized with UV-light. In the example described here, a poly-acrylamide gel is used. A small portion of the inlet of the channel is kept clear from separation gel to enable the sample to be easily introduced.



Band width for a standard continuous gel (left) and a channel gel (right).

As a test sample, a fluorescent labeled DNA size ladder is used consisting of a mixture of different lengthed strands of DNA, 50 base pairs apart, ranging from 50 to 500 base pairs in total length. The sample is then introduced at the inlet of the channel and the voltage is applied. The sample molecules will electrokineticly be stacked against the separation gel, giving rise to a 100 µm thick sample plug. The underlying mechanism behind stacking is that the sample molecules have a higher mobility in the free solution than in the gel.

Fluorescent Detector

For maximum separation, a detector with the same spatial resolution as that of the sample plug is needed. A confocal laser induced fluorescent microscope is a good candidate for test trials. This configuration has a very high geometrical resolution in the focal plane and allows signals from only a thin slice of the object in the focal plane to reach the detector. This results in a high signal-to-noise ratio. Of course, it is not feasible to use this microscope in products. Nevertheless, it is a very valuable detection technique for prototype work.

Results

Results from DNA electrophoresis testing on the plastic channel replicas indicates that separation time can be decreased to at least 15 minutes for each 300 base pairs sequence compared with two hours for commercial systems based on continuous slab gels. The main reasons for this improvement are the introduction of narrow sample bands onto the gel and the small sample volume. Although more complicated, both of these parameters could be better controlled in a slab gel system as well, but channels give additional advantages, such as a well-defined sample introduction compartment and easy alignment of the detectors with the gel.

In the figures, the difference in the width of the bands is easily seen. In addition, heat loads on the gel can be minimized with channels, since heat is generated only where separation occurs and that heat is more effectively conducted away. Future work will determine whether channels will win over slab gels.

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MSK '95

As a follow-up to the successful workshop, *MSW*, last year, a two day micromachining course was held in Uppsala on March 28–29. This course was well received by the 52 participants who predominantly came from industry. A new and extensive course material, almost 350 pages long, is available. *MSK* covered subjects like materials, processing techniques, sensor and actuator principles, and industrial aspects.

As previously decided, *MSK* will be held again two years from now. Next year, the second *Micro Structure Workshop* (*MSW '96*) will be held.

Jan Söderkvist
Coordinator MSK, MSW

Applied Physics in Linköping

Interdiciplinary Research in the Field of Micronics

The research at the Laboratory of Applied Physics is interdisciplinary. The members of the laboratory are electrical engineers, physicists, chemists, biochemists and biologists. Several projects fall within the area defined as "Micronics", since they combine biological, chemical or physical phenomena with small geometrical structures. The research activities also contain fundamental interdisciplinary research predominently within surface physics. Many projects are run in collaboration with other laboratories in the Department of Physics and Measurement Technology and with external collaborators. The Laboratory has been appointed as one of NUTEK's competence centers, the Center for Bio and Chemical Sensor Science and Technology, "S-SENCE". The research areas include the following, of which the three first areas will be further presented:

- chemical and biosensors
- catalytic reactions
- conjugated polymers
- bioprocess measurement technology and micronics
- interface biology
- biomaterials
- thin film optics
- scanning force microscopy
- fourier transformed infra-red and Raman spectroscopy

Chemical and Biosensors

The main effort within this area is to develop chemical sensors based on Metal-Insulator-Semiconductor (MIS) structures with gates of catalytic metals, and to increase the understanding of the physical and chemical properties of such structures. Also, new applications of chemical sensors are investigated, often in cooperation with external collaborators.

One area that has already been commercialized is the use of neural networks for evaluating the response of sensor arrays in an electronic nose. The sensor array normally contains MISsensors, commercially available tin dioxide sensors and a carbon dioxide monitor. A number of applications, such as the identification of various types of papers and estimation evaluation of the quality of food, have been investigated. In a recent project, it was shown that the electronic nose could be used to estimate whether tomatoes had been irradiated or not. Classification of bacteria into different types has also been demonstrated. In addition, it has been shown that fungal and bacterial infections of grains can be detected. One such instrument is now available from Nordic Sensor AB.

A development of gas sensitive field effect devices based on silicon carbide instead of silicon has recently started. These sensors can be operated at much higher temperatures, as will be described below.

Another area that will be further explored is the creation of artificial olfactory images. Continuous sensing areas with gradients in metal composition and temperature are evaluated by a scanning light spot that creates an image of the gas mixture supplied over the sensor area. This scanning light pulse technique can also be used to study the spillover phenomena on the surrounding oxide of a catalytic metal gate.

The portable electronic nose.

Catalytic Reactions

Catalytic reactions on thin catalytic metal films are studied to increase the understanding of the sensing mechanism of field effect structures. Basic knowledge regarding the catalytic reactions is also sought. Studies are performed both at UHV and at atmospheric pressure. Kelvin probes and capacitance-voltage (C(V)) measurements are performed together with mass spectrometer analyses. The UHV system is also equipped with surface analysis tools.

One interesting project uses a palladium membrane to separate reactants and products from each other in chemical reactions. Thus, the equilibrium of the reaction is shifted by lowering the chemical potential on one side of the membrane.

Long (about 5 cm) and very narrow (100 nm) channels are fabricated using micromachining in silicon, somtimes with a lid of glass. The transport and chemistry of molecules that pass along these long and narrow channels are studied by placing a mass spectrometer at the channel end. The interior of the channel may be coated with different catalytically active metals. Since every molecule that passes through the channel has had a very large number of wall contacts (proportional to $(L/d)^2$), it is believed that this could be a very efficient way of studying improbable catalytic reactions.

Conjugated Polymers

In the area of polymer electronics, substituted polythiophenes incorporated in polymer light emitting diodes are now capable of covering the full visible spectrum into the near infrared. These materials give electroluminiscence peaks from 460 nm to 800 nm (blue, green, orange and red). LED's based on blends of these polymers, which are self organized nanostructured materials, give voltage controlled color sources.

Very small light sources are obtained using nanometer sized electrodes as contacts in polymer LED's. Light sources as small as 100 nm have been verified with optical microscopy. Micro-fabrication of integrated polymer LED's and polymer waveguides, as well as micro-cavity effects in polymer LEDs, are under study. Micro- and nano-structures are used to study electrical transport in doped oriented polymers.

The conducting polymers change volume due to doping or undoping. Micromuscles have been created by micromachining techniques. Small fingers of size 10 µm × 10 µm and recntly small boxes, size 100 µm × 100 µm × 100 µm have been produced. In a liquid environment they can be opened or closed by changing the applied voltage from –1.0 V to + 0.5 V. This work will be further described in a coming issue of this journal.

OUTLOOK: Smart Miniaturized Sensor Systems

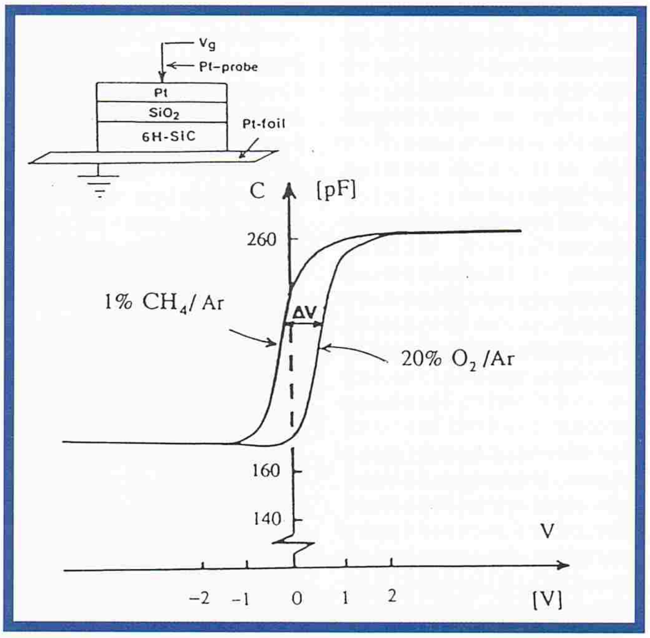
Several of the described technologies lend themselves to be included in micro-structured devices. The MIS-structures are suitable for integration with micromachining, since they are based on standard semiconductor technology. Sensor arrays can be combined with a miniaturized gas mixing systems consisting of valves and cavities, some of which can hold small quantities of solvents used for the calibration of the sensors. A palladium membrane can be used to create hydrogen as a reference, or to increase the yield of a chemical reaction. Integrating narrow channels into the system is another possibility to increase the likelihood of an improbable reaction and to separate the constituents of a gas mixture. The integrated electronics should contain addressing to the different sensors in the array as well as a part for the evaluation of the signals. The output from a two dimension array of gas sensors could be visualized as an "olfactory image" on a screen with the aid of conducting polymer materials.

Anita Lloyd Spetz

Silicone Carbide, a High Temperature Sensor Material for the Future

Catalytic Metal-Oxide-Silicon Carbide, MOSiC structures, are used as gas sensors at high temperatures. These devices can be operated up to at least 700°C. They respond to gases like hydrogen or hydrocarbons, in either oxygen or inert atmospheres. A catalytic metal gate of platinum and tantalum silicide (100+10 nm) gives a very rapid gas response, as shown in the figure below. The signal changes abruptly from a low signal for hydrocarbons in an excess of oxygen, to a high signal for an excess of hydrocarbon. The MOSiC devices give a response complementary to that of a lambda sond. This makes them useful for many applications, for example, in the exhaust gases of cars for a fast and efficient control of the fuel to air ratio.

Silicon carbide is a semiconductor material with a large bandgap (2.9 eV for 6H-SiC compared to 1.1 for Si) and a high thermal conductivity (4.9 W/cm•K for SiC compared to 1.5 for Si). This gives the advantages of a high operating temperature and a very high density of components. Other groups are working on the development of electronics in silicon carbide. This will enable integrated smart sensor systems to be placed on one silicon carbide chip. The sensor chip can be used in high temperature environments, such as inside an engine or turbine. It might also be possible in the future to make micromechanical structures in silicon carbide. For example, heating of the sensor will consume very little power, if a sink is etched out underneath the sensor structure. This sensor system is already realized in silicon, but would be much more efficient in silicon carbide.



The typical capacitance voltage curves of MOSiC sensors (see insert) show a negative shift of the flat band voltage when the ambient contains hydrogen containing gases (650°C).

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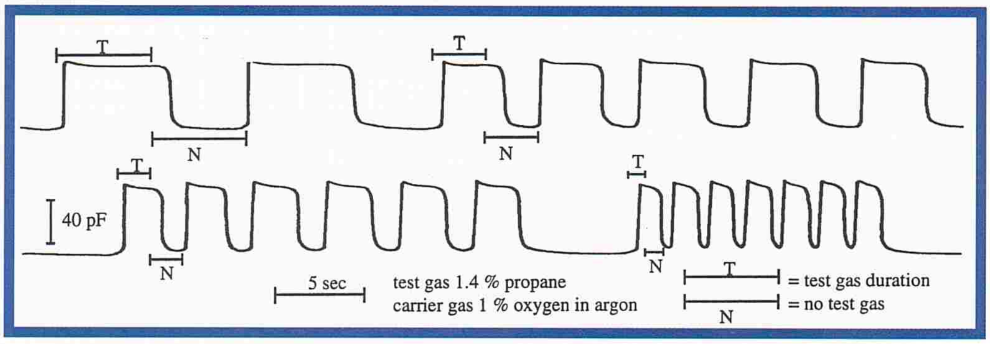

Changes in capacitance at a constant voltage when propane (1.4%) is pulsed into oxygen (1%). The time constants (and delays) are determined by the gas mixing systems. The time constants of the sensor are shorter than 100 ms (550°C).

Flexible Network Creates New Companies

Big organizations achieve greater efficiency by reducing hierarchies into smaller units by networking downwards. Small companies can network together to become stronger. In Sweden, a new network has proven very constructive in creating new resources and knowledge into new ventures. Over the last two years, three higly skilled small industries have been created.

Analysis and separations equipment, such as HPLC pumps, solvent degassers, on column capillary fluorescence detectors, closed flow titrators, are some examples of new instruments currently on their way to the market. The various inventors involved have common interests in matters such as finance, production facilities, opto-electronic skills, and mechanical and micromechanical specialties. By connecting the inventors with interested reference customers (for example, Nobel Chemicals, and Universities like Royal Institute of Technology in Stockholm and the Center for Surface and Microstructure Technology in Uppsala) on one hand, and financial, production and management resources on the other, the dynamics of these new companies becomes very convincing. In addition, a sales organization recently joined the network to take care of the marketing of new instruments.

Other inventors will find a home in this environment and they are welcomed. They will find that they can remain concentrated on the tasks they do best and leave many other tasks to specialists provided by the network.

Micromechanical structures enter the network on different levels. One company is considering using a micromechanical pressure sensor designed for very high pressures. Micro chromatography columns are being considered, and as miniaturization is a key issue in the next generation of analytical equipment, micromechanics becomes very important.

Locating the network in Karlskoga, Sweden, offers an immediate advantage in the access of well trained personnel for the development and manufacturing of instruments. As Karlskoga is the hometown of Bofors, a well known defense contractor, the network will also finds micromechanical synergies.

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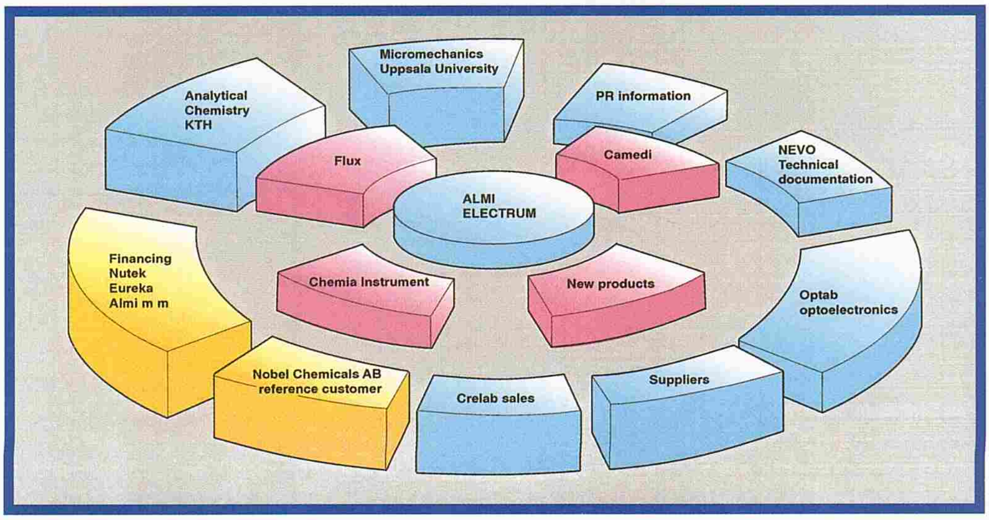

A high technology network for supporting academic and commercial users. Their combined competence creates new jobs and export business in the troubled Karlskoga area of Sweden. Almi and Electrum in the center, are involved in the financial and support matters of newly started enterprises. The red squares show new companies and products attracted to Karlskoga. Blue squares represent specialist functions, and the yellow, support functions.

Dissertations

Mårten Jansson

MSB congratulates Mårten Jansson, the Royal Institute of Technology, on successfully having defended his Ph.D. thesis, *Miniaturization and Improved Separation Methods for Capillary Electrophoresis* (ISBN 91-7170-808-1), on February 24. Reducing the size of a electrophoresis column is advantageous in respect to heat dissipation. However, reducing size degrades sample capacity and detection sensitivity. Thermal aspects limit the electric field strength more for circular than for rectangular capillaries with a high cross-sectional aspect ratio. In this context, the use of silicon as column material is advantageous.

A multi-sample holder for capillary electrophoresis was fabricated on a single crystalline silicon wafer. A large number of well-defined cavities, with volumes in the nanoliter range, were created by anisotropic etching. A thin layer of gold on the surface served as a cathode during electrokinetic injection of the sample. Evaluations were carried out by injecting and separating oligonucleotides using capillary gel electrophoresis. No loss of performance was observed when compared with injections from classical sample vials.

Thomas Laurell

On May 19, Thomas Laurell, Lund University, presents his Ph.D. work: *Microdialysis and Continuous Glucose Monitoring – Towards Wafer Integration* (ISRN LUTEDX / TEEM -- 1056 -- SE , ISSN 0346-6221). His work studied a system for the continuous glucose monitoring in diabetics. The system is based on a microdialysis probe used as a subcutaneous glucose sampling unit, a glucose oxidase enzyme reactor, and a Clark-type oxygen electrode.

An enzyme reactor consisting of several deep parallel channels was created with anisotropic etching of silicon. The reactor measured 3×15 mm^2

A Resonant Silicon Tube Structure for Fluid Density Measurements

The silicon sensor and actuator group at the Royal Institute of Technology, Stockholm, has designed the first resonant silicon tube structure for fluid density measurements. The silicon structure consists of a planar double-loop tube, as shown in the figure. This structure is oscillated in a high-Q (2,800 in air and 8,000 in vacuum) torsional resonance mode. A high Q with low energy losses, is important for high resolution and stability. Using a balanced vibration mode and a material with a high intrinsic Q ensures this.

The detection principle is based on that a change in density of the fluid within the tube affects the mass loadning, and thus the resonance frequency, *f*. The mass loading follows a general relation, $f^{-2} = C \cdot A_f\rho_f + A_t\rho_t)/(A_f+A_t)$, where C is a constant that depends on the loop dimensions and vibration mode, ρ_f and ρ_t are the densities of the fluid and the tube material, and A_f and A_t are the cross-section areas of the fluid and the tube wall.

It can be concluded that the sensitivity is maximized if the tube to fluid area ratio is minimized, i.e. if the tube is made thin. Furthermore, the density of the tube material should be as low as possible to achieve a high sensitivity. The theoretically expected density sensitivity of 200 ppm/(kgm^{-3}) agrees well with the measured sensitivity of about 230 ppm/(kgm^{-3}). The measured temperature sensitivity was only -23 ppm/°C.

The fact that silicon has a lower density (2,300 kg/m^3) than steel (7,900 kg/m^3) means that a silicon densitometer with an area ratio of 0.65 has a 2.5 times higher sensitivity than a similar structure in steel (at a fluid density of 1,000 kg/m^3).

The structures are fabricated using double-sided anisotropic KOH silicon etching of two silicon wafers bonded together using the Silicon Fusion Bonding technique. This technology provides a high dimensional control. The SEM-photo shows a cross-sectional view of the tube. The silicon tube system (without the frame) is 8.6 by 17.7 mm, the wall thickness 0.1 mm and the external thickness of the tube 1 mm. The structure is electrostatically excited and the vibration optically detected.

The tube structure can be oscillated in many other ways: optically, electromagnetically or piezoelectrically. Thermal excitation and piezoresistive detection is possible. Electrostatic excitation and capacitive detection is a very suitable combination for this type of silicon micromachined resonant device.

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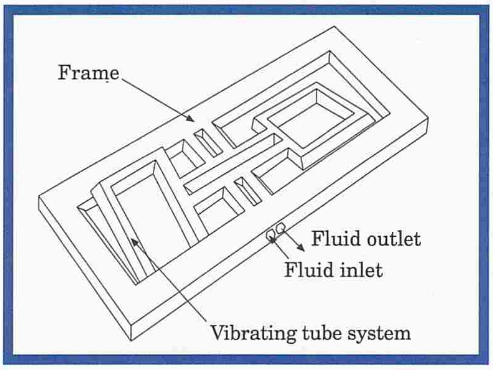
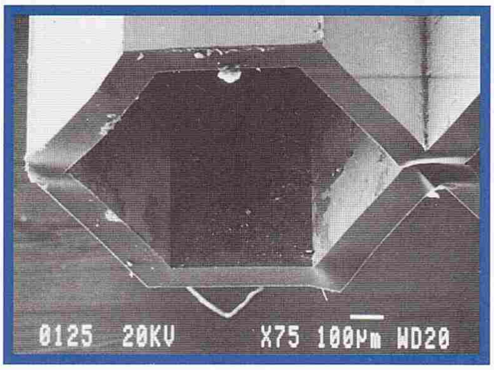


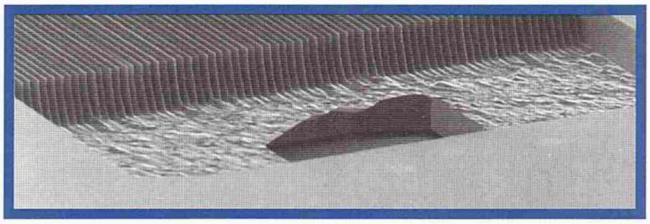


The fluid density sensor design and torsional vibration mode (left). An SEM-photo of a cross-section of the silicon tubes (right).

and offered either 30 channels, 165 µm deep, or 75 channels, 235 µm deep. Glucose oxidase was immobilized onto the structures. Measurements displayed a three times higher enzyme activity (EA) for the 75-channel reactor due to its larger surface area.

A new approach to increase the available surface area in the silicon reactor is to use an anodized current-controlled electrochemical hydrofluoric etch. This results in a thick porous surface layer that can extend all the way through the wafer. Structures formed with a 10 mA/cm^2 current density display a more than 30 times increase in EA compared to an unetched wafer. Lower current densities yield higher EA due to a finer porosity.

MSB takes the opportunity to congratulate Thomas Laurell in advance, and wishes both Mårten Jansson and Thomas Laurell a well deserved summer vacation.



The micromachined enzyme reactor exhibits a large surface area for enzymatic reactions. The inlet hole is in the lower part of the figure. For clarity, the photo was taken before the cover was added.

PUBLICATIONS

Some MST-related results published during the last months:

- Silicon Wafer Integrated Enzyme Reactors; T. Laurell, J. Drott (LTH) and L. Rosengren (UU); *Biosensors & Bioelectronics*, **10** (1995) 289–299.
- The Effects of HF-cleaning Prior to Silicon Wafer Bonding; K. Ljungberg, Y. Bäcklund, A. Söderbärg (UU) M. Bergh, M.O. Andersson, and S. Bengtsson (CTH); *J. Electrochem. Soc.*, **42** (4) (1995) 1297–1303.

MICRO STRUCTURE BULLETIN NO.2 MAY 1995

NEXT ISSUE

Next issue will report on highlights from the conference TRANSDUCERS'95 • EUROSENSORS IX

Silicon Accelerometers

A NEXUS workshop on silicon accelerometers will be organized in Uppsala prior to *Transducers • Eurosensors*. The intention is to give an insight into the typical problems and solutions encountered when developing accelerometers and related sensors. International experts will discuss both academic and industrial aspects.

For more information, please contact Prof. Bob Puers, K.U., Leuven, Belgium (Fax: +32-16-32 19 86) or Jan Söderkvist (Fax: +46-(0)8-510 116 15).

MME '95

The 6th *Micromechanics Europe* workshop in Copenhagen this September will focus on the physics and technology of micromechanical devices, microsensors and microsystems. *MME* is unique in that it not only seeks to "take the pulse" of European research in the field, but also leaves ample time for participants to be informally brought up to date through discussions and "in-depth" reviews.

For more information, please contact Prof. Otto Leistiko, MIC, Denmark (Fax: +45-4588 7762).

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A ✂-sign on your address label means that there is a 20% risk for each issue of you being removed from the mailing list.

The editors also encourage you to put *MSB* on circulation.

FUTURE EVENTS

Knowledge Fair in Biotechnology, Göteborg, Sweden, June 15–16, 1995. For information contact: Annika Derner, Fax: +46-(0)31-778 14 65.

Silicon Accelerometers, see separate note.

Transducers '95 • Eurosensors IX, Stockholm, Sweden, June 25-29, 1995. For information contact: Congrex AB, Fax: +46-(0)8-612 62 92.

MME '95 (MicroMechanics Europe), see separate note.

Laser Microengineering (course), MIC, Lyngby, Denmark, Sept. 12–13, 1995. *NEXUS may pay your registration fee if you act quickly.* For information contact: FSRM (Switzerland), Fax: +41-38 24 71 45, or Matthias Mullenborn, Fax: +45-4588 7762.

MicroSim '95, Southampton, UK, Sept. 26–28, 1995. For information contact: Sue Owen, Fax: +44-170 3292 853.

Materials for Microstructures (course), Uppsala, Sweden, Dec. 5–6, 1995. *NEXUS may pay your registration fee if you act quickly.* For information contact: Jan Söderkvist, Fax: +46-(0)8-510 116 15.

MEMS '96 (Micro Electro Mechanical Systems), San Diego, U.S.A., Feb. 11–15, 1996. *Abstract deadline: Sept. 15.* For information contact: Preferred Meeting Management Inc., Fax: +1-(619) 298 3459.

THE AIM OF the *Micro Structure Bulletin* is to promote micromechanics and micro structure technology. It constitutes one part of Uppsala University's effort to share scientific and technological information.

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