

SYMPOSIUM Y

Materials, Mechanisms, and Systems for Chemical and Biological Detection and Remediation

April 14 - 15, 2004

Chairs

Bill Colston

Lawrence Livermore National Laboratory
L-174
7000 East Ave.
Livermore, CA 94550
925-424-5854

Vinayak Dravid

Dept of Materials Science & Engineering
Northwestern University
Evanston, IL 60208
847-467-1363

Jay Grate

Pacific Northwest National Laboratory
K8-93
P.O. Box 999
Richland, WA 60208
509-376-4242

Roberto Raiteri

Dept of Biophysical & Electronic Engineering
University of Genova
Genova, 16145 Italy
39-10-353-2762

Thomas Thundat

Oak Ridge National Laboratory
MS-6123, 4500S
Oak Ridge, TN 37831-6123
865-574-6201

* Invited paper

8:30 AM *Y1.1

Micromechanical Sensors. James Gimzewski, Dept of Chemistry and Biochemistry, University of California-Los Angeles, Los Angeles, California.

Abstract Not Available

9:00 AM *Y1.2

Analysis of Materials Using Micromechanical Cantilever Sensors. Ruediger Berger, Susana Moreno Flores, Gina-Gabriela Bumbu, Liangsheng Ling, Renhua Zhang, Karlheinz Graf and Jochen Gutmann; Max Planck Institute for Polymer Research, Mainz, Germany.

An essential part of scanning force microscopy is a micromechanical cantilever which transduces a force acting on the tip into a deflection or a resonance frequency change. However, not only normal forces acting on the tip lead to a deflection, but also expansive or contractive forces acting on one side of the cantilever surface result in a bending. Frequency changes of the resonating cantilever can also be induced by a change of the mass loading of the cantilever sensor. This behavior together with their very small size (typically 0.5 μm thick, 50 μm wide and 500 μm long) make them very attractive as sensors, to study the interaction of molecules and to detect small amounts of materials. Changes of tensile and compressive surface stress arise when molecules specifically adsorb on one side of the cantilever surface. In the case of a self assembled monolayer of alkanethiol molecules this leads to a compressive surface stress change. It was found that the compressive surface stress change is proportional to the length of the alkyl-chain. This sensor principle can be extended by using a coating of receptor molecules which have specific functionalities such as key-and-lock binding. Molecules in the environment, which have a specific lock, can now bind to the appropriate key-receptor on the cantilever sensor. This binding causes a deflection of the cantilever and the signal can be used as a monitor for environmental control. In particular, the surface stress changes can be measured in liquid, which is a requirement for most biochemistry applications. In the field of biotechnology, DNA hybridization between self-complementary strands leads to conformational changes, which result in a cantilever sensor bending. This technique was used to differentiate a single base mismatch between 12-mer oligonucleotides. Polymers can be used as a model system to study the influence of different functional groups on the effect of surface stress change. Polymer brushes have been synthesized on cantilevers and are exposed to different types of solvents, which induce conformational changes in the polymer chains. A sample - typically a few nanograms - can be attached to the apex of a cantilever. Mass changes of this sample can be determined with picogram resolution by detection of resonance frequency changes of the cantilever sample system. A mass changes can result from swelling of a polymer in a solvent. Results of the swelling behavior of Polystyrene spherical particles with μm -diameters in toluene are discussed. Tracking the resonance frequency in dependence of temperature allows to study chemical reactions of very small samples. Quantitative analysis of dehydration, desorption and decomposition reaction of pico- to nanogram materials are presented. The small size of the cantilever offers the possibility to arrange several of these cantilever sensors in an array on a single chip or to use several chips carrying individually coated cantilevers.

9:30 AM Y1.3

Explosive Vapor Detection Using Uncoated Silicon Microcantilevers. Lal Pinnaduwa¹, Dechang Yi¹, Richard

Lareau² and Thomas Thundat¹; ¹Oak Ridge National Lab, Oak Ridge, Tennessee; ²Department of Homeland Security, Atlantic City, New Jersey.

Fast and sensitive detection of explosive vapors is important for a number of applications ranging from passenger baggage-screening at airports to disarming of landmines. Such a sensor system based on silicon cantilevers would be additionally desirable due to its compactness and possible low cost. We recently reported a novel detection scheme based on the deflagration of explosive material deposited on a piezoresistive microcantilever by heating the piezolever with a 10V, 10ms voltage pulse [1]. In that preliminary work, we used a vapor generator that had a cross sectional area of about 0.07 cm². Since the cross sectional area of the piezoresistive cantilever is only about 10-4 cm², most of the vapor emitted from the vapor generator was "wasted". We have conducted experiments with modified vapor delivery tubes with radii varying from 150 to 500 microns. We will present data taken with these narrow delivery tubes that enabled us to detect smaller vapor concentrations. This work was supported by the Department of Homeland Security. Oak Ridge National

Laboratory is managed by UT-Battelle, LLC, for the U. S. Dept. of Energy under contract DE-AC05-00OR22725. 1. L. A. Pinnaduwa, Gehl, D. L. Hedden, G. Muralidharan, T. Thundat, R. T. Lareau, T. Sulchek, L. Manning, B. Rogers, M. Jones, J. D. Adams, "A Microsensor for trinitrotoluene Vapour", Nature 425, 474 (2003).

10:15 AM *Y1.4

Cantilever Arrays for Multiplexed Biosensing. Arun Majumdar, Department of Mechanical Engineering, University of California, Berkeley, Berkeley, California.

An accurate, rapid, and quantitative method for analyzing variety of biomolecules, such as DNA and proteins, is necessary in many biomedical applications and could help address several scientific issues in molecular biology. Recent experiments have shown that when specific biological reactions occur on one surface of a microcantilever beam, the resulting changes in surface stress deflect the cantilever beam. To exploit this phenomenon for high-throughput label-free biomolecular analysis, we have developed a chip containing a 2-D array of cantilevers with a thin gold coating on one surface. Integration of microfluid cells on the chip allows for individual functionalization of each cantilever of the array, which is designed to respond specifically to a target analyte. An optical system to readout deflections of multiple cantilevers was also developed. Based on thermomechanical calibration, the repeatability of the cantilevers and their drift can be characterized. Subsequently, we have used these 2-D cantilever arrays for studying DNA hybridization, antigen-antibody binding, as well as whole bacteria detection. This talk will report the progress in our research towards making such 2-D cantilever arrays universal platform for biological and chemical sensing.

10:45 AM Y1.5

Silicon Cantilever Surfaces for Electronic Detection of DNA in Liquid. Joseph Tringe, Bradley Hart, Todd Sulchek and Brandon Weeks; Chemistry and Materials Science Directorate, Lawrence Livermore National Laboratory, Livermore, California.

Sensors based on functionalized silicon micromachined cantilevers can identify very small concentrations of pathogens and other biological agents in liquids. In order to ensure the robust operation of such a sensor in environments with reactive agents such as dithiothreitol (DTT) or high temperatures, it is advantageous to make use of a strong amide bond at the silicon cantilever surface. An additional requirement for some applications is that the sensor operates in an opaque medium, or in a confined volume that precludes the use of traditional "beam bounce" optical measurement methods. For these conditions, piezoresistive or piezoelectric cantilevers are suitable, but must be electrically isolated to ensure reasonable signal to noise ratios in liquid. In this study, silicon cantilevers are fabricated that have been electrically isolated with silicon dioxide or silicon nitride, then modified on one side with physically vapor-deposited silicon. The silicon surface is functionalized via a hydrosilylation reaction before being exposed to oligonucleotides that have been post-synthetically modified with a 3'-amino group. The sensitivity of the cantilevers to the hybridization of complementary single-stranded DNA is reported, together with an analysis of the temperature dependence of the system. Multiple cantilevers are measured in parallel, with some cantilevers being chemically functionalized or left bare as controls. Differential signals are obtained in steady state and as a function of externally-driven vibration frequency.

11:00 AM Y1.6

Polymeric Cantilever Arrays For Biosensing Applications. Montserrat Calleja^{1,2}, Javier Tamayo², Alicia Johansson¹, Peter Rasmussen¹, Laura Lechuga² and Anja Boisen¹; ¹MIC, Lyngby, Denmark; ²CNM-CSIC, Tres Cantos, Spain.

We report the design, fabrication and characterisation of arrays of SU-8 cantilevers for cantilever-based bio/chemical detection. Basically, a bio/chemical reaction at the cantilever surface will cause the cantilever to bend and this bending is detected either optically or by integrated sensors. We have recently reported an array of four cantilevers with integrated strain gauge readout in a microliquid system[1] and here we present cantilever-based biosensors for optical readout fabricated in a polymeric material. The use of a polymer as the component material for the cantilevers provides sensors with very high sensitivity due to a low Young's modulus. Furthermore, the developed fabrication technique shows to be most convenient for the integration of tens of cantilevers into a unique sensor chip for multiple detection and the cantilevers are relatively easy and fast to process. The fabrication process is based on spin coating of the photosensitive polymer and near-ultraviolet exposure. The method allows obtaining well-controlled and uniform mechanical properties of the cantilevers. Arrays of 9, 17 and 33 cantilevers of different dimensions were fabricated and characterised. Characterization of the present devices shows that they are suitable for performing both static and dynamic measurements for bio/chemical detection either in air or liquid

environments. The elastic constant of the cantilevers was measured and their dynamic response was studied. The sensors were used to monitor the immobilization process of cystamine on a gold-coated SU-8 cantilever. A reason for the growing interest in the development of a new kind of biological sensor based on the cantilever principle is the possibility of performing local, high resolution and label-free molecular recognition measurements on a portable device. Normally, micrometer-sized cantilevers fabricated in silicon and designed for atomic force microscopy (AFM) imaging are used in cantilever-based bio/chemical detection. Here we present a new fabrication process in which the sensors are realised in the epoxy-based polymer SU-8. The use of SU-8 polymer for different applications has experienced a rapid growth recently. It has especially become interesting for the fabrication of passive components such as microfluidic devices. Also, AFM probes[2] and cantilevers with an integrated gold resistor[3] have been fabricated in SU-8. To our knowledge, this is the first time that polymer cantilevers are designed and used for bio/chemical detection and also the study of the frequency response of the polymer cantilevers in air and liquid is new. [1]M. Calleja, P. Rasmussen, A. Johansson, A. Boisen "Smart Sensors, Actuators, and MEMS", Proc. SPIE, Vol. 5116, p.314-321, 2003 [2]G. Genolet, J. Brugger, M. Despont, U. Drechsler, P. Vettiger, N. F. Rooij, D. Anselmetti, Rev. Sci. Instrum. 70, 2398-401, 1999 [3]J. Thaysen, A. D. YalVinkaya, P. Vettiger, A. Menon, J. Phys. D: Appl. Phys. 35, 2698-2703, 2002

11:15 AM *Y1.7

Measurement of the changes in the mechanical, electrochemical and optical properties induced by the DNA/protein interaction. Eric Finot¹, Guillaume Legay¹, Jean Claude Weeber¹, Rita Meunier-Prest², Suzanne Raveau², Pierre Andreoletti³ and Mustapha Cherkaoui³, ¹Laboratoire de Physique, University of Burgundy, Dijon, France; ²Laboratoire de Synthèse et d'Electrosynthese Organometallique, University of Burgundy, Dijon, France; ³Laboratoire de Biologie Moléculaire et Cellulaire, University of Burgundy, Dijon, France.

The development of label-free detection methods is essential to the kinetic analysis of the protein interaction with DNA. Microfabrication technology can be used to produce microcantilever, interdigitated electrode and surface plasmon strips with an active surface area of (10 x 100) μm^2 . We compare the performances of such biosensors to gain information about the mechanical, electrochemical and optical properties of the biolayer. Cantilever deflection is used to measure the difference between the surface tensions of the cantilever which is proportional to the surface energy of the biolayer. Electrochemical method is used to investigate the charge transfer through the biolayer and therefore to have a chemical signature of the interaction. Surface plasmon enables to estimate the layer thickness as well as the layer optical index. The Atomic Force Microscopy and the Photon Scanning Tunnelling Microscopy are also used to interpret the sensor signal by locally characterizing the surface modification and the ligand/protein interaction in terms of surface rigidity, thickness, surface charge and optical constant. This study is applied to the analysis of binding of the (DNA)24ds with a protein (ARN polymerase T3)

SESSION Y2: Detection Systems I
Chairs: Bill Colston and Roberto Raiteri
Wednesday Afternoon, April 14, 2004
Room 3016 (Moscone West)

1:30 PM *Y2.1

Cantilever Based Sensing Devices with Integrated Readout. Anja Boisen, MIC, Technical University of Denmark, Lyngby, Denmark.

The majority of the cantilever based sensing experiments reported to date have been based on the optical leverage method used in atomic force microscopy. This method is very accurate but for many applications it would be an advantage to have a more compact read-out mechanism that requires less adjustment and alignment. A readout integrated on the cantilever would also greatly facilitate operation of arrays of cantilevers and work in liquid. In order to reduce background noise such as thermal drift and turbulence around the cantilever, a reference cantilever is crucial. For monitoring changes in surface stress we have developed various types of micromachined silicon based cantilever devices with integrated piezoresistive read-out and built-in reference cantilevers. The cantilever dimensions as well as the placement and the doping of the piezoresistors have been optimised for surface stress detection. For plug-in experiments a two-cantilever chip has been developed and applied for gas measurements as well as for detection of biomolecules in liquid (1). Arrays of up to 10 cantilevers have been integrated in micro-channels offering a method to develop compact biosensors with a simple read-out scheme (2). Recently, we have realised cantilever sensors in the polymer SU-8, which is a negative resist with excellent

mechanical, thermal and chemical properties. The use of polymer makes the fabrication process simple, cheap, fast and flexible. Thus, it will be possible to realise a large variety of sensor designs and sensor types in a short period of time. Moreover, the polymer technology opens up for new ways of detecting cantilever bending, which might increase the sensitivity. Large arrays of SU-8 cantilevers with varying dimensions have been used to study the mechanical performance of polymer cantilevers (3). The elastic constant of the cantilevers was measured, and their dynamic response was investigated.

Characterization of the devices shows that they are suitable for both static and dynamic measurements for biochemical detection in either air or liquid environments. Furthermore, complete devices consisting of polymer microchannels and polymer cantilevers with integrated metallic strain gauges have been realized (4). For mass detection we have developed silicon-based nanocantilevers with integrated capacitive read-out and electrostatic actuations. Due to problems with high stray capacitances the sensor is monolithically integrated with a CMOS chip for on-chip amplification of the signal (5). In order to investigate the bottom-up approach we are currently pursuing ways of fabricating carbon nanotube based resonators. (1) R. Marie et al, Ultramicroscopy 91, pp 29-36 (2002) (2) J. Thaysen et al, Proceedings of MEMS, pp. 401-404 (Interlaken 2001) (3) M. Calleja et al, Sensor Letters, vol. 1. No.1 pp. 1-5 2003 (4) M. Calleja et al, Proceedings of μTAS 2003, pp. 207-210 (2003) (5) Z. Davis et al, Sensors and Actuators A, 105 (3), pp. 311-319 (2003)

2:00 PM Y2.2

MOSFET Integrated Microcantilevers for Novel Electronic Detection of "On-Chip" Molecular Interactions.

Vinayak P Dravid¹ and Gajendra S Shekhawat²; ¹Material Science, Northwestern University, Evanston, Illinois; ²Institute for Nanotechnology, Northwestern University, Evanston, Illinois.

We have developed a novel bio-chem sensor system based on silicon chip technology for electronic detection of biomolecules. The sensing element is an integrated MOSFET transistor, placed at the high stress region of the micro cantilever. The reverse side of the micro cantilever is functionalized with appropriate sensing layer. As selective binding occurs during bio-chem exposure, the well-known bending of the cantilever leads naturally to significant, measurable and reproducible change in the drain current, providing a novel signal transduction mechanism. Such piezo-MOS in the present configuration provide significant advantage over conventional optical detection, including sensitivity, liquid-gas cell operation, ease of integration, among others. Our initial result indicates clear high sensitivity of MOS detection, down to less than 2 nm cantilever deflection through external actuator. The location of the MOS chip is precisely calculated after numerous modelling and simulation. MOSFET platform not only improves the sensitivity, but also has almost negligible noise figure (large signal to noise ratio), ease of integration with CMOS and RF components. By the use of stress sensitivity MOSFETs as active loads, the size of the transistor is considerably reduced when compared to diffused piezoresistors. The presentation will cover device architecture and proof-of-concept sensing examples of biological and gas-chemical analytes.

2:15 PM Y2.3

Cantilever array with integrated readout made in SU-8 and integrated in a micro liquid system. Peter Andreas Rasmussen, Montserrat Calleja, Alicia Johansson, Rodolphe Marie and Anja Boisen; Mikroelektronik Centret (MIC), Technical University of Denmark, Lyngby, Denmark.

We report on a cantilever array integrated in a micro fluidic channel, both of which are made of the photosensitive polymer SU-8. We have previously developed a cantilever platform in silicon[1] and the SU-8 chip represents a new cantilever platform with a much faster fabrication cycle and the possibility to make on-chip fluid channels and encapsulation that will reduce demands on the subsequent packaging of the chip. The readout from the cantilever array is made with gold strain gauges that are encapsulated in the SU-8 cantilevers. The estimated performance of this sensor is at least comparable to that of present cantilever sensors with polysilicon piezoresistors, since the low gauge factor of the metal is compensated by the lower stiffness of the cantilever, and because the noise in the gold resistors is lower than in the polysilicon resistors. The SU-8 device is glued together with SU-8 and packaged in a PDMS/PMMA case tailored by laser machining. Measurements of the noise and the sensitivity have been performed[2]. We are presently working on making the electrical interconnection between the sensor and the readout circuitry via flip chip bonding, and on the testing of the device in a micro liquid handling setup. [1] J. Thaysen, R. Marie and A. Boisen, Cantilever-based bio-chemical sensor integrated in a microfluidic handling system, MEMS 2001, Proceedings pp.401-404. [2] M. Calleja, P. Rasmussen, A. Johansson and A. Boisen, Polymeric mechanical sensors with strain gauge readout in a microfluidic system, microTAS 2003, Proceedings pp. 207-210.

2:30 PM *Y2.4

Photoacoustic Chemical Sensor Using Microelectromechanical Structures. Paul M. Pellegrino¹, Ronald G. Polcawich² and Samara L. Firebaugh³; ¹Attn: AMSRL-SE-EO, U.S. Army Research Laboratory, Adelphi, Maryland; ²Attn: AMSRL-SE-RL, U.S. Army Research Laboratory, Adelphi, Maryland; ³Electrical Engineering, U.S. Naval Academy, Annapolis, Maryland.

Photoacoustic spectroscopy is a useful monitoring technique that is well suited for trace gas detection. The technique also possesses favorable detection characteristics when the system dimensions are scaled to a micro-system design. The objective of present work is to incorporate two strengths of the Army Research Laboratory (ARL), piezoelectric microelectromechanical systems (MEMS) and chemical and biological sensing into a monolithic MEMS photoacoustic trace gas sensor. Examination of a $\frac{1}{4}$ scale macro-cell indicates a pathway to incorporate a photoacoustic resonance structure in a micro-mechanical platform. In order to maintain sensitivity and characteristics of typical photoacoustic cell, work has centered on transmission-line modeling of the resonant microstructures. Initial models examined a typical H-style open tube resonance structure including acoustic buffering zones. Results from this modeling will be directly incorporated into cell design and will include all pertinent parameters. MEMS work is centered on improving design and fabrication of a lead zirconate titanate (PZT) microphone subsystem to be incorporated in the full photoacoustic device. Second generation microphones have been designed, fabricated and acoustically tested. Presently, the piezoelectric microphone performance has revealed the possibility of using a PZT microphone as the passive acoustic detection mechanism of a photoacoustic resonant cavity. MEMS acoustic cavity designs have investigated multi-layer stacking arrangements using several low-temperature bonding schemes, which allow monolithic integration. Results will be presented describing the miniature photoacoustic cell capabilities, current MEMS microphone performance and initial cell designs based on transmission-line modeling.

3:30 PM *Y2.5

Materials and Processing Challenges Related to the Fabrication of a MEMS Micro Gas Chromatograph.

Edward T Zellers^{1,2,3}, Judy Zhong^{1,2}, Michael C Oborny^{1,2}, William H Steinecker^{1,3}, Jamie Nichols^{1,3}, Michael P Rowe^{1,3}, Adam J Matzger^{1,3}, Gordon Lambertus^{1,3}, Andrea Elstro^{1,3}, Joshua Whiting^{1,3}, Richard D Sacks^{1,3}, Joseph A Potkay^{1,4}, Masoud Agah^{1,4}, Kensall D Wise^{1,4}, Helena K L Chan^{1,4}, Stella Pang^{1,4}, Luciana DaSilva^{1,5}, Massoud Kaviani^{1,5}, Jaeh Kim^{1,6}, Cagliyan Kurdak^{1,6}, Yang Lu^{1,8}, Dean Aslam^{1,8}, Jin Zheng^{1,7} and Paul Bergstrom^{1,7}; ¹Engineering Research Center for Wireless Integrated Microsystems (WIMS), University of Michigan, Ann Arbor, Michigan; ²Environmental Health Sciences, University of Michigan, Ann Arbor, Michigan; ³Chemistry, University of Michigan, Ann Arbor, Michigan; ⁴Electrical Engineering and Computer Sciences, University of Michigan, Ann Arbor, Michigan; ⁵Mechanical Engineering, University of Michigan, Ann Arbor, Michigan; ⁶Physics, University of Michigan, Ann Arbor, Michigan; ⁷Electrical and Computer Engineering, Michigan Technological University, Houghton, Michigan; ⁸Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan.

The fabrication and testing of a MEMS micro gas chromatograph (μ GC) subsystem are described, with an emphasis on the novel materials and processes employed. Key components of the μ GC include a calibration-vapor source, preconcentrator/focuser (μ PCF), separation column, and an integrated array of four chemiresistors. All devices and structures have been fabricated from Si using MEMS technology. Interconnection through a μ -fluidic substrate and capillary tubing has permitted repeated analyses of multi-vapor mixtures with a duty cycle of a few minutes. The calibration-vapor source comprises a Si/glass/Si diffusion cell with a porous-Si (PS) reservoir that can passively generate a nearly constant stream of vapor for extended time periods. PS reservoirs made by anodizing p-type Si have low-dispersity pre-patterned macropores $>400 \mu\text{m}$ deep with diameters of 2-6 μm . Through-wafer processing of such PS has recently been achieved using electrolytic liquid backside contact. Initial versions of the 3-stage μ PCF employed commercial granular adsorbents packed between high-aspect-ratio deep-reactive-ion-etched (DRIE) Si heater structures. We are now exploring alternative adsorbents with tailored porosity, surface area, and functionality. For example, multi-walled carbon nanotubes (MWNT) have been grown on heater-structure surfaces using FeNO_3 catalyst to thicknesses of $>50 \mu\text{m}$. Separate testing shows excellent adsorption/desorption properties for moderately volatile organic vapors. ZnO4-carboxylate metal-organic frameworks (MOFs) with exceptionally high surface areas and affinities for light organics have also been grown on micromachined-Si heater structures, however further improvements are needed in film uniformity. Separation columns comprising square-spiral DRIE-Si channels (3m long, 3.3 x 3.3cm footprint) have

been made and treated with poly-dimethylsiloxane and poly-trifluoropropylsiloxane stationary phases deposited dynamically from solution and thermally cross-linked. Columns providing up to 11,000 theoretical plates have been made. The integrated chemiresistor sensor array uses Au-thiolate nanoclusters as chemically sensitive interface layers and provides characteristic response patterns and calculated LODs as low as 20 ppt for 1-L preconcentrated vapor samples eluting from a bench-scale GC. Nanoclusters are made by a new single-phase synthesis that avoids residual contamination by the phase transfer catalyst used in the standard (Brust) method. Nanocluster thin films show non-linear I-V characteristics at room temperature (100-nm electrode gaps) and noise that varies as $1/f$ and increases dramatically upon vapor exposure. Micro TE coolers for the sensor array are being made from BiTe and SbTe alloys. Patterned thick films have been made by a low-temperature co-evaporation process, and initial working devices have been tested. These and other highlights of the Michigan μ GC development effort will be presented.

4:00 PM Y2.6

Development of MEMS-based Micronozzles for Gas Separation. Sheng Li¹, Carl B. Freidhoff², Robert M. Young² and Reza Ghodssi¹; ¹Electrical and computer engineering, Univ. of Maryland, College Park, Maryland; ²Northrop Grumman Electronic Systems Inc., Baltimore, Maryland.

Advances in micro-electro-mechanical systems (MEMS) technologies enable the development of devices that can sense and analyze minute amounts of fluid in the nano or pico liter range. MEMS-based micronozzles can act as real-time preconcentrators for miniaturized chemical sensors to enhance their sensitivity. We report the designed and fabrication of linear contoured micronozzles using a low temperature wafer-level adhesive bonding technique. A selection of wafer bonding tests with SU-8 as the intermediate bonding material is performed to investigate the influence of different parameters on the bond quality. For comparison, micronozzles with similar dimensions are fabricated using silicon-glass anodic bonding in conjunction with deep reactive ion etching (DRIE). The fabricated nozzles are packaged to interface with a gas flow test setup that is developed to measure gas flow rates and pressure distributions of gas flows in the micronozzles. Leakage test is performed to characterize the seal of packaging and wafer bonding, and similar behaviors are observed in both micronozzles fabricated with the two different bonding techniques. The micro-to-macro interconnections are experimentally tested to withstand up to 300 kPa of gas pressure without leaking. Gas flow test results show that sonic gas flows are generated in the micronozzles. Mass flow efficiency, which is the ratio of actual mass flow to the theoretical mass flow, is measured to vary from 0.43 at a Reynolds number of 62 to 0.81 at a Reynolds number of 262. These measurements and developed techniques provide a basis for the design and fabrication of gas separation devices that are envisioned for miniaturized chemical sensors.

4:15 PM *Y2.7

Development of a Miniature Ion Mobility Spectrometer-based System for Contraband Detection. Kent Bryant Pfeifer¹, Steven Barney Rohde³ and Kenneth Allen Peterson²; ¹Microsensor Science and Technology, Sandia National Laboratories, Albuquerque, New Mexico; ²Thin Film, Vacuum, & Packaging, Sandia National Laboratories, Albuquerque, New Mexico; ³Integrated Microsystems, Sandia National Laboratories, Albuquerque, New Mexico.

Ion Mobility Spectroscopy (IMS) is an attractive technology for production of miniaturized gas analysis hardware for several reasons. These include the ability to operate at atmospheric pressure, eliminating the need for a vacuum interface, the ability to determine species of ions in the sample, and extremely low detection limits. Physical effects that reduce the resolution of the system limit miniaturization of IMS technology. We have developed a miniaturized IMS system that fits on a 6 cm by 10 cm circuit board, weighs less than 80 gm, and has adequate resolution for detection of the principle components of most major military and industrial explosives. Current IMS designs require complex assemblies of electrodes and insulators coupled with large series arrays of resistors to produce the uniform electric fields desired in the IMS drift tube. This results in systems that are both labor intensive and expensive to produce. We have demonstrated a miniaturized conventional design and have migrated this design into a simple-to-assemble and inexpensive IMS drift tube constructed from rolled, low-temperature co-fired ceramic (LTCC) using a commercial materials system. This approach eliminates as many as 150 individual parts from our conventional design while producing a more chemically inert internal surface. The hermeticity and ruggedness are improved by the elimination of more than 100 drift tube joints. Also eliminated are 200 board joints associated with resistors and approximately 51 welds to individual drift tube electrodes since the potential gradient is produced using on-tube, thick-film resistors. Internal components of the IMS such as the control grids are constructed using LIGA techniques while the ion

apertures and ion target are photodefined and etched from a sheet of kovar plated with nickel. These parts are then inserted into the tube and electrical contact is made by press fitting the parts via integral spring structures and then soldering the structures to make them appropriately rigid. Applications requiring tube temperatures above ambient are accommodated by inclusion of an integral heater into the rolled tube structure. Thus, we have produced an IMS tube with the promise of low-cost production for application in the detection of contraband materials important to law enforcement, first-responders, security screening, and many other applications of interest. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

SESSION Y3: Detection Materials
Chairs: Ida Lee and Arun Majumdar
Thursday Morning, April 15, 2004
Room 3016 (Moscone West)

8:30 AM *Y3.1

An Electronic Nose from Arrays of Polymer Composite Vapor Sensors. Nathan S Lewis, ¹CCE, California Institute of Technology, Pasadena, California; ²CCE, California Institute of Technology, Pasadena, California.

A method is described for generating a variety of chemically diverse, broadly responsive, low power vapor sensors. A key to our ability to fabricate chemically diverse sensing elements is the preparation of processable, air stable films of electrically conducting organic polymers. An array of such sensing elements produces a chemically reversible, diagnostic pattern of electrical resistance changes upon exposure to different odorants. Such conducting polymer elements are simply prepared and are readily modified chemically to respond to a broad range of analytes. In addition, these sensors yield a fairly rapid, low power, dc electrical signal in response to the vapor of interest, and their signals are readily integrated with software or hardware-based neural networks for purposes of analyte identification. Principle component analysis has demonstrated that such sensors can identify and quantify different airborne organic solvents, and can yield information on the components of gas mixtures.

9:00 AM *Y3.2

Noble Metal-Polymer Nanocomposites as Versatile Materials for Vibrational Spectroscopy, Sensing, and Microfluidic Separations. Michael J. Sepaniak, Maggie Connatser, Kathleen Giesfeldt, Lance Riddle, Dutta Pampa and Marco De Jesus; University of Tennessee, Knoxville, Tennessee.

Nanocomposite materials are formed by physical vapor deposition of noble metals onto pliable poly-(dimethylsiloxane) (PDMS) films. When silver or gold is deposited at an appropriate rate and thickness, subsurface morphologies are created in the phase-separated layer of the polymer that exhibit unique optical properties including surface enhanced Raman scattering (SERS) activity. The characterization of the nanocomposites by microscopy, XPS, and DC conductivity will be reported. Among the unique fundamental properties of these pliable materials is an ability to alter and optimize plasmon resonances and SERS responses by simple physical manipulation. Since SERS spectra are rich with vibrational information, high selectivity and structural elucidation are possible in sensing applications. Moreover, the PDMS functions as a solid phase extractor to concentrate analytes while protecting the metal surface from degradation in the sample matrix. Finally, the nanocomposites can be molded into practical devices such as titer plates and, using soft photolithographic techniques, microfluidic separation platforms. Integrated devices that combine electrokinetic separation capability with vibrational spectral acquisition on a single platform will be demonstrated.

9:30 AM Y3.3

Organic TFT Gas Sensors for Electronic Nose Applications. Frank Liao, Christopher Chen and Vivek Subramanian; Electrical Engineering Computer Sciences, University of California, Berkeley, Berkeley, California.

Organic TFTs are promising candidates for use in electronic nose applications because they exhibit sensitivity to a broad range of chemicals and can be fabricated using solution-based methods. Upon exposure to an analyte, organic TFTs show shifts in multiple device parameters including transconductance, threshold voltage, and leakage and drive current. Using additive solution based processing techniques such as inkjetting, multiple active materials may be incorporated into an array of sensing devices to form an e-nose. This is attractive because it reduces the likelihood of false positive readings and enables signature-specific detection of multiple targets. To this

end, we have demonstrated the concept of an electronic nose using multiple solution-processed organic devices integrated into a single testbed. We report on the use of this testbed to examine different solvent systems and have observed different responses for each type of active material. These results indicate that organic devices may have the capacity to be used as in sensor arrays for electronic nose applications. We also report on previously unexplored characteristics of organic TFT sensors including sensor consistency and transient dynamics during prolonged exposure periods. Finally, we also present some work on use of these devices to detect food spoilage.

9:45 AM Y3.4

Estimation of Chemical and Physical Characteristics of Analyte Vapors Through Analysis of the Response Data of Arrays of Polymer-Carbon Black Composite Vapor Detectors. Brian C Sisk and Nathan S Lewis; Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California.

Analysis of the signals produced by a collection of organic polymer-carbon black composite vapor detectors has been performed to assess the ability to estimate various chemical and physical properties of analyte vapors based on information contained in the response patterns of the detector array. A diverse array of composite chemiresistive vapor detectors was exposed to a series of 75 test analytes that had been selected from among five different chemical classes: alcohols, halogenated hydrocarbons, aromatics, unsubstituted hydrocarbons, and esters. The algorithmic task of interest was to use the resulting array of response data to assign one of the five chemical class labels to a test analyte, despite having left that analyte out of the model used to generate the class labels. Algorithms evaluated for this purpose included principal components analysis (PCA) and k-nearest-neighbor (k-NN) analysis employing either Euclidean or Mahalanobis distance calculations. Each data cluster that was produced by replicate exposures to an individual analyte was well resolved from all of the other 74 analyte clusters. Furthermore, with the exception of the halide cluster, the analyte response clusters could be robustly grouped into supersets such that each of the five individual chemical classes was well separated from every other class of analytes in principal component space. Accordingly, using either of the k-nearest-neighbor algorithms, in excess of 85% of the non-halide test analyte exposures were correctly assigned to their chemical classes, and halides were only routinely confused with aromatics or esters but not with alcohols or hydrocarbons. The detector array response data also was found to contain semi-quantitative information regarding physicochemical properties of the members of the test analyte series, such as the degree of unsaturation of the carbon chain, the dipole moment, the molecular weight, the number of halogen atoms, and type of aromatic ring in the test analytes. The performance in these types of tasks is relevant for applications of a semi-selective array of vapor detectors in situations when no prior knowledge of the analyte identity is available and when there is no assurance that the test analyte will have been contained in the training set database produced by a compiling a library of responses from the detector array.

10:30 AM *Y3.5

Self-assembling, self-orienting porous silicon photonic crystal "Smart Dust" sensors. Mike Sailor, Dept of Chemistry, University of California-San Diego, La Jolla, California.

Small particles of microporous Si are used to detect chemicals by measurement of their optical reflectivity spectrum. Each particle contains in its nanostructure the necessary components to allow self-assembly, spectroscopic identification, and chemical sensing. The particles are generated by electrochemically etching discrete porous one-dimensional dielectric stack (rugate) mirrors into silicon. The complex multilayered structure produces a distinctive reflectivity spectrum that serves as a robust code, allowing positive identification and discrimination of many different types of particles. The intensity and wavelength of reflected light is determined in part by the refractive index of the porous nanostructure, which is modified by adsorption of vapors or by specific chemical reactions within the chemically modified porous Si matrix. Sensing is accomplished when liquid or vapor infuses into the porous mirrors, inducing predictable shifts in the optical spectra. Chemically asymmetric particles are also described, which can spontaneously align at an organic liquid/water interface. Thus the micron-sized photonic crystals of porous Si can self-assemble, orient, and sense their local environment. Application of these materials for sensing of pollutants and toxic chemicals will be described.

11:00 AM *Y3.6

Ultrasensitive and Multiplexed Protein Detection with Nanoparticle-based Biobarcode. Chad A. Mirkin, Northwestern University, Evanston, Illinois.

An ultrasensitive method for detecting protein analytes has been

developed. The system relies on magnetic microparticle probes with antibodies that specifically bind a target of interest [prostate specific antigen (PSA) in this case] and nanoparticle probes that are encoded with DNA that is unique to the protein target of interest and antibodies that can sandwich the target captured by the microparticle probes. Magnetic separation of the complexed probes and target followed by dehybridization of the oligonucleotides on the nanoparticle probe surface allows one to determine the presence of the target protein by identifying the oligonucleotide sequence released from the nanoparticle probe. Because the nanoparticle probe carries with it a large number of oligonucleotides per protein binding event, there is substantial amplification and one can detect PSA at 30 attomolar concentration. Alternatively, one can do polymerase chain reaction on the oligonucleotide barcodes and boost the sensitivity to 3 attomolar. Comparable clinically accepted conventional assays for detecting the same target have sensitivity limits of ~ 3 pM, 6 orders of magnitude less sensitive than what is observed with this method.

11:30 AM Y3.7

Single Enzyme Nanoparticles: A New Nanostructure for Enzyme Stabilization. Jay W Grate and Jungbae Kim; Pacific Northwest National Laboratory, Richland, Washington.

Nanoparticles and nanoparticle-based materials are attracting great interest for their unique properties and potential for application in diverse areas. We have developed a new nanostructure containing an enzyme within a hybrid organic/inorganic polymer network with sufficient porosity to allow substrates to diffuse to the active site.⁽¹⁾ The synthetic procedure, entailing enzyme modification and two orthogonal polymerization steps yields nanoparticles containing a single enzyme, which can be observed by transmission electron microscopy. In experiments with chymotrypsin, incorporation into the nanostructure dramatically increased the enzymatic stability. Furthermore, the nanoscale structure around the enzyme is sufficiently thin that it does not impose a significant mass transfer limitation on the substrate. Because these nanoparticles remain soluble or suspended in solutions, they can be processed into a variety of forms. They can be used in solution, cast into thin films, or adsorbed within larger mesoporous structures. Given enzymatic specificity and the great diversity reactions catalyzed by enzymes, coupled with the flexibility in the use of single enzyme nanoparticles, these new nanostructures are very promising for many applications including biosensors, bioreactors, microfluidic devices, decontamination, remediation, antifouling, and protective layers. (1) Jungbae Kim and Jay W. Grate, "Single-Enzyme Nanoparticles Armored by a Nanometer-scale Organic/Inorganic Network", Nano Letters, 3 (2003) 1219-1222.

11:45 AM Y3.8

Multiplex Biodetection Using Solution Arrays Based on Encoded Nanowire Particles. George Dougherty¹, Frank Chuang², Satinderpal Pannu¹, Klint Rose¹, Michael Sha³ and Sharron Penn³; ¹Center for Micro and Nano Technology, Lawrence Livermore National Laboratory, Livermore, California; ²UC Davis Cancer Center, Davis, California; ³Nanoplex Technologies, Inc., Mountain View, California.

We are developing a new platform for performing multiplex biodetection assays with the goal of providing flexibility, user reconfigurability, ease of use, portability, and low cost. The platform uses the solution array concept of a suspension of different uniquely identifiable particle types, each type independently functionalized for affinity to a unique biochemical target that may be present in the sample. The particles employed are Nanobarcode(R), metallic nanowire particles a few microns in length and 50-250 nm in diameter. Synthesized by sequential electrodeposition of different metals within porous anodic alumina templates, these unique particles are encoded with patterns of alternating high and low reflectivity, resulting in stripe patterns similar to those of supermarket barcodes. In contrast to earlier implementations of the solution array concept, such as those using fluorescent beads, the use of these particles allows the assay results to be read using standard microscope optics and image processing software, without the need for flow cytometry. In addition, the metallic properties of the particles make it possible to manipulate them within automated fluidic sample processing systems using magnetic and other forces. After an overview of the Nanobarcode technology, we present results demonstrating the effective biochemical functionalization of the Nanobarcode particles with self-assembled monolayers for surface property control, and antibodies against several biochemical targets. We demonstrate the use of these novel functionalized particles to carry out the specific detection of biochemical targets within a multiplex solution assay.

SESSION Y4: Detection Systems II
Chairs: Jay Grate and Mike Sepaniac

1:30 PM *Y4.1

Chemical Sensing with a MEMS-Based Correlation Radiometer. Michael B Sinclair and Kent B Pfeifer; Sandia National Laboratories, Albuquerque, New Mexico.

We describe the use of a MEMS-based correlation radiometer for remote chemical sensing. At the heart of the radiometer is a MEMS programmable diffraction grating called the Polychromator. The Polychromator contains an array of 1024 electrostatically actuated reflective beams that are 10 microns wide by 1 cm long, and have a vertical travel of approximately 2 - 4 microns. The deflection profile of the array can be programmed to tune the spectral transfer function of the radiometer. In this manner, the Polychromator serves to replace the reference cell of conventional correlation radiometry. Switching of the radiometer's spectral transfer function can be accomplished in as little as 2 ms. Advantages of this approach to correlation radiometry include the ability to detect multiple chemical species with a compact instrument, the ability to optimize the reference spectra using multivariate techniques to eliminate chemical interferences, and the ability to produce reference spectra for hazardous and transient species. The operation of the radiometer will be discussed and initial results for chemical sensing will be presented. Portions of this work were performed at Sandia National Laboratories. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000

2:00 PM Y4.2

High-Index Glass Microspheres as Highly Sensitive Collectors for Remote Sensing Applications. Anthony Esposito, Chad Talley, Christopher Hollars, Thomas Huser and Charles Stevens; Chemistry & Materials Science, Lawrence Livermore National Laboratory, Livermore, California.

Sensitive remote chemical detection is of significant interest for national security purposes. We have investigated the use of high-refractive-index glass microspheres as collection elements for such remote sensing applications. Optical characterization of these microspheres indicates that their light focusing and gathering properties are comparable to those of high numerical aperture microscope objectives, but are more applicable to use in the field due to the intrinsic omnidirectional retroreflection of the spherical lens. We have obtained fluorescence spectra from dilute dye solutions (10^{-9} to 10^{-11} M), and show single-molecule sensitivity when the microspheres are employed as the light focusing and collecting elements. Chemical functionalization of the microsphere surfaces with fluorescent labels or metal nanoparticles for surface-enhanced Raman spectroscopy should enhance the collected signal, and comparison of the Raman signal to a spectral database will allow for identification of specific compounds in the collection volume of the microsphere.*
*This work was performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under contract number W-7405-Eng-48.

2:15 PM Y4.3

Multi-Wavelength Excitation and Fluorescence From Single Bioaerosol Particles. Alan L. Huston, Vasanthi Sivaprakasam, Jay Eversole and Cathy Scott; Optical Sciences Division, Naval Research Laboratory, Washington, District of Columbia.

A new, fluorescence-based bioaerosol sensor technique is described that uses 266 nm and 355 nm laser pulses from diode-pumped Nd:YAG lasers to sequentially excite single, micron-sized aerosol particles. A number of bioaerosol detection systems have been developed that utilize 266 nm excitation to provide an indication of whether an aerosol particle contains biological material or not. This determination is based on the observation of laser-induced fluorescence in the 340 nm range from aromatic amino acids that are present in all biological organisms. The problem with this approach is that there are a number of non-biological interferences that also fluoresce when excited at 266 nm. As a result, the rate of false alarms is too high for practical implementation. Systems have also been described that utilize 355 nm UV excitation to excite bioaerosol fluorescence. In this case, the bio-fluorescence is due to chemicals such as NADH and flavinoid compounds that are metabolic products of living cells. The system that we have developed utilizes a continuous wave, 800 nm diode laser to detect the presence of an aerosol particle in an optical/aerosol chamber. The scattered light amplitude is used to estimate the size of the particle as well as to trigger the firing of two Nd:YAG lasers. The first laser is used to generate a 355 nm laser pulse that excites the detected aerosol particle. Fluorescence generated by the 355 nm pulse is detected in two broad wavelength bands centered at 450 nm and 550 nm. A second laser is used to generate a pulse at 266 nm. This pulse is delayed by 1 microsecond

with respect to the 355 nm pulse and excites the same aerosol particle. The fluorescence excited by the 266 nm pulse is detected in a broad band centered at 350 nm and in the 450 nm and 550 nm bands that were used to detect the 355 nm excited fluorescence. The photomultiplier tube that is used to detect the 350 nm band is gated to respond only to the fluorescence generated by the 266 nm pulse, otherwise, intense scattered light from the 355 nm excitation pulse would damage the detector. Six distinct pieces of information are acquired for every aerosol particle that enters the chamber, including particle size and five fluorescence signals. The light scattering and fluorescence characteristics of several aerosol particle standards, proteins, bacterial and fungal species were investigated and analyzed using the technique of Principle Component Analysis (PCA). The PCA results obtained to date indicate promise in helping to classify different types of aerosol particles. Fluorescence signals from fungal spores, bacteria and proteins are clustered into distinct regions in the PCA plots. Further work is continuing to determine the degree of discrimination that may be possible using the two-wavelength fluorescence excitation approach.

2:30 PM *Y4.4

Carbon nanofiber interface to molecular-scale processes in viable cells. Michael L Simpson^{1,2,3}, Timothy E McKnight^{1,3} and Anatoli V Melchko^{1,2,3}, ¹Molecular-Scale Engineering, Oak Ridge National Lab, Oak Ridge, Tennessee; ²Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee; ³Center for Environmental Biotechnology, University of Tennessee, Knoxville, Tennessee.

One of the key challenges for advanced bio-detection strategies and post-genomic biology in general is the detailed understanding and characterization of the molecular-scale components and their interactions that produce complex cell functionality. As they reside at the same size scale as the biomolecular machines of cells, engineered nanoscale devices may provide the means to construct tools for monitoring and manipulating cellular processes. Other investigators have highlighted this interface of nanotechnology and biotechnology such as the use of non-bleaching fluorescent nanocrystals in place of dyes, but usually as a means of monitoring rather than controlling cellular processes. Pulled glass capillaries with nanoscale tips have been implemented for cellular and subcellular electrophysiological monitoring and for microinjection of membrane-impermeable molecules (e.g. proteins, DNA), but these devices require manipulating cells one at a time using individual elements observed under a microscope, and thus provide only a serial interface to cells. Parallel embodiments of these devices have been fabricated using silicon microfabrication methods, but as with all micromachining techniques there are limitations to the ultimate size scale and density of features (tip radii and spacing of the silicon needles) and a limited choice of substrate materials (i.e. not easily fabricated on transparent substrates convenient for cell culture). In contrast, carbon nanotubes (CNTs) and related nanostructures avoid these limitations and provide the means to construct addressable functional nanoscale devices including chemically specific AFM probes, electrochemical probes, and electromechanical manipulators. Within this family of structures, carbon nanofibers are uniquely suited for the construction of intracellular devices because of the ability to exquisitely control their synthesis. Deterministic arrays of closely-spaced (pitch = 1 μ m) vertically aligned carbon nanofibers (VACNFs) may be grown on a wide variety of substrates (including quartz and glass slides) with wide bases that provide mechanical strength while still generating a small diameter tip (= 5 nm tip radius) appropriate for insertion directly into cells. We present a critical enabling step toward the hardware tools needed for the coupling of molecular-scale cell function theory and modeling with experiment by demonstrating the controlled synthesis, directed assembly, and then functional integration of VACNF elements within viable cells. The viability of the cells after VACNF insertion is demonstrated by the long-term expression of a constitutively-expressed green fluorescent protein (GFP) gene carried on nanofiber-borne plasmid molecules.

3:30 PM *Y4.5

The Autonomous Pathogen Detection System. John Dzenitis, Benjamin Hindson, Mary McBride, Anthony Makarewicz, Bruce Henderer, Ujwal Sathyam, Shanavaz Nasarabadi, Kodumudi Venkateswaran, Dora Gutierrez, Sally Smith, Tom Metz and Billy Colston; Lawrence Livermore Natl. Laboratory, Livermore, California.

An Autonomous Pathogen Detection System (APDS) unit is an automated, podium-sized system that monitors the air for all three biological threat agents (bacteria, viruses, and toxins). The system has been developed by Lawrence Livermore National Laboratory (LLNL) to protect people in critical or high-traffic facilities and at special events. The system performs continuous aerosol collection, sample preparation, and multiplexed biological tests of two types: The primary screen is a multiplexed immunoassay (protein-based) using a Luminex flow cytometry platform. More than ten agents are assayed

at once. The confirmatory test is polymerase chain reaction (PCR, DNA-based) using a real-time TaqMan assay. The system can operate completely autonomously processing a multiplexed sample every hour for one week, at which point only simple reagent refills are required. The APDS detected releases of live plague and anthrax in a chamber test at Dugway Proving Grounds, and has been field-tested in a public airport and subway. Recent laboratory and field tests will be presented. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

4:00 PM Y4.6

Renewable Surface Immunoassay for Rapid Detection of Botulinum Toxin. Cynthia J. Bruckner-Lea¹, Brian Dockendorff¹, Jay W Grate¹, Marvin Warner¹, Norman C Anheier², Michael Feldhaus³ and James D Marks⁴; ¹Chemical Sciences Division, Pacific Northwest National Laboratory, Richland, Washington; ²Electronic Systems Group, Pacific Northwest National Laboratory, Richland, Washington; ³Biological Sciences Division, Pacific Northwest National Laboratory, Richland, Washington; ⁴UCSF, San Francisco, California.

Rapid detection of botulinum toxin was demonstrated using a renewable surface immunoassay, which included on-column detection of a fluorescently labeled secondary antibody in a sandwich immunoassay. Yeast display and directed molecular evolution were used to create a high affinity antibody to the botulinum toxin heavy chain receptor binding domain, AR1. This antibody was modified with biotin and coupled to streptavidin-coated Sepharose 4B beads. A secondary antibody that binds to the botulinum heavy chain 3D12 was labeled with a fluorescent dye, Alexa 633. A rotating rod renewable surface microcolumn system was used to automatically capture the Sepharose beads to form a microliter-sized column, perfuse the beads with sample and wash solutions, deliver fluorescently labeled secondary antibody, and monitor the on-column fluorescence. After analysis, the beads were automatically flushed from the system. Detection was accomplished in less than 5 minutes, with a total processing time, including column regeneration, of about 10 minutes. On-column detection was at least 10-fold more sensitive and much faster than flow cytometry analysis using the same reagents. These results demonstrate how a combination of high affinity reagents and a sensor configuration enabling rapid mass transport can be combined for rapid, sensitive biodetection.

4:15 PM Y4.7

Micro electrical impedance spectroscopy of healthy and cancerous cells. Luis Jose Cruz-Rivera, Kumar Ravula and A. Bruno Frazier; ECE, Georgia Institute of Technology, Atlanta, Georgia.

Empirical data has shown that dielectric properties of cells depend on the cell type and physiological status. The specific impedance of mammalian cells, describe their complexities and membrane barrier functions. While these have been shown to respond to cell pathologic and physiologic changes, of particular interest is the frequency response and characteristic dispersions measured by micro-electrical impedance spectroscopy (μ -EIS) and the differentiation of the electrical properties between normal and cancerous cells. This report describes the fabrication and characterization of a μ -EIS microsystem and its use to characterize the cellular impedance differences in a microfluidic system to study the electrical behavior of cells. The elucidation of the impedance characteristics and related phenomena provides us the ability identify cells and construct a model to further deduce cellular changes from the parametric data. Experimental measurements of magnitude and phase were taken within the range of 40.0 Hz and 3.0 MHz. Measured cells are injected into a microreservoir and manipulated by both vacuum and dielectrophoretic schemes for proper placement prior to data collection. Results were subsequently compared to air and PBS solution measurements in comparable conditions. Results indicate that phase and magnitude data differ significantly for healthy and cancerous cells. The theoretical and experimental criteria for the design and operation of such devices is also presented.

4:30 PM Y4.8

Biosensors Based on Organic Thin Film Transistors. Jeff Mason¹, Zhengtao Zhu¹, Sanjay Aggarwal¹, Jamie Schnapp¹, Ellen Huang¹, George Malliaras¹, Nate Cady², Carl Batt² and Brian Ray³; ¹Materials Science and Engineering, Cornell University, Ithaca, New York; ²Food Science, Cornell University, Ithaca, New York; ³Agave BioSystems, Ithaca, New York.

Chemical and biological sensors based on organic thin film transistors (OTFTs) have begun to attract significant interest. Such devices can be easily and inexpensively fabricated on a variety of substrates, and the organic semiconducting molecules can be chemically synthesized or modified for specific applications. We have previously shown that pentacene OTFTs can be used to make sensitive humidity sensors. In the current work, we demonstrate the applicability of OTFTs for

detecting biological species in aqueous environments. Pentacene OTFTs were fabricated on Si substrates with SiO₂ as the gate dielectric, and poly(dimethyl siloxane) (PDMS) microfluidic channels were used to expose the active regions of the transistors to aqueous solutions with a range of ionic strengths and pH as well as buffer solutions containing rat basophilic leukemia (RBL) cells. We will also discuss unusual field-effect transistors based on the doped conducting polymer poly(ethylenedioxy-thiophene)-polystyrenesulfonate (PEDOT:PSS) and the application of these transistors to DNA and glucose sensing.

4:45 PM Y4.9

Ultrasensitive Nanowire Sensors for Drug Discovery and Medical Diagnostics.

Wayne Uei Wang^{1,2}, Yi Cui¹, Gengfeng Zheng¹, Keng-hui Lin¹ and Charles M Lieber¹; ¹Dept. of Chemistry and Chemical Biology, Harvard University, Cambridge, Massachusetts; ²Biophysics, Harvard University, Cambridge, Massachusetts.

Semiconductor nanowires represent an important class of nanostructured materials with the potential to impact applications from nanoscale electronics to biotechnology. For example, silicon nanowires can be configured as field-effect transistors (FETs) with characteristics exceeding that of conventional planar devices. These electronic properties make silicon nanowire FETs ideal transducers for electrically addressable, real-time and label-free sensing. Here we describe modification of nanowire surfaces with biological receptors to create nanoscale biosensors for both protein and drug molecule detection. First, antibody modified nanowires have been used to investigate limits of detection for PSA, a marker for prostate cancer. Nanowire sensors are shown to enable PSA detection at concentrations as low as 0.025 pg/ml, and moreover, the generality of this approach is demonstrated with detection of other cancer marker proteins such as carcinoembryonic antigen. The implications of these results for basic biology and medical diagnostics will be discussed. In addition, we demonstrate detection of small molecule/protein interactions in real time for Abl kinase/ATP/Gleevec, which serves as a model pathological system. The binding profile of ATP/Gleevec with the Abl kinase measured with nanowire sensors is consistent with expectations for a receptor/agonist/competitive antagonist system, and more significantly, the nanowire sensor is shown to be capable of differentiating small molecule drugs with different affinities. The implications of these results for development of tools for high-throughput drug discovery will be discussed.

SESSION Y5: Poster Session: Materials, Mechanisms, and Systems for Detection and Remediation
Chairs: Anja Boisen and Lal Pinnaduwage
Thursday Evening, April 15, 2004
8:00 PM
Salons 8-9 (Marriott)

Y5.1

Multi-Channel Piezoresistive Micromechanical Sensor Readout System.

Zhiyu Hu¹, Nazmul Islam², Syed Islam² and Thomas G. Thundat¹; ¹Life Sciences Div., Oak Ridge National Laboratory, Oak Ridge, Tennessee; ²Dept. of Electrical and Computer Engineering, University of Tennessee, Knoxville, Tennessee.

Recently micromechanical sensors such as microcantilever sensor have demonstrated ultra-high sensitivities as chemical, biological and physical sensors. Traditionally optical level detection method is very sensitivity but it is difficult to scale down to chip level. Using AMI 0.5 micrometer process we have designed and fabricated 8-channel piezoresistive micromechanical sensor readout system with a size of 2.2 mm x 2.2 mm x 300 micrometers. This module designed readout system can be integrated with different piezoresistive microcantilever array as well as its micro-fluidic system. To minimize the thermal heating effect special attention was devoted to maintain constant current and diminish power dissipation. Entire system is powered by +5 V DC battery source with less than 0.8 mW power consumption per channel. Two-stage op-amp design offers very high gain and stable output with noise compensation circuit. Up to 2.2 MHz gain band-width offers natural frequency measurement feasibility to micromechanical sensors. This multi-channel readout chip can be used to construct miniature high sensitivity biological and chemical detection system.

Y5.2

Dynamic thermal actuation for resonating cantilevers.

Alexander V. Grigorov, Peter Rasmussen and Anja Boisen; MIC, Technical University of Denmark, Lyngby 2800, Denmark.

We report a novel method to excite resonating cantilevers for MEMS. The operation of the devices is independent of the medium (vacuum, air or water), which allows for mass detection in water, or for viscosity measurements of different mediums, hitherto difficult to achieve. They

are simple to fabricate in one layer of (potentially) any conducting material- metals or polysilicon. The device consists of a cantilever with resistor device(s) perpendicularly attached close to its base (other arrangements are possible). The small size of the resistor and large surface allow for quick heating and cooling giving extremely fast thermal actuation when AC current is passed. To operate the devices a square pulse signal is applied to the resistor. At the peaks of the signal the resistor heats up and expands; at the troughs (i.e. zero current) the resistor cools down and shrinks. This longitudinal motion actuates the cantilever laterally at the frequency of the driving signal. Electrostatic, piezoelectric and electromagnetic actuation that are commonly used for resonant excitation have inherent drawbacks compared to the proposed fast thermal actuation. Electrostatic actuation only works in air and vacuum and suffers from electrostatic problems, electromagnetic actuation requires and an external field - or on chip it makes the device bigger, more complicated or layered, and piezoelectric actuation requires the use of more than one material. Different actuator designs were produced using a 2.5 um thick LPCVD polysilicon layer on top of a 2 mm thick oxide layer on a Si substrate. The structures were defined by dry plasma etching of the polysilicon layer and subsequently the oxide was under-etched in HF to release the cantilever and the resistor. The design parameters that were varied were resistor width (2 or 4 um), number of fins on the resistor (0 to 8), fin length (2 or 4 um) and a single or double actuator design. The maximum actuating frequency depends on the devices' cooling rate, hence the power input/outputs of the different designs were measured at 100 deg. C in water by using incrementing DC current and the onset of boiling (bubbles) as a set temperature point. The results showed the optimal actuator configuration and suggest a maximum actuating frequency of 1 MHz in air and 10 MHz in water (for the given actuator size). The optimal device was tested dynamically with a 160x2 um cantilever- resonance was observed at 54 kHz and the Q factor was determined to 21. The resonant frequency of a cantilever is linearly inversely proportional to its size (W/(LxL)). The heat stored in the resistors depends on their volume i.e. cubically proportional to size. The heat transfer to the substrate is linearly proportional to the cross-sectional area of the resistors i.e. quadratically proportional to size. Hence, both the resonant frequency and the maximum actuating frequency increase linearly as size goes down, and the actuation principle is fully scalable.

Y5.3

Calorimetric Detection of DNA Hybridization Using Microcantilever.

Sangmin Jeon, Karolyn Hansen and Thomas Thundat; Oak Ridge National Lab, Oak Ridge, Tennessee.

Hybridization of DNA was detected with silicon microcantilever array sensors that were coated on one side with a thin layer of gold. By increasing the temperature of the array higher than the melting temperature of the double-stranded DNA (dsDNA) hybrid, the consequent surface stress change causes the cantilever array elements to bend. This bending is due to the denaturation of the dsDNA hybrid. Since the melting temperature of double-stranded DNA depends on the number of A-T and C-G base pairs and the length of the oligomer, it is used to detect single mismatched base pairs. Cantilever array deflection due to DNA denaturation occurred when the DNA was immobilized on either the gold-coated side or the silicon side of the cantilevers.

Y5.4

Optimising the design of a cantilever biosensor with single crystalline silicon piezoresistor.

Peter Andreas Rasmussen and Anja Boisen; Mikroelektronik Centret (MIC), Technical University of Denmark, Lyngby, Denmark.

We present a cantilever with piezoresistive readout optimised for the task of measuring isotropic surface stresses found for example when biological species immobilise on the surface of the cantilever[1]. The cantilever sensor is based on an SOI substrate. The optimisation takes into account the different stress regimes encountered on the cantilever as a function of clamping effects, and uses the results to specify optimum design, placement and doping of the piezoresistors. Particularly, this model clearly pinpoints the difference between cantilevers with integrated readout developed for AFM and biosensing, respectively. A finite element analysis is used to back up the analytical results. Most piezoresistive cantilever sensors today use p-doped resistors, where a very high longitudinal gauge factor is easily obtainable on standard (100) silicon wafers by bulk anisotropic etching along the [110] directions. For the AFM case the forces acting on the apex of the cantilever will create a dominantly longitudinal stress, since only a fraction will be converted to transversal stress through Poisson contraction. This will give a high sensitivity. However, the stress created on the surface of a cantilever when measuring the immobilisation of biological species will be isotropic in nature, and on long slender beams, where the effect of the clamping is vanishing, the effective gauge factor will be the sum of the longitudinal and transversal gauge factors. For the above mentioned

p-type resistor (on (100) silicon) the sensitivity will be very low, as the longitudinal gauge factor and the transversal gauge factor are of almost equal size but of opposite sign. Using the maximum values for the gauge factor found at low doping levels[2] the effective gauge factor will be almost an order of magnitude higher with an n-type resistor than with a p-type resistor. For short resistors, where the stress development is restricted by the clamping, i.e. the cantilever should be treated as a plate, the situation becomes more complex, and the sensitivity will depend on the ratio of the longitudinal to the transversal part of the resistance, and hence be very design specific. [1] R. Raiteri, M. Grattarola, H. Butt, and P. Skladal, Micromechanical cantilever-based biosensors, *Sensors and Actuators B: Chemical*, 79(2-3):115-126, 2001. [2] C.S. Smith, Piezoresistance effect in germanium and silicon, *Phys. Rev.*, 94(42), 1953.

Y5.5

Detection & Characterisation of Leaks in Pressurised Fluid Pipelines Using Microcantilevers. Philip G Evans^{2,1}, Ali

Pasian¹, Aude L Lereu^{2,1}, Venugopal K Varma¹, Thomas L Ferrell¹ and Thomas G Thundat¹; ¹Oak Ridge National Laboratory, Oak Ridge, Tennessee; ²Dept. of Physics & Astronomy, University of Tennessee, Knoxville, Tennessee.

Oil and natural gas account for a substantial percentage of energy produced in the USA and are transported by upwards of half a million k.m. of pipelines. Almost any length of these pipelines is vulnerable to failure: corrosion, material flaws, accidental damage and acts of terrorism are all very real possibilities. It is therefore crucial that any pipeline failure is detected as soon as possible for the safety of the public and economic well-being of the country. A simple and cost-effective MEMS sensor platform to detect, characterise and locate leaks in pressurised gas pipelines is presented. The response of a piezoresistive microcantilever to frequency components of an acoustic field is measured and information regarding a possible leak gathered. By using arrays of similar microcantilevers and a novel tuning technique, the location, size and geometry of the leak site may be found. The MEMS sensor is shown to function favourably both inside and outside of pressurised pipelines and may be implemented with complementary leak detection systems to provide a full suite of leak protection.

Y5.6

High Resolution Visualization of Pathogen Signatures and Function by In Situ Atomic Force Microscopy.

alexander j malkin², marco plomp² and alexander mcpherson¹;

¹Department of Molecular Biology and Biochemistry, University of California, Irvine, California; ²Department of Chemistry and Materials Science, Lawrence Livermore National Laboratory, Livermore, California.

We will present high-resolution in situ atomic force microscopy (AFM) studies of several pathogens including plant and human viruses, bacteria and bacterial spores. Several virus families, which span the size range from 17 nm diameters for satellite plant viruses to nearly half a micron for vaccinia, were characterized (1-3). In the case of icosahedral T=3 small plant viruses of 28 nm diameter, it was demonstrated that not only protein capsomeres on the virion surfaces could be clearly resolved, but also different T=3 virus families could be discriminated on the basis of capsid structure(1). The lateral resolutions of the order of 2.0-2.5 nm were achieved on viruses, in situ. Further dissection of viral particles with detergents and enzymes, as was demonstrated(2) with Herpes Simplex Virus (HSV-1) revealed in addition to the intact, enveloped viruses: (a) the underlying capsids with associated protein complexes and membranes, (b) the capsomeres composing the HSV-1 capsid, and their surface arrangement, and (c) the encapsidated viral DNA. The work on individual virus particles, in situ, culminated in the study of vaccinia virus(3). Vaccinia virus, the basis for the smallpox vaccine, is a laboratory model for what many consider to be one of the most significant bioterrorist threats. It is one of the largest and most complex viruses to have human tropism. Intact vaccinia virions were visualized under physiological conditions. The vaccinia virions were subsequently treated individually, or with a series of chemicals and/or enzymes intended to degrade the particles from the outside to the inside, and ultimately to reveal the internal structure of the virus. Consecutive subviral structures including core envelopes, viral cores, nucleoprotein filaments, as well as, viral DNA were visualized. A novel structural model of vaccinia virion based on the hierarchy of observed substructures will be presented(3). In the case of bacterium *Chlamedia trachomatis*, we were able to identify surface exposed proteins versus proteins embedded in the outer membrane. Differences in morphological signatures and high-resolution structural attributes of various bacterial spores species were demonstrated. Furthermore dissection of spores revealed strain-specific nanometer scale features of internal structures. Dynamic morphological changes of dormant bacterial spores in response to the environment were visualized. These results demonstrate that AFM could be incorporated into the array of

early detection techniques for identifying infectious and pathogenic organisms and agents. 1. A.J. Malkin, M. Plomp and A. McPherson, A. (2004). In: *DNA Viruses: Methods and Protocols*. (Ed. P.M. Lieberman). *Methods in Molecular Biology Series*. The Humana Press Inc., in press. 2. M. Plomp, M.K. Rice, E.K. Wagner, A. McPherson and A.J. Malkin (2002). *American J. of Pathology*. 160, 1959-1966. 3. A.J. Malkin, A. McPherson and P.D. Gershon (2003) *J. Virology*, 77, 6332-6340.

Y5.7

Optimization for High-Specificity Sensor Coating. Ida Lee¹, Karolyn M Hansen² and Thomas G Thundat², ¹Electrical & Computer Engineering, University of Tennessee, Knoxville, Tennessee; ²Life Science Division, Oak Ridge National Lab., Oak Ridge, Tennessee.

MEMS/NEMS-based sensors can be made into multiple arrays to detect specific chemical and biological agents. The sensing mechanisms are nanomechanical deflection due to molecular interactions and/or resonance frequency shift due to mass loading from adsorption. The kinetics of the reaction is controlled by the concentration of the agent and also is a function of the surface properties of the coating. In a liquid system, the boundary layer of the liquid at the surface acts as a barrier, affecting the diffusion of the agent, and can change the reaction kinetics. Thus, optimization of the density of self-assemble surface coating plays an important role in obtaining consistent and ultrasensitive signals. For the reaction in which diffusion is the rate-limiting step, the closely packed self-assemble coating may not be the optimal solution. Here we describe by using the Q-control atomic force microscope (AFM) we can visualize the distribution of individual molecule coated on surface. Epitaxially grown Au{111} on mica substrates were functionalized with 5-end thiolated DNA oligomers of known length and sequence. Two DNA oligomers were used: the first was 20 bases in length with no secondary structure, and the second was 23 bases in length and formed a hairpin loop by self-hybridization. Discreet 3 micro liter drops of DNA solution were placed on the gold surface and incubated for various hours in a humid atmosphere. Substrates were washed sequentially with phosphate buffer, deionized water, and ethanol and dried under a nitrogen stream. The substrates were then imaged by AFM under ultrapure nitrogen with 0 relative humidity. The first kind of DNA oligomers appeared tilted slightly on the surface. For the sample incubated for 2 hours, the vertical length from the top of the molecule to the lowest valley that the AFM can reach is 0.9 nm, while for extremely closed packed (incubated for 40 hours) condition, the vertical distance is only 0.4 nm. This indicates that the diffusion will be more difficult when the DNA is closely packed. The second kind of DNA oligomers (hairpin loop) have less surface coverage compared with the first kind with the same incubation time, and the vertical distance is 1.4 nm which indicates the higher accessibility for agent diffusion.

Y5.8

Abstract Withdrawn

Y5.9

Cationic inorganic materials for remediation of anionic pollutants. Scott R. J. Oliver, Tolulope O Salami and Dat T Tran; Department of Chemistry, SUNY-Binghamton, Binghamton, New York.

The EPA has listed many anionic species, such as chromate, selenite and arsenate, as priority pollutants. Current anion-getters have limited thermal and pH stability, which forbids their use in the harsh conditions of many waste solvents. We are interested in developing a new class of thermo-chemically stable cationic open-framework metal oxide hosts with anionic guests. These microporous materials have potential application in anion-exchange, anion-trapping and base catalysis. The properties are opposite to those of zeolites, which are primarily anionic hosts, and therefore limited to cation-based properties. Our efforts towards the solvothermal synthesis and characterization of cationic extended materials will be discussed. We have discovered a cationic inorganic material that expels nitrate and sorbs a variety of anions, such as dichromate and benzoate, in high capacity. Anion-exchange of pertechnetate analogs, for the potential application of pretreatment of high-level radioactive waste (HLW), has also been successful. Our materials could also be used to remove chromate from these wastes, which are known to decompose the glass melts that trap the HLW. Certainly, anion remediation is a high priority and our cationic materials have the potential to address some of these problems.

Y5.10

A Label-Free Colloidal Assay for Membrane Binding Affinity. Esther M. Winter¹, Michael M Baksh² and Jay T Groves³; ¹Chemical Engineering, University of California, Berkeley, Berkeley, California; ²Biophysics, University of California, Berkeley, Berkeley, California; ³Chemistry, University of California, Berkeley, Berkeley, California.

Two-dimensional dispersions of silica microbeads, coated with fluid lipid bilayer membranes, exhibit transitions between dispersed and condensed phases. The collective phase behavior of this novel colloidal system is governed by membrane-membrane interactions and serves as a readily detectable amplifier for subtle molecular events occurring at the membrane surface. This study explores the efficacy of colloidal phase transitions as a label-free and power-free assay of membrane surface binding affinity. Equilibrium dissociation constants for the interactions between ligands and membrane-associated receptors are measured with the colloidal assay and compared to conventional read-out techniques. The membrane-derivatized colloid assay is compatible with high-throughput screening technologies.

Y5.11

The Role of Film Thickness in Organic Thin Film Transistor Gas Sensors. Christopher Chen, Frank Liao and Vivek Subramanian; Electrical Engineering and Computer Sciences, University of California, Berkeley, Berkeley, California.

Organic thin film transistors (TFTs) have shown much promise for use in a variety of gas sensing applications. When exposed to an analyte, gas molecules interact with the organic active layer of these devices and influence transistor behavior in a variety of ways, including shifts in on-current, transconductance, mobility, and threshold voltage. Furthermore, because several organic semiconducting materials are soluble in common solvents, TFTs can be easily fabricated by spin-casting or inkjetting the active layer. In this study, we analyze the relationship between film thickness, analyte exposure time, and the response of organic TFTs. We report on transient adsorption and desorption characteristics for sensors with different film thicknesses exposed to a variety of analyte compounds. Experimental results indicate that film thickness greatly influences certain sensor characteristics, while other parameters are thickness-independent. We report a strong correlation between film thickness, exposure time, and change in on-current. Our analysis indicates distinct relationships between sensor film thickness and shifts in transconductance, threshold voltage, and mobility. Finally, we demonstrate the existence of an optimal active layer thickness for each parameter examined.

Y5.12

Directed Assembly of Nanomaterials for Miniaturized Chemical Sensors. Ming Su and Vinayak Dravid; Department of Materials Science and Engineering, Northwestern University, Evanston, Illinois.

The abundant knowledge about synthesis of nanomaterials has paved the way for the controlled construction of miniaturized sensors and sensor arrays. However, self-assembly methods lack precise position control, the ability to pattern multiple species and overall reproducibility, among other considerations. In view of these challenges, directed assembly of nanobuilding blocks by various site-specific patterning approaches is desired. We have resorted to dip-pen nanolithography (DPN) to pattern diverse nanomaterials (semiconducting metal oxides, conducting polymers, and hybrid/organic materials) as various building blocks which assemble into final functional form. The site-specific and multiple-ink patterning capabilities of DPN are put to use in fabrication of miniaturized chemical sensor arrays with either sol-based inks which yield gas sensitive semiconducting metal oxides, or "monomer" inks which polymerize to form conducting polymer structures. The miniaturized sensors constructed in this manner show ultra-fast response and rapid recovery, and can be further integrated to form sensor array that have excellent gas recognition capabilities. We have recently extended this approach to "electronic nano-nose", whereby multiple sensor elements are integrated in an array for multiplexed gas sensing.

Y5.13

Detection and Classification of Volatile Organic Amines and Carboxylic Acids Using Arrays of Carbon Black-Dendrimer Composite Vapor Detectors. Ting Gao and Nathan S Lewis; Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California.

Carbon black-polymer composite chemiresistive vapor detectors have been prepared using dendrimers as the polymeric constituent of the composite. Amino-terminated dendrimer-carbon black composites exhibited an enhancement in detection sensitivity of 103 for volatile carboxylic acids as compared to non-dendrimeric insulating polymer-carbon black composites. Similarly, protonated carboxylate-terminated and protonated amino-terminated dendrimer-carbon black composites showed an 103-104 increase in sensitivity for detection of volatile amines relative to the response of non-dendrimeric insulating polymer-carbon black composites. The protonated amino-terminated dendrimer carbon black composite detectors exhibited a signal-to-noise ratio (S/N) of 22.4 ± 0.9 upon

exposure to 2.7 ppb of butylamine in air, whereas poly (ethylene oxide)-carbon black composites exhibited a S/N of 3.5 ± 1.2 at 54 ppm of butylamine. The protonated amino-terminated dendrimer-carbon black detectors additionally exhibited relatively small responses to water vapor. Compositional diversity in an array of protonated amino-terminated dendrimeric vapor detectors was obtained by varying the type and generation of the dendrimer, and the type and concentration the acid dopant. 15 analytes chosen from primary amines, branched amines, anilines and non-amine organic analyte vapors were all robustly discriminated from each other by their different response patterns on the dendrimer-containing detector array. The signals produced by these 15 analytes additionally clustered into groups based on the chemical class of the analyte.

Y5.14

Synthesis of nanostructured electrodes for biosensor applications. Maria P. Gil, Donghai Wang and Yunfeng Lu; Chemical and biomolecular engineering, Tulane University, New Orleans, Louisiana.

One of the current driving forces in the research field of glucose biosensors for food, biological and medical applications is the miniaturization of the sensors for in vivo applications and reduction in the size of portable monitoring devices. We are proposing a new type of material for the transducer of the sensor. Two of the main characteristics of nano-scale materials are their small size and dramatically high surface area. An electrode with high surface area would allow the immobilization of a high amount of enzyme, and in consequence, the sensor would display a high sensitivity while reducing its size. Nano-scale materials for application as electrodes for amperometric glucose biosensors were synthesized by the following steps: 1) construction of a nanostructured mesoporous thin film template by self-assembly of surfactant and silicate species, 2) electrodeposition of platinum within the pores of the silica template, 3) removal of the silica template, and 4) immobilization of the enzymes on the platinum electrode. X-ray diffraction (XRD), transmission electron microscopy (TEM) and cyclic voltammetry were used to characterize the synthesized materials, and the glucose sensitivity was determined amperometrically. Two types of platinum nanostructured materials (2D nanowires or 3D nanomesh structures) were synthesized depending on the surfactant used to prepare the silica template. The sensors prepared with these platinum electrodes show a sensitivity to glucose ranging from 0.6 $\mu\text{A-cm}^2/\text{mM}$ to 1.4 $\mu\text{A-cm}^2/\text{mM}$ and retain this sensitivity for about a month. In comparison, an electrode prepared by chemical vapor deposition showed a sensitivity of 0.15 $\mu\text{A-cm}^2/\text{mM}$ and retained this sensitivity for about 3 days, after which it began decreasing.

Y5.15

Nanotechnology for Explosive Detection: The use of fluorescence nanoparticles for sensing NO₂ containing organics. Sorelis Nieto¹, Lewis Mortimer Gomez¹, Alberto Santana¹, Samuel P Hernandez¹, R. Thomas Chamberlain³, Richard Lareau³ and Miguel E. Castro¹; ¹Chemical Imaging Center and Center for Chemical Sensor Development Department of Chemistry, The University of Puerto Rico at Mayaguez, Mayaguez, USA, Puerto Rico; ²Chemistry, University of Puerto Rico at Mayaguez, Mayaguez, USA, Puerto Rico; ³Federal Aviation Administration, William Hughes Technical Center, Atlantic City.

Homeland security is an important area that requires input from a number of science and engineering areas. Nanotechnology is expected to play a central role in contributing to improvements in homeland security issues. Small and lightweight and field deployable systems that can be used for explosive detection systems can be developed with new and emerging nanotechnologies. In this presentation, we will present recent results on the use of fluorescent nanoparticles for the detection of trace amounts of TNT, a powerful explosive. Quantum dots, excited with a femto second laser pulse, exhibit a decrease in fluorescence signal intensity and small shift in emission wavelengths upon addition of solutions containing traces of TNT. The direction of the shift depends on the particle size. The chemistry that takes place on the nanoparticle surface is interrogated with a battery of surface analytical techniques that includes X-ray photoemission spectroscopy, near field optical microscopy, atomic force microscopy and Fourier transform infrared and Raman microscopy.

Y5.16

Ultranancrystalline Diamond as a Platform Material for Biosensor/MEMS Technologies. Jennifer E Gerbi¹, James Birrell¹, Jeff W. Elam¹, Wei Fan², Nicole M. Haralampus Grynawski¹, Sanjib X. Saha¹, Jian Wang¹, Xingcheng Xiao¹, Millicent A Firestone¹, Orlando Auciello¹ and John A. Carlisle¹; ¹Materials Science, Argonne National Laboratory, Argonne, Illinois; ²Northwestern University, Evanston, Illinois.

Ultranancrystalline diamond (UNCD) is a fine-grained (3-5nm),

smooth, biocompatible diamond material. Here we give an overview of various properties that make this material particularly suited as a platform for biosensors and MEMS devices: mechanical (high hardness and fracture strength), electrical (highly conductive with significant mobilities), chemical (surface modification), and tribological (low friction and stiction). We will discuss the microwave plasma-enhanced chemical vapor deposition growth process that is used to produce UNCD films, the various seeding processes employed, the effects of alternate plasma chemistries, and doping. We show how the nanostructure of the films is affected by these processes, and discuss this in the context of UNCD as a platform biosensor/MEMS material. In particular, we will discuss conductive UNCD as a basis for electrically active FET and MEMS devices, and address device integration issues critical to the development of real-world MEMS structures, such as the fabrication of diamond-to-metal contacts and the growth of oxides on UNCD films by both sputter deposition and atomic layer deposition.

Y5.17

Laser Ablation of Polydimethylsiloxane for Extracellular Neuronal Electrode Arrays. Rachel Yotter, Tai Chen and Robert Bruce Darling; Electrical Engineering, University of Washington, Seattle, Washington.

Biological researchers need to simultaneously record electrical signals from multiple locations in order to fully understand a neuronal system. An ideal electrode array would conform to the physical layout of the neurons, and it would also be optically transparent and biocompatible. Polydimethylsiloxane (PDMS) is a material that fits these requirements. An additional benefit of PDMS is that the biological tissue can be pinned directly onto the PDMS. In this work, we present a new fabrication method to create small features in thick (1mm) PDMS. First, we developed recipes to deposit PDMS uniformly onto a substrate. By adjusting the spin speed and ramp-up time, we were able to spin on PDMS coatings with a uniform thickness between 500 μ m and 1mm. Then, using the ESI-4440 laser system, we successfully developed methods to "cutout" shapes and drill holes in 1mm-thick PDMS, without damaging the underlying substrate. The average diameter for a 1mm-deep hole was 43 μ m. For cutouts, we made partial cuts through the PDMS, then either pulled or punched out the inside material using sharp tweezers. We successfully punched out 100 μ m squares using this method. The initial results indicated that the feature depth can be controlled to within 8 μ m, and the typical edge roughness of holes was 8 μ m. We characterized the feature depth in relation to the number of laser pulses, and the two values were related through a linear curve. We then used this curve to successfully predict the feature depth of subsequent holes and shapes. By spin-coating PDMS onto a hydrophobic surface, the patterned PDMS was easily peeled off and placed onto a glass substrate patterned with gold. This eliminated any damage to the gold electrodes by the laser micromachining process. We have also developed methods to create channels in PDMS, which help with the placement of neuronal axons. Using laser micromachining, we have taken the first step to developing an improved electrode array to record extracellular neuronal signals. The fabrication process is both simple and flexible. A hole diameter of 43 μ m is compatible with the size of the neurons, and a thickness of 1mm allows biological researchers to use existing methods to physically fix the neuronal tissue.

Y5.18

Lithographically Patterned Microcoils for High Sensitivity NMR Chemical and Biological Detection. Julie Lynn Herberg¹, Sarah C Chinn¹, Robert Maxwell¹, Anthony F Bernhardt² and Vincent Malba²; ¹Chemistry and Material Science, Lawrence Livermore National Laboratory, Livermore, California; ²Electronics Engineering Technologies Division, Lawrence Livermore National Laboratory, Livermore, California.

It is well known that nuclear magnetic resonance (NMR) offers a non-destructive, powerful, structure-specific analytical method for the identification of suspect analytes and biological systems. NMR, however, is hampered by relatively poor sensitivity. Lithographically produced NMR receiver coils offer a unique solution to increasing the sensitivity of a conventional NMR system while retaining the high information content data streams NMR provides. Recent advances in micro-receiver technology, for example, have demonstrated a >70-fold increase in mass sensitivity, but are typically limited to solenoidal coil forms. Lithographic production methods, however, possess a level of flexibility that goes beyond other production methods enabling the fabrication of a diverse range of coil forms. Here, detailed electrical characterization of microcoils produced by a unique three dimensional laser lithography system that can pattern on curved surfaces will be discussed, along with integration of these microcoils into a conventional and portable NMR systems. Initial NMR results obtained with both 1 mm and 360 mm O.D. receiver coils will be presented along with ongoing methods of resolution enhancement and the future integration

of a Capillary Liquid Chromatography separation-detection system. Current and future applications of lab-based microcoil NMR techniques will be discussed.

Y5.19

Surface wave properties of AlN epitaxial layers. Jianzeng Xu, Qianghua Wang, Hao Ying and Gregory Auner; Electrical & Computer Engineering, Wayne State University, Detroit, Michigan.

Hexagonal AlN, known for its high acoustic velocity, is a promising candidate for highly sensitive chemical or biological detection. A lot of study has been done regarding Rayleigh wave propagating on AlN for microwave devices. However, these devices are not suitable for biological detection due to the acoustic energy dissipation in liquid. We hereby report the study on excitation of Rayleigh wave and transverse mode wave on AlN epitaxial layer. AlN thin film has been deposited on C-plane, R-plane, and A-plane Sapphire by plasma enhanced molecular beam epitaxy method (PSMBE). XRD shows the highly epitaxial growth of (0001) AlN// (0001) Al₂O₃, (11 0) AlN//(1 0 2) Al₂O₃, and (10 0) AlN//(11 0) Al₂O₃. The velocity dispersion with respect to kh is presented, where, k is the wave number and h is the film thickness. The effect on the transverse mode excitation due to the misalignment between wave propagation direction and crystal axis has been studied. Linear temperature coefficient is derived on these AlN acoustic wave devices. Device mass sensitivity of is also determined experimentally. The study would help to clarify the excitation mechanism for transverse mode on AlN and provide practical guidance for developing AlN based biological sensors.

Y5.20

Investigation on Al/AlN/Al Structure for Ultrasonic Biological Transducers. Qianghua Wang¹, Jianzeng Xu¹, Ratna Naik² and Gregory W Auner¹; ¹Department of Electrical and Computer Engineering, Wayne State University, Detroit, Michigan; ²Department of Physics and Astronomy, Wayne State University, Detroit, Michigan.

Here we report the progress on developing AlN based piezoelectric transducers for ultrasonic imaging applications. Aluminum nitride (AlN) thin films was sandwiched by two metal layers deposited on silicon substrate. In order to avoid thermal damage to silicon device and integrated circuit, the deposition temperatures was chosen at 450°C, lower than 650°C for epitaxial AlN films grown by plasma source molecular beam epitaxy (PSMBE) system. X-ray diffraction (XRD) shows the films exhibit with a c-axis texture. Oxygen component is about 1% from X-ray photoelectron spectroscopy (XPS) analysis. The roughness of film surface determined by AFM decreased with increasing target-substrate distance and increasing nitrogen concentration. Electrical properties of Al/AlN/Al sandwich structure have been measured as a function of the frequency in 1kHz - 10MHz region. Optimizing the processing conditions is important to utilize AlN thin films for various devices.