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Air Force Office of Scientific Research (AFOSR)4015 Wilson Blvd, Room 713 Arlington, VA 22203-1954LT COL Paul C. Trulove, Ph.D. Program Manager, Surface and Interfacial Science AFOSR/NLPhone (703)696-778711. SUPPLEMENTARY NOTES DURIP Equipment Grant/Cooperative Agreement20030915 020					
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13. ABSTRACT (Maximum 200 Words) We purchased an InfraScope II thermal imaging microscope from Quantum Focus Instruments Corporation. The machine was purchased within a month after receiving the DURIP funds, and was installed in the Micro and Nanotechnology Laboratory. The total cost of the OEL InfraScope II Infrared Imaging Microscope is \$ 129,540 (charged to the core account) plus \$ 23,373, charged to					
a match account provided by the Electrical and Computer Engineering Department of the University of Illinois.					
We have used the machine in a number of scientific research projects. The unique infrared microscopy capability provides capability to conduct temperature measurement on many micro devices, including: thermally active dip pen nanolithography (DPN) probes, Parylene surface micromachined fluid shear stress sensors, <i>in situ</i> Parylene thickness sensors, micro heaters for fluidics applications, and three dimensional hot wire anemometers.					
The measurement capability allows us to have accurate rate in short time, providing significant benefits for many DoD funded work. Results obtained using the microscope have been used to validate design concept and to confirm accuracy of numerical modeling. Results are published in 3 peer reviewed conference papers, 1 journal paper, and 2 journal paper submissions.					
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DURIP REPORT

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Project Title

A Proposal to Acquire a Micro Thermal Imaging Microscope

(F49620-02-1-0221; Fiscal Period: 5/1/02 to 4/30/03)

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We purchased an InfraScope II thermal imaging microscope from Quantum Focus Instruments Corporation. The machine was purchased within a month after receiving the DURIP funds, and was installed in the Micro and Nanotechnology Laboratory.

The total cost of the QFI InfraScope II Infrared Imaging Microscope is \$ 129,540 (charged to the core account) plus \$ 23,373, charged to a match account provided by the Electrical and Computer Engineering Department of the University of Illinois.

We have modified the InfraScope II to accommodate micromanipulator probes. This allows chips to be directly probes and biased by bypassing packaging efforts.

We have used the machine in a number of scientific research projects. The unique infrared microscopy capability provides capability to conduct temperature measurement on many micro devices (listed below).

- (1) Characterization of temperature of thermally active dip pen nanolithography (DPN) probes
- (2) Characterization of surface temperature of Parylene surface micromachined fluid shear stress sensors
- (3) Characterization of surface temperature of Parylene thickness sensors
- (4) Characterization of surface temperature of micro heaters for fluidics applications
- (5) Characterization of surface temperature of three dimensional hot wire anemometers.

The measurement capability allows us to have accurate rate in short time, providing significant benefits for many DoD funded work. Results obtained using the microscope have been used to validate design concept and to confirm accuracy of numerical modeling. Results are published in 3 peer reviewed conference papers, 1 journal paper, and 2 journal paper submissions.

Detailed work on the first four projects is summarized in the following.

Project #1: Characterization of temperature of thermally active DPN probes

We have developed active DPN probes using thermal bimetallic actuation principles for high throughput DPN lithography, Figure 1. The thermal bimetallic actuation provides simplicity of materials and fabrication and small footprints. The probes are heated using ohmic resistors to selectively control the writing process. The temperature of the probe and tip during operation is of a major concern. If the temperature is overly high, it may disrupt sensitive biochemical molecules, or inks. Heat on one probe may be transferred to neighboring probes, causing unwanted crosstalks.



Figure 1: (a) SEM micrograph of a 10-probe arrayed DPN probe. (b) An individual heating resistor located at the base of the DPN probe is shown.

We have developed an analytical thermal transfer model considering both longitudinal heat conduction and convection loss [1]. Results of the modeling effort are shown, Figure 2. However, the results are not completely reliable due to non-ideality of modeling and variation of thermal properties of thin film materials.





Using the infrared thermal microscope, we were able to measure definitively the emission from the probe during operation. This gives independent confirmation of temperature profile as well as thermal cross talk. Because of the results, we were able to calibrate the analytical model to provide more accurate modeling. Results enabled by the thermal microscope has been included in [2] and a submitted journal paper.



Figure 3: Thermal microscope image of a heated DPN probe.

Project #2: Characterization of surface temperature of Parylene surface micromachined fluid shear stress sensors

We have developed a new surface micromachining process using Parylene as structural material and photoresist as the sacrificial layer. The new process and device characterization results are described in a new paper submitted to the Journal of MEMS [3]. It is a low cost alternative to conventional surface micromachined sensors that use silicon nitride as the structural layer and silicon oxide as the sacrificial layer.

Using the process, we have developed a fluid shear stress sensor. The sensor consists of a resistor located on top of a suspended Parylene membrane that is spaced 30 μ m from the substrate. The resistor serves as both a heater and a temperature sensor. Heat is generated by passing an electric current to the resistive heater. The temperature rise compared to the ambient temperature is defined as over-heat ratio. The heat is lost through air convection. The suspended membrane limits heat loss to the substrate. The convection heat transfer coefficient is a function of the local flow shear stress, which is correlated to the viscosity and the flow rate gradient in the boundary layer.



Figure 4: Optical micrograph of a shear stress sensor, consisting of a heater/sensor suspended on a 400-µm-diameter membrane made of Parylene.

The temperature of the resistive element under a giving heating power is critical for the design and operation of this device. Due to the three dimensional nature of the membrane geometry and the fact many thermal properties of thin film materials are not known exactly, modeling of temperature profile is difficult and not entirely accurate. Using the thermal microscope, we were able to calibrate the temperature directly Figure 5. The microscope provides needed spatial resolution for temperature measurement. The direct thermal measurement was used to calibrate our analytical models to increase accuracy.



Figure 5: Image of a membrane with serpentine heater. At overheat ratio of 0.2, the average temperature is 92 °C.

Project #3: Characterization of surface temperature of Parylene thickness sensors

Parylene is an emerging material for MEMS. It is an organic material that is grown by using the chemical vapor deposition method at room temperature. The deposition thickness is commonly controlled by the amount of solid-phase dimer loaded in a sublimation chamber. In a conventional deposition machine, the end point of the process is designated by the moment the dimer is exhausted. However, this end-of-process criterion does not offer precise, repeatable control of film thickness.

We developed an *in situ* end-point detector for a Parylene chemical vapor deposition process. The detector is based on the thermal transfer principle and can be implemented on commercial Parylene deposition systems with minimal system modification. Such a sensor enables a user to stop the deposition when a targeted thickness is reached. The end point detector is very simple to implement on existing Parylene deposition systems.

The schematic diagram of the Parylene sensor is shown in Figure 6. The sensor consists of a heating element and a temperature sensor. The heater and the temperature sensor are located at distal ends of two diving-board-type cantilever beams. The distance between the distal ends of the two cantilever beams, denoted d, is well defined in the mask layout. Using microlithography, the size of the gap can be accurately defined. The heater, made of thin-film metal coil, generates ohmic heating when an electrical current passes through. This heat may be transmitted to the sensor by two possible heat-transfer modes. Under the first transfer mode, the heat can be conducted through the gap between the distal ends of the two beams if a thermally conducting medium (such as air or Parylene) is present. Under the second transfer mode, the heat can travel the lengths of the two cantilever beams and the supporting silicon substrate. Obviously, the second heat transfer mode involves a much longer heat conduction path and greater thermal mass.

Parylene is deposited in a low-pressure environment, with the typical deposition pressure ranging from 20 to 40 mtorr. When a sensor with an open gap is placed in a vacuum, the thermal conduction through the gap is negligible. The second heat transfer mode dominates at this point.



Figure 6: Schematic diagram of Parylene end point detector.

As Parylene is deposited in a conformal fashion, the distance between the two distal ends of cantilevers is gradually reduced, Figure 7a. When the Parylene thickness reaches d/2, the two Parylene fronts will meet, thereby filling the gap and completing a thermal conduction path Figure 7b. As the gap is filled with Parylene, a thermally conducting medium, heat can be transferred by both the first and the second transfer modes. Heat generated by the heater now has a "thermal short-cut" to reach the temperature sensor. This change of thermal transfer characteristic is used to infer the process end point. A single sensor with a gap d can indicate when the Parylene thickness reaches d/2.



Figure 7: Side profile of sensor during the Parylene deposition process.

Using the infrared microscope, we were able to generate thermal images to prove the concept of the device and to verify performance specifications. The heater is shown to the left and the temperature sensor is to the right in Figure 8. Without the Parylene, the temperature of the temperature sensor is clearly much lower than the temperature of the heater. The instrument was critical in helping us making rapid progress on this sensor, optimize sensor designs, and publish two peer-reviewed technical papers [4, 5].



Figure 8: IR image showing the heater and the temperature sensor before Parylene is deposited.

Project #4: Characterization of surface temperature of micro heaters

We are building microfluid devices for a DNA detection sensor under a DURINT/Symbiosis program support. Resistive heaters and thermal resistive temperature sensors are needed for the proposed microfluid system, for heating samples for stringency tests, and for optional PCR operations.

We have developed a method of using *in situ* silver and gold deposition to produce resistive heaters <u>without</u> masks. A chemical solution is flown through a pre-fabricated serpentine flow channel shaped as a resistor. The solution is a mixer of three parts: A: silver nitrate, ammonium hydroxide; B: sodium hydroxide, ammonium hydroxide, and C: formaldehyde. The channels are molded in PDMS. The PDMS piece is made to contact the substrate where the resistors will be placed on. Optical micrograph of the resistor is shown in Figure 9.



Figure 9: Optical micrograph of a resistor made on a silicon dioxide surface using in situ fluid reaction.

We have used the thermal microscope to measure the heat generation process. Without the microscope for temperature measurement, confirmation of temperature profile would be prohibitively difficult and time consuming. The microscope allows us to quickly establish the proof-of-concept. The IV characteristics of the resistor were established by measurement as well. A paper on this topic is to be submitted to the Applied Physics Letters.



Figure 10: At 50 mA current input, the mean temperature is 61.3 oC while the maximum temperature is 75.4 °C. b. At 70 mA, the average temperature is 79.8 °C.

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