Precision Deposition of Functional Layers for Microcantilever Sensor Generation

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Abstract

A new platform of highly sensitive chemical and biological sensors using microcantilever arrays as transducers has emerged. In the critical step of functionalization, a chemically selective layer is deposited onto the cantilever surface making the sensor specific to binding or adsorption of a target analyte. Very precise deposition in volume and spatial accuracy has been achieved through the development of an inkjet functionalization system. In this paper, the methods for achieving maximum resolution are discussed. A high precision motion control system is presented which includes an X-Y stage powered by linear electric motors to achieve sub-micron positioning. A novel laser-optical registration method is developed to achieve the desired accuracy between the nozzle tip and the substrate. The inkjet functionalization system has enabled research to understand the effects of polymeric coatings on cantilever stiffness and quality factor. [redacted]

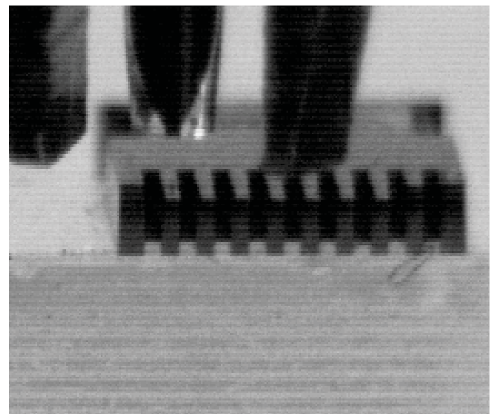

Figure 1. *Silicon cantilever array comprised of eight cantilevers with dimensions 500 μm in length, 100 μm in width and 1 μm thick positioned beneath piezoelectric inkjet dispenser.*

Introduction

Silicon microcantilevers were initially developed for atomic force microscopy (AFM) where the force sensing capabilities are used for imaging surfaces at atomic resolution. Microcantilevers were later proposed as transducers for sensing applications for a variety of physical properties including chemical, thermal, magnetic and electrical [1]. Since the mid-1990s, there has been substantial interest in microcantilever chemical and biological sensors due to the high sensitivity that can be achieved. Applications of gas phase, liquid phase and biosensors are reviewed in [2] that include the detection of mercury vapor, humidity sensors, pH detection, DNA hybridization and E. coli detection.

Silicon cantilevers comprise the physical transducer, while a chemically selective layer is used to provide affinity to a specific target analyte. Selective layers are designed using concepts of molecular and biomolecular recognition. Commonly used materials include polymers, self-assembled monolayers, proteins and antibody-antigen. Functionalization of microcantilevers refers to the formation of thin layers of the chemically selective materials on the cantilever surface. Common methods for microcantilever functionalization include micropipetting, capillary tube immersion and bulk functionalization. However, these methods do not allow control over the deposited mass or precision in location of the deposited material. Inkjet deposition of functional layers offers the benefits of a non-contact approach to rapid cantilever functionalization (Fig. 1).

Inkjet printing was first described for formation of self-assembled monolayers (SAMs) [3] and microcantilever functionalization [4] by the University of Basel working jointly with IBM in 2004. Cantilevers with plain view dimensions of 500 μm by 100 μm were successfully functionalized with various polymers, alkanethiols and DNA using a commercial inkjet system (MicroDrop GmbH).

In order to functionalize cantilevers with smaller dimensions, control the distribution of coating on a single cantilever and reduce the amount of cross-contamination between adjacent cantilevers, material deposition resolution and accuracy need to be increased. The sources of drop placement error must be addressed and minimized. This paper outlines the development of a high resolution inkjet system for functionalizating microcantilevers. A high performance motion control system is used to minimize substrate positioning error during on-the-fly printing. A laser-optical registration method is proposed for precisely determining the location of target cantilevers with respect to the print head. The inkjet system has enabled precise layer deposition allowing for investigation into the effect of layer distribution on cantilever transducer response and biosensor functionalization of differential cantilevers without cross-contamination.

Inkjet Functionalization System

A custom-built inkjet functionalization system (Fig. 2) has been developed to meet the strict performance criteria required for printing onto microcantilevers. Microcantilevers are fabricated with a wide variety of dimensions depending on the requirements of the application. In this research the cantilever dimensions were in the range of 100-750 μm in length, 20-100 μm in width and 0.5-1 μm in thickness. The amount of mass required to functionalize a cantilever can be as small as a few nanograms, which can be delivered in a single droplet of solution containing the func-

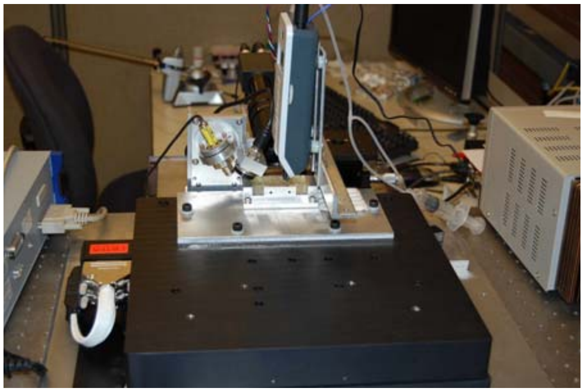

Figure 2. *Photograph of the inkjet functionalization system uniquely developed for printing functional layers on microcantilevers or other substrates with high resolution.*

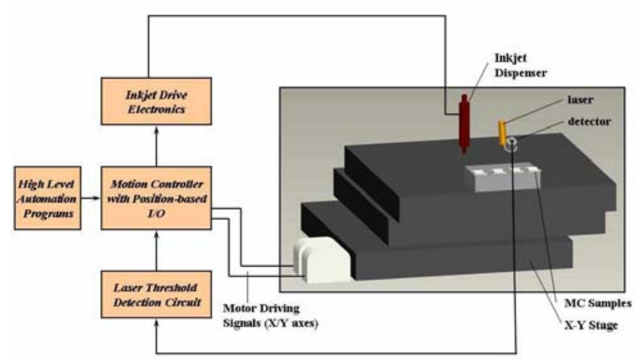

Figure 3. *The schematic diagram shows the integration of the system components. The controller is used for determination of sample positions and triggering inkjet drop ejection.*

tional material. The deposited material must be strictly controlled to insure that each drop is placed accurately.

The functionalization system has been built to accommodate two different types of dispensers. Piezoelectric squeeze-mode dispensers (MicroFab) with nozzle diameters of 30 μm or 60 μm can be used. Alternatively, the HP TIPS thermal inkjet system with up to 18 nozzles can be installed. The TIPS system produces drops that range in volume from 2 to 220 pL. Motion control of the substrate is accomplished using an X-Y stage (Anorad) driven by linear electric motors with optical encoder feedback using sin-cos interpolation to achieve 0.5 μm resolution. The system also contains a CCD camera (Sony) for viewing droplets in flight and after being deposited onto the substrate. An LED is used to control the exposure time per frame to capture snapshots of the high speed droplets while in flight. A laser-detector unit is also used for precisely locating the position of cantilevers and will be discussed in the following section.

A DSP-based controller (ACS Motion Control) is used to implement programs and automate all aspects of the system. Figure 3 displays the integration between the different subsystems. In addition to automation, the contoller also implements the control algorithm for trajectory tracking. Drop ejection is triggered by the controller based on the stage position with minimal time delay ($< 0.1 \mu sec$).

Laser-Optical Sample Registration

Using inkjet printing to functionalize microcantilevers imposes the challenge of precisely locating the samples relative to the inkjet nozzle. The use of a CCD camera to assist in locating the drop position relative to desired features has been previously reported to achieve less than 5 μm resolution [5]. Using this method, the entire substrate can be registered by calibrating to a single feature. However, for functionalization of multiple microcantilever arrays, the location of each array would need to be calibrated, which is not practical.

The laser-optical sample registration method has been developed for registration of multiple cantilever arrays using a CCD camera to calibrate to a single location. This method consists of precisely determining the location of the cantilevers and a dummy target with respect to the system. Calibrating the position of the nozzle to the dummy target results in registration of all cantilever targets at once since the relative position of the cantilevers with respect to the dummy target is determined.

Determination of the dummy and cantilever positions is accomplished by reflecting a collimated 23 μm diameter laser beam (Schafter + Kirchoff) off of the substrate while monitoring the level of reflected light through the use of a photosensitive diode. When a reflective surface such as a Silicon or Au-coated cantilever passes the laser beam, the amplitude of the reflected light surpasses a predefined threshold level indicating the edge of a target cantilever. An amplifier circuit using a Schmitt trigger converts the signal to logic on/off value which is monitored by the controller. The coordinates of the position of the stage are recorded whenever the edge of a cantilever is detected. By performing a seek routine, the positions of both the dummy target and cantilevers can be determined with a high level of accuracy. Alignment between the inkjet nozzle and all cantilevers is then known before a single drop is printed onto a cantilever.

Motion Control System

To minimize the effect of uncertainty in the drop trajectory associated with the first drop, on-the-fly printing is employed whenever possible. To implement this strategy, it is required that the substrate be positioned with high speed and accuracy. Steady-state error in point-to-point positioning can be reduced to the sensor resolution through the use of an integrator in the control algorithm. However, the position error during motion can be significantly larger and may contribute to error when printing on-the-fly. To minimize this source of drop position error significant effort was placed on development of a high performance motion control system.

Typical linear positioning systems use rotary motors combined with transmission components such as gears, belts and pulleys or lead screws. In the functionalization system, an X-Y stage using direct drive linear electric motors is used. The linear electric motors eliminate the transmission components, which are the cause of backlash, reduced speed, vibrations and non-linear fric-

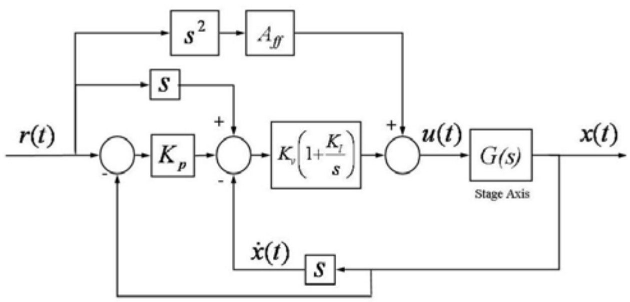

Figure 4. PIV control structure used for trajectory tracking of the X-Y stage.

tion. Epoxy core linear motors are used to further reduce the effect of force ripple. Sensor feedback resolution of 0.5 μm was obtained using optical encoders with sin-cos interpolation.

Each motion axis is controlled using a PIV (Proportional position, Integral and proportional Velocity feedback) control structure with acceleration feedforward as shown in fig. 4. After performing system identification, the controller was designed using linear-quadratic integrator (LQI) optimal control formulation [6]. A closed-loop bandwidth of approximately 100 Hz was achieved for the X and Y axes.

The performance of the closed-loop system was measured by the ability to track a high acceleration, high velocity trajectory. A 1600 mm/s^2 acceleration was used with a maximum velocity of 300 mm/s. Position and velocity versus time are shown in fig. 5 along with the error between the reference position and the actual position. It can be seen that the position error does not exceed 2 μm for the X axis and 1 μm for the Y axis.

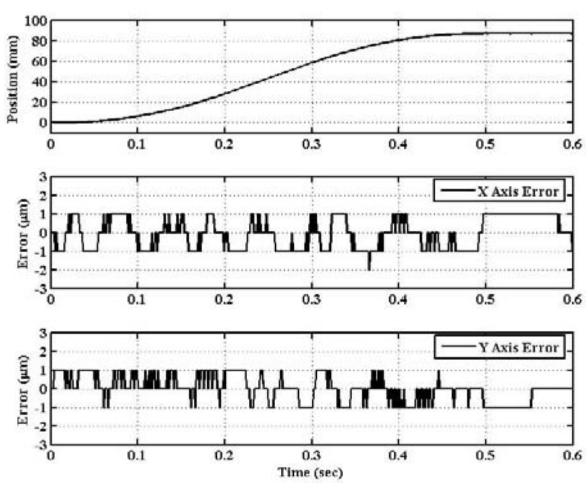

Figure 5. Axis position and tracking error for a 90 mm change in position with acceleration of 1600 mm/s^2.

Minimizing Drop Placement Error

The error due to trajectory variation in the ejected droplets is known to be the main source of error in inkjet systems. Bending in

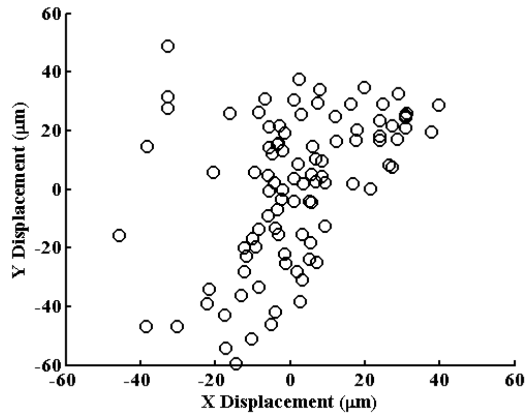

Figure 6. A large amount of drop placement variation occurred using a bipolar waveform with drop velocity of 0.4 m/s.

the trajectory can be minimized through careful filtration of fluids and tuning of the driving pulse. Variation in the drop trajectory results in deviation in drop placement from one drop to the next. The extent of drop displacement variation can be characterized using the standard deviation in radial displacement of a series of drops.

It has been found that the variation in drop placement can be reduced by tuning the driving waveform to achieve the highest initial velocity. Both unipolar and bipolar trapezoidal waveforms were used to actuate the piezoelectric dispenser. The acoustic velocity of the fluid was used to guide in selecting the pulse width of the waveform as described in [7]. Black-dyed printer ink (Canon BCI-21) was used in the experiments so that drops would be visible after being printed onto a paper substrate. The inkjet nozzle was positioned with an offset of 2 mm from the substrate to magnify the observed amount of drop displacement. Multiple series of drops were printed onto the paper with a fixed pitch of 100 μm between drop spots. The driving waveform characteristics (unipolar vs. bipolar) and ejection frequency were modified to produce stable drops under different driving conditions. The drop size and velocity were measured using the CCD camera with LED illumination for reduced exposure time.

After printing arrays of drops, high-resolution photos of the patterns were obtained using a CCD camera with calibrated pixel resolution of 6.35 $\frac{\mu m}{px}$ (4000 dpi). Using Matlab post-processing, the position of the centroid of each drop was determined. The measurements were then used to obtain the variation in drop displacement. Figure 6 shows the locations of 50 drops ejected using a bipolar pulse at 50 Hz ejection frequency. The initial velocity of these drops was 0.4 m/s and the resulting standard deviation in placement was 27.8 μm. The standard deviation was improved by using a unipolar waveform with an ejection frequency of 500 Hz as shown in Fig. 7. The initial velocity of these drops was increased to 1.9 m/s and the standard deviation in drop displacement decreased to 6.8 μm.

This experiment suggests that higher initial velocity will lead to less variation in trajectory. This may be due to the reduced effect of Brownian motion as the velocity is increased.

Drop displacement variation was also reduced by using higher ejection frequencies. Largest drop displacement fluctu-

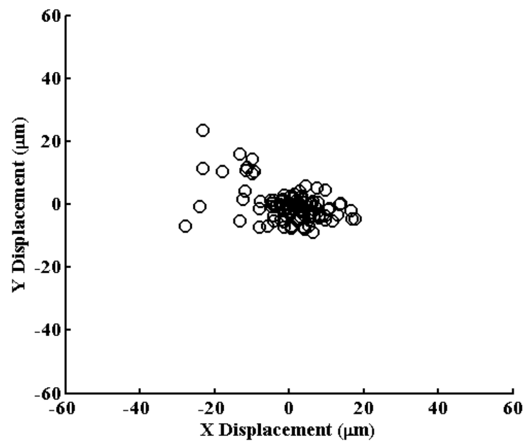

Figure 7. *Drop placement variation was reduced using a unipolar waveform that resulted in a drop velocity of 1.9 m/s.*

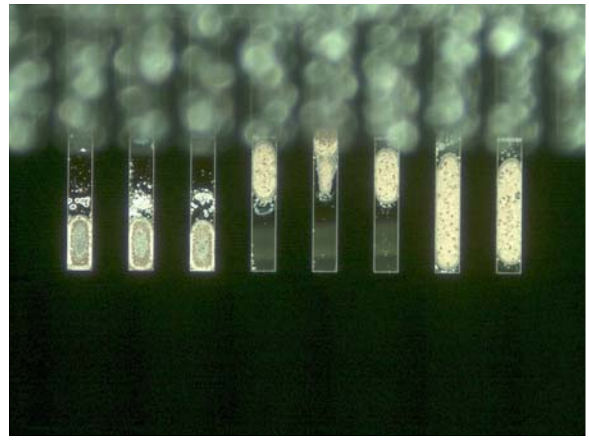

Figure 8. *Inkjet functionalization system enabled cantilevers to be arbitrarily coated over a portion of the length. PMMA has been used to partially coat individual cantilevers, 500 μm long, in a cantilever array.*

ation occurred in the first several drops in a series. This phenomenon is related to the first drop problem which is well documented in inkjet printing. Increased drop placement accuracy can be obtained by operating the dispensers at a higher frequency and reducing the duration of pauses when printing a pattern.

Effects of Layer Distribution on Microcantilever Properties

The inkjet functionalization system was used to investigate basic properties of micromechanical cantilevers by examining the effects of polymer films on the stiffness and resonance frequency. The ability to deposit precise amounts of mass at arbitrary positions on the cantilever was key to enable these experiments.

The motion of microcantilevers can be approximated by the dynamics of a simple harmonic oscillator,

$$m\frac{d^2z}{dt^2}+c\frac{dz}{dt}+k=F(t) \tag{1}$$

where m is the effective mass of the functional layer and cantilever mass, c is the apparent damping, k is the spring stiffness of the cantilever and z is the verticle displacement of the cantilever tip. Microcantilever sensors detect the presence of a target analyte through adsorption of molecules in a functional layer. This adsorption leads to a change in mass which can be detected by a shift in the resonant frequency of the cantilever. The first mode resonant frequency is determined by the equation

$$f_0=\frac{1}{2\pi}\sqrt{\frac{k}{m}} \tag{2}$$

Theory of vibration has shown that the cantilever is more sensitive to mass added near the tip of the cantilever. Additionally, mass added toward the base may increase the stiffness making changes in mass difficult to detect. Researchers have typically functionalized the entire cantilever, neglecting the change in stiffness. To examine the effect of added mass on the stiffness and resonant frequency the inkjet functionalization system has been used to deposit localized layers of mass.

Microcantilever arrays (Veeco) consisting of eight cantilevers were used with each cantilever having the dimensions of 500 μm in length, 100 μm in width and 1 μm in thickness. Both PMMA and an inkjet ink (Canon) consisting of ethylene glycol were used in this experiment. Three of the cantilevers were coated from the tip to the midpoint. Another three were coated from the middle of the cantilever to the base. The remaining two cantilevers were fully coated (fig. 8). For the partially coated cantilevers, approximately 90 ng of mass was added while 225 ng was added to the fully coated cantilevers. After coating, the spring constant and resonant frequency of each cantilever were obtained using thermal noise response.

The average spring constant and resonant frequency prior to functionalization were 0.0438 N/m and 5.01 kHz, respectively. The average stiffness of the cantilevers coated over the rear half increased to 0.0820 N/m, an 87% increase, while the measured change in stiffness was negligible, -7%, for the cantilevers coated toward the tip. Similarly, the change in resonant frequency was large for the cantilevers coated toward the tip, decreasing to an average of 2.92 kHz (-54%). While the change was insignificant for rear-coated cantilevers, decreasing by an average of 0.24 kHz (-4.4%). This study indicates that functional material added to the rear portion of the cantilever does not improve the sensitivity to shifts in the resonant frequency and will cause an increase in the cantilever stiffness.

For microcantilever sensors, high quality factor is desired to improve the confidence of resonant frequency measurements and subsequent signal-to-noise ratio. In this experiment, the correlation between the added mass and the quality factor of cantilever resonance was investigated. An increasing amount of mass from 17 to 136 ng was added to the tips of eight cantilevers in an array (fig. 9). Black ink composed of ethylene glycol was used due to the jettability of the fluid and the fact that ethylene glycol has good adhesion properties allowing for the addition of significant localized mass. After coating the cantilevers, the quality factor was obtained using the thermal noise response. Table 1 shows the quality factor of each cantilever before adding mass, the theoretically predicted and actual quality factors after adding mass. Using a simple harmonic model, the quality factor can be estimated us-

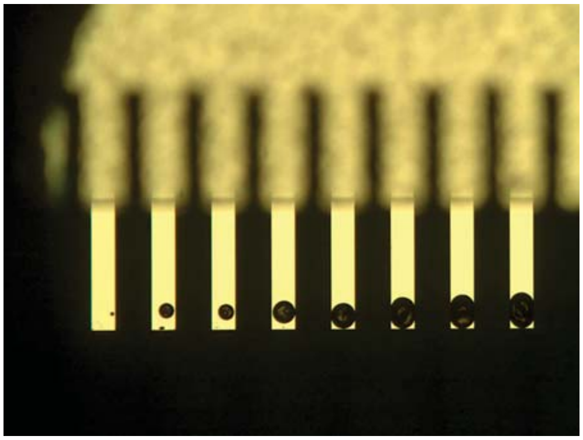

Figure 9. *Cantilevers were coated with an increasing amount of mass. The quality factor of the resonance increased as the amount of mass increased.*

ing the effective mass, stiffness and damping [8],

$$Q = \frac{\sqrt{m \cdot k}}{c}, \tag{3}$$

equation (3) shows that the quality factor should increase with the square root of the added mass if changes in the cantilever stiffness and damping can be neglected.

As predicted, the quality factor increased with the addition of mass. However the actual change in quality factor exceeded the predicted value. This study indicates that the addition of localized mass to the tips of cantilevers can be used to increase the quality factor when desired. It must be noted that additional mass will decrease the sensitivity to shifts in the resonant frequency reducing the sensitivity to analyte detection.

Table 1: Effect of localized mass on the quality factor and comparison to theory.

Added Mass (ng)	Q-factor before	Q-factor after	Q-factor predicted
17	16.0	21.3	19.6
34	15.9	24.0	21.7
51	16.4	22.7	20.4
68	16.8	30.7	25.7
85	17.0	36.3	31.0
102	16.6	47.3	34.2
119	16.5	48.1	36.0
136	17.1	47.5	38.0

[redacted]

Conclusion

A custom-built inkjet functionalization system has been presented as a method for rapidly depositing functional layers at the micro-scale. Laser-optical registration has been used as a method for precisely detecting the position of microcantilevers or other reflective substrates. Drop placement error has also been reduced

[redacted]

through improved motion control and tuning of the inkjet driving waveform. The system has been applied to several microcantilever experiments that could not be carried out using traditional functionalization methods. Polymers have been deposited with high precision to show the effect of film distribution on the stiffness and shift in resonant frequency of microcantilevers. [redacted]

Author Biography

Nicholas Post, M.S. student in the School of Mechanical Engineering at Purdue University in West Lafayette, Indiana. He received his B.S.E. in Mechanical Engineering from the University of Michigan in 2001. He worked as a design engineer at EGS Electrical Group from 2002 to 2005. His research interests include mechatronics, control of dynamic systems, and inkjet printing.

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