A Flexible Nanograting Integrated Onto Silicon Micromachines by Soft Lithographic Replica Molding and Assembly

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Abstract—We report a new elastomer-silicon hybrid photonic microelectromechanical systems device incorporating a soft lithographically patterned and assembled tunable elastic grating that can be strained by on-chip silicon microactuators at high speed. The fabrication of this optical device involves an innovative process that allows the integration of polymer and silicon structures on the microscale with the inclusion of nanometer features. The nanograting is capable of varying its grating period over 13% at actuation bandwidth of 2 kHz. The unique structure is modeled theoretically and compared to experimental results. Further experimental characterization determines the device’s ability to be used in combination with a single point detector for high speed, highly sensitive spectroscopic measurements. The spectroscopic detection of varying mult wavelength signals is demonstrated with the use of our device and a single detector. This demonstrated capability makes this particular device ideal for rapid detection of weak fluorescence in demand for several bioassay applications.

Keywords—Poly(dimethylsiloxane) (PDMS), soft lithography, spectroscopy, strain control, tunable gratings.

I. INTRODUCTION

A field with substantial growing interest is photospectroscopy within microsystems. Optical spectroscopy measures the intensity at each wavelength for a certain range of the electromagnetic spectrum, typically visible light. Along with being an important tool in chemistry and physics to identify substances by analyzing the spectral profiles, it has more recently become valuable in microfluidic applications for the detection of fluorescent markers within the flowing samples [1], [2]. Detecting multiple wavelengths correlates to achieving simultaneous assays of a sample and generating more data for scientists to analyze and derive relationships between analyzed factors [3]–[5]. The problem, however, is that the inherently small sample volumes in microsystems can lead to low intensity fluorescent emissions difficult to detect. Standard benchtop spectrometers cannot detect weak fluorescence without long integration times that hinder the rapid output desired in applications such as spectral flow cytometry [3], [6].

One possible solution that we propose is to implement a tunable grating for spectroscopic measurements. A grating is the dispersion element of spectrometers that diffracts the optical signal to detect the separate wavelengths in the spatial domain. A tunable grating can actively control the direction of diffraction and allow spectral measurements to be constructed in the time domain. A detector is needed for only a single position in space allowing the use of single point detectors, which are more sensitive and more rapid than spatial detectors such as a standard charge-coupled device (CCD) [7]. The important properties of a tunable grating for this application are the tuning range, speed, and groove density, which corresponds to spectral range, acquisition time, and spectral resolution, respectively. Speeds over 1000 measurements per second are desired for several spectroscopy applications such as rapid analysis in spectral flow cytometry [8], real-time detection of the binding, folding, and diffusion of single biomolecules, including proteins, DNA, RNA, and enzymes, both inside and outside a cell [9], and detection of inherently fast biological processes such as calcium propagation [10]. To tune a grating fast enough for kilohertz speeds, microelectromechanical systems (MEMS) actuators are a practical solution. Several MEMS tunable gratings have been developed for varying applications, but all lack a suitable range, speed, groove density, or optical flatness [11]–[14]. To overcome the limits of other MEMS devices, we have developed a process to create hybrid MEMS devices that incorporate silicon with elastomer structures on the micrometer scale with nanometer feature sizes.

Our hybrid microsystem consists of a passive polymer microstructure functionalized as a tunable optical microcomponent by active MEMS actuators, i.e., electrostatic comb drives. The polymer material selected in our paper is cross-linked poly(dimethylsiloxane) (PDMS) elastomer, which is popular for rapid prototyping of microfluidic devices based on micro/nanoscale replica molding known as soft lithography [15], [16]. Some transducer studies have focused on the elastic...
property of PDMS for high-strain microfluidics applications including valves [17], fluidic pressure sensors [18], and tunable nanochannels [19]. The optical transparency of PDMS yields good transmission of visible light, which permits on-chip optofluidic fluorescence detection in microfluidic systems [20], [21]. Our microsystem combines PDMS and silicon in selected on-chip places with high precision to optimize the advantages of both materials.

The key component of our device is a PDMS microbridge connected on one side to a silicon MEMS actuator, with the other side connected to a fixed silicon anchor. The microbridge contains a nanoimprinted grating pattern on the top surface, creating an effective optical transmission grating. Applying a voltage difference across the comb drive generates a force that mechanically stretches the microbridge, altering the grating spacing on the top surface. The grating can elastically stretch and tune the grating period at the high speeds of the MEMS actuator. By utilizing the hybrid properties of the device, our tunable grating has the desired large range, fast actuation speed, and high groove density. We show that the ability to dynamically change the spacing of the soft grating can lead to a new compact photospectroscopic setting that permits high-speed, high-sensitivity spectral acquisitions using a single-point detector such as a photomultiplier tube (PMT).

We first introduced the concept of integrating PDMS microstructure with nanoscale patterns into a silicon MEMS actuator in [22]–[24]. Based on these previous studies, this paper presents a new design of the hybrid MEMS device, a model for its mechanical behavior, data showing its long-term reliability, and in-depth characterization of its spectroscopic capabilities.

II. DEVICE DESIGN AND MODELING

For the tunable grating to be used in the aforementioned applications, the device must be capable of producing high strain of the microbridge at fast speeds. A large strain value of at least 10% will lead to a significant tunable range of wavelengths in the single point detector setup. A MEMS actuator with large displacement, high force, and high speed is required. An electrostatic comb drive was selected as the simplest and best actuator that fits our requirements. A comb drive’s actuation speed is limited by its spring stiffness and mass, which make speeds over 1 kHz easily possible for an appropriately designed system. To calculate the forces needed from our actuator, we modeled our device as a combination of linear and nonlinear springs.

The force generated by the actuator is resisted by a combination of the silicon springs and the elastic stretching of the PDMS bridge. The silicon springs are pairs of single fixed beams chosen for their high stiffness in the lateral direction to prevent snap-in of the comb drive. Another reason for the single beam springs is their durability during fabrication, as opposed to folded spring designs which seem to have a higher probability of breaking. A downside of the spring design is extensional axial forces in the beam which lead to nonlinearity for larger displacements. The displacement-force relationship is calculated by modeling them as a clamped-clamped beam with a force on the center.

The derivation for this model can be found in [25]. The relationship between force and displacement for a single beam is given by the equations

\[
P = \frac{8EI(2I A)^{1/2}}{L^3} u^3 \times \left( \frac{3}{2} - \frac{1}{2} \tanh^2 u - \frac{3 \tanh u}{2} \right)^{-1/2}
\]

\[
\delta = 2 \left( \frac{2I A}{E} \right)^{1/2} \left( u - \tanh u \right)
\]

\[
\times \left( \frac{3}{2} - \frac{1}{2} \tanh^2 u - \frac{3 \tanh u}{2} \right)^{-1/2}
\]

for

\[
u = \frac{L}{2} \sqrt{\frac{S}{EI}}
\]

where \(P\) is applied force, \(\delta\) is the displacement, \(E\) is Young’s Modulus of silicon, \(I\) is moment of inertia of the beam, \(A\) is cross-sectional area of the beam, \(L\) is the length of the beam, and \(S\) is the internal axial force considered constant along the bar. With \(S\) unknown, (1) and (2) are solved simultaneously for varying values of \(u\) to determine the force-displacement relationship.

Along with the silicon springs, the stretching of the PDMS bridge also requires force from the actuators. For this model the PDMS is considered linearly elastic and the force-displacement relationship is given by

\[
P_P = \frac{A_P E_P d}{L_P}
\]

where \(P_P\) is the applied force on the PDMS bridge, \(A_P\) is the cross-sectional area of the bridge, \(E_P\) is the Young’s Modulus of PDMS, \(L_P\) is the bridge length, and \(d\) is the displacement of the bridge end connected to the actuator. The total force needed for displacing the system is the addition of the force needed for the silicon springs and the PDMS bridge. It can be calculated by the equation

\[
P_{total} = CP(\delta) + P_P(\delta)
\]

where \(C\) is the number of silicon springs which are all acting in parallel. However, one problem is that \(d\) and \(\delta\) are not the same for the system due to the internal stress of the PDMS bridge. When the PDMS is cured, it retains a tensile stress. Once the bridge is released and suspended during fabrication, the volume shrinks, and the length will decrease approximately 2.5% [26]. This internal stress pulls the comb drive in the opposite direction of actuation and the silicon springs have an initial displacement behind their rest position, illustrated in Fig. 1(a). The force-displacement relationship of the silicon springs in the opposite direction is the negative of the forward relationship due to symmetry. With actuation the comb drives displace and stretch the grating, illustrated in Fig. 1(b). The force-displacement relationships of the silicon springs, PDMS...
bridge, and combined system are plotted in Fig. 1(c) with the offset included, where deflection is measured from the position of the silicon springs being perfectly straight. The graph shows that the forces needed for displacement are mainly due to the PDMS bridge at small displacements and dominated by the silicon springs at larger displacements. The comb drive is then designed to be able to generate these relatively high forces for large displacement of the total system and also retain the highest actuation speed possible determined by the resonant frequency.

Another important aspect in the design of this device is the grating pattern on the PDMS microbridge. The pattern is small enough to not affect the PDMS strain profile, thus allowing its shape to uniformly vary with the stretched PDMS microbridge. In general, grating efficiency—the ratio of the power of incident light to the spectral order and is highly affected by the grating profile. The nominal profile of our grating is determined by the grating master mold that we copy using soft lithography (discussed further in Fabrication). The master mold grating available for our process is a square shape profile with 700 nm period. GSolver software (Grating Solver Development Company, Allen, Texas) is used to determine the efficiency of the grating. Fig. 2(a) shows the results of the simulation showing a first-order efficiency of 13%–23% for our wavelength range of interest. As the device is actuating, the grating period is also changing, and simulations are run to determine how this change affects efficiency. Fig. 2(b) shows minimal change in efficiency for a wavelength of 633 nm as the grating period is shifting between 700 and 900 nm. It indicates that the efficiency at a given wavelength virtually stays constant during the strain-tuning operation of our device. The difference in efficiency for varying wavelengths is later calibrated into our spectral measurements.

III. FABRICATION

Our PDMS-silicon hybrid microsystem construction method employs a multistep process that fabricates PDMS and silicon structures independently before assembly. The process involves three major steps: 1) formation of the PDMS structures through soft lithography and sandwich micromolding; 2) fabrication of the MEMS structures through standard silicon bulk micromachining; and 3) surface tension-assisted precise alignment/bonding and release of the molded PDMS layer onto the silicon structures. These steps meet the following outstanding technical requirements: 1) the PDMS micromolding process needs to be compatible with silicon MEMS fabrication; 2) the entire integration processes need to be performed at low temperature to maintain their compatibility with CMOS technology; and 3) the PDMS-silicon hybrid microstructures need to be assembled in high precision (an alignment error < ±2 µm) without debonding and delamination.

Fig. 3 shows the fabrication process of our device. The process starts by first fabricating the top and bottom molds to form the PDMS into the desired microstructure shape. For the top mold in Fig. 3(a), photore sist is spun onto a transparent glass wafer and then cut into 2 cm square die pieces. The photore sist is nanoimprinted at 150 °C and 4500 kPa using a silicon master mold of periodical surface nanopatterns fabricated by electron beam lithography, which can be shaped into an array of gratings, pillars, circular grooves, or any other arbitrary structures. The bottom mold in Fig. 3(b) is made by a two step deep-reactive ion etch (DRIE) using ultraviolet lithography for patterning. A thin carbon fluoride (CF$_2$) coating is applied to prevent the PDMS from sticking to the bottom mold.

The top and bottom molds are then configured to create the PDMS microstructures as shown in Fig. 3(c). PDMS is mixed using the Sylgard 184 silicone elastomer kit (Dow Corning Corporation, 10:1 base-curing agent ratio), and a small drop is placed onto the bottom mold. The top and bottom molds are sandwiched together, and the PDMS is cured at 150 °C and 4500 kPa for 45 min. As the PDMS cures, it conforms to the shapes of the two molds, taking the form of both the microstructure and the nanoscale surface pattern. Once cured, the bottom mold is removed, and the molded PDMS stays attached to the top mold. A reactive ion etch of the top mold removes any residual PDMS.

On a separate silicon-on-insulator (SOI) wafer, the silicon MEMS device is fabricated during the fabrication step in Fig. 3(d). The back and front of the wafer are patterned with photore sist, and the frontside is etched with DRIE down to the oxide layer. The created MEMS structures are 50 µm thick and
made from single crystal silicon. The wafer is cut into 2 cm square dies with approximately 40 MEMS structures on each die. The SOI wafer and the PDMS microstructure attached onto the top mold are next combined to create the PDMS-silicon hybrid microstructures as shown in Fig. 3(e). Both pieces are treated with oxygen plasma to activate the surfaces and promote the permanent bonding of PDMS and silicon. A drop of water is placed on the SOI die, and the top mold is positioned on top. The 3-D PDMS structure is designed to fit into position holes of the silicon MEMS. The water surface tension assists with alignment until the PDMS falls into the correct location of the MEMS device. Fig. 4 displays a cross-sectional illustration of the alignment and bonding steps. The alignment accuracy for this step is approximately 2 μm. After water evaporation, the PDMS is now permanently bonded to the silicon by silicon-oxygen covalent bonding [27]. The back side of the SOI wafer is etched through and the oxide layer is removed to suspend the MEMS device. Finally, the photoresist of the top mold is dissolved to release the PDMS microstructures from the top mold. The final device has suspended silicon MEMS structures with PDMS attachments and contains a submicrometer pattern on the PDMS top surface.

Fig. 5(a)–(c) show the SEM images of the PDMS microbridge connected on one side to a silicon comb drive with the other side connected to a fixed silicon. The microbridge contains a nanoimprinted grating pattern on the top surface as shown in Fig. 5(d). The entire bridge is 200 μm long, 100 μm wide and 15 μm thick, and the grating pattern has a period of 700 nm. The devices are next experimentally characterized and tested as high speed tunable gratings.
Fig. 5. SEM images of the device. (a) Image of the entire MEMS device. (b) Closer view of the suspended PDMS grating bridge connected to the silicon comb drive. (c) View of the PDMS-Si attachment interface. (d) Zoomed image of the edge of the PDMS bridge showing the nanograting pattern on its surface.

Fig. 6. Images from the bond reliability test. (a) Test structure at its initial state. (b) Test structure with the PDMS bridge stretched 40% using mechanical probes.

Fig. 7. Optical setup for device characterization. The PSD measures the movement of the laser spot diffracted at the first order. This data is used to calculate the change in diffraction angle and the shift in grating period.

IV. EXPERIMENTAL RESULTS AND DISCUSSIONS

A. Bond Reliability

After fabrication, the first experiment was to test the reliability of the silicon-PDMS bond. A PDMS microbridge was attached to two suspended silicon structures that were pushed apart using probe tips. Optical images were taken, and the PDMS bridge was measured to stretch over 40% without the silicon-PDMS bond breaking as seen in Fig. 6. The corresponding stretching force was on the order of several millinewtons, which is approximately four times larger than the actuation force used in our normal conditions. The PDMS was then stretched to failure, and the bridge itself ripped before the silicon-PDMS bond was broken. The strong bond also sustained the stretching actuation of the PDMS microbridge by the MEMS actuator over 100 million cycles.

B. Tunable Grating

Since there are few data available for the mechanical behavior of cross-linked PDMS at the size scale of our microsystem, we first set out to study its response to microscale actuation. To test the device, we implemented it into an optical experimental setup in Fig. 7, which directed laser light
to pass through the transmission grating from the underside of the device. A position sensitive diode (PSD) (C4674, Hamamatsu Photonics) tracked the changing first-order diffraction angle of a monochromatic source and hence the change in grating period. The calculations for this characterization revolve around basic geometry and the transition grating equation

\[ m \lambda = a (\sin \theta + \sin \beta) \]  

(6)

where \( m \) is the diffraction order, \( \lambda \) is the incident light wavelength, \( a \) is the grating period, \( \theta \) is the diffraction angle, and \( \beta \) is the incident angle. At the first diffraction order \((m = 1)\), with the incident angle at the normal of the grating \((\beta = 0)\), (6) is simplified to

\[ \lambda = a \sin \theta. \]  

(7)

Measuring the variations in \( \theta \) for normally incident monochromatic light through the grating allows us to determine the variation in \( a \) from (7).

Using the PSD experimental setup with a 632.8 nm HeNe laser (25-LHP-991-249, Melles Griot), we used the change in \( a \) to measure the PDMS engineering strain at a quasi-static condition while applying a sine wave voltage of 0–160 V at 0.1 Hz onto the silicon comb drives. The PDMS grating varied 13% from 700 to 791 nm over the voltage cycle. This measurement is compared to our theoretical force-displacement model. The voltage needed to generate the actuator force is calculated by the comb drive equation [28]

\[ V = \sqrt{\frac{P_{\text{total}} g}{N \varepsilon h}} \]  

(8)

where \( g \) is the gap between the comb drive teeth, \( N \) is the number of teeth, \( \varepsilon \) is the dielectric constant, and \( h \) is the height of the comb drive. The theoretical model generally agrees with the strain profile of the experimental results as seen in Fig. 8. Therefore, it serves as a good instrument to understand the nonlinear shape of the change in the grating period profile. Discrepancies, however, are likely due to the PDMS strain not acting linearly with force. Hysteresis is also evident as the profile differs for the rise and fall of the strain cycle.

The PDMS grating microbridge was next held at a constant force over time to see if the material displayed creep behavior. A voltage of 150 V was applied and held constant, and the strain of the PDMS was optically measured by tracking the change in first-order diffraction. The strain of the PDMS microbridge was plotted over time in Fig. 9 and showed change of strain of \( 3 \times 10^{-3} \) after 20 min. This change in strain correlates to a grating period drift of 2.1 nm. It is clear that we must account for the changes in the PDMS response over time.

We also measure the PDMS strain during the rise and fall of an actuation cycle of a 20–160 V sine wave at various frequencies. The frequencies 20, 200, and 2000 Hz were measured and compared as seen in Fig. 10 to study the dynamic response of our device to different strain rates. For higher frequencies the maximum strain tends to drop while the asymmetry of the rise and fall cycle becomes more pronounced. With all three frequencies below the calculated resonant frequency of 5.54 kHz for the MEMS device and the electrostatic force being dependent only on voltage amplitude, we believe that the change in the strain cycle with the frequency should be caused by the PDMS’s strain rate-dependent response [29].
The device was also tested for drift of the PDMS strain over repeated cycling. The device actuation voltage was a 20–160 V sine wave at 200 Hz. The rise and fall strain cycle was measured every 20 min for an hour, with the first and last measurement graphed in Fig. 11. The maximum deviation between the cycles and the initial cycle was plotted over time as shown in Fig. 11 inset graph. The PDMS strain varied up to $1.7 \times 10^{-3}$ after 1 h of cyclic straining.

Through our experimentation we have observed a deviation of our device’s behavior from the theoretical prediction in Fig. 8. This is a result of the nonlinear stress–strain behavior of the PDMS microbridge. In particular, elastomers are commonly known to exhibit signs of hysteresis, cyclic softening, and rate-dependence in their mechanical behavior [30]–[32]. Our above data indicate these characteristics for the microscale PDMS structure, thus warranting much care for practical operation of the device. Here, the PDMS response to cyclic loading is difficult to predict as a function of voltage amplitude and frequency using the theoretical model. Therefore, we determine the grating period of the PDMS structure as a function of the location within a full cycle at a given voltage amplitude and frequency directly from the experimental data for spectroscopic measurements using our device. The very repeatable and predictable dynamic response of the PDMS structure under a fixed condition makes this approach valid. To eliminate errors resulting from the creep or drift of the PDMS over time, a quick calibration measurement is taken every 10 min using a monochromatic light source and the PSD. This calibration measurement reduces the error in the spectral measurements to less than 1 nm. While the ultimate lifetime of the PDMS has not been experimentally measured, one device has seen approximately 50 h of use and $3.6 \times 10^5$ cycles and is still accurate after calibration.

C. Spectroscopy Measurements

Following these device characterization procedures, we demonstrated high-speed spectral acquisitions of multiwavelength optical signals. A voltage actuation at 2 kHz was applied to the actuators. First, a calibration measurement was taken to measure the grating period change in time using the PSD. The PSD detector was then replaced with a PMT and a slit covering the opening as seen in Fig. 12. To simulate an unknown multil wavelength signal, red and yellow ($\lambda = 593.5$ nm) (Rigel-2, Laserglow Technologies) lasers were both incident onto the underside of the transmission grating. The red laser was also pulsed at 1 kHz using an optical chopper (300CD, Scitec Instruments). This pulsing of the red laser serves as a good simulation of the emissions of fluorescent tags flowing through a microfluidic channel at a rapid rate that are optically focused using a microscope objective lens. The PMT was placed at a known angle, and the changing wavelength incident onto the detector slit is calculated using (7) and the PSD data as seen in Fig. 13(a). The PMT detection signal plotted in Fig. 13(b) includes peaks corresponding to the two laser inputs as the tunable grating sweeps a wavelength range over the detector. This signal was directly compared to the incident wavelength data for the same section of the actuation cycle to determine the intensity at each wavelength. The wavelength intensities were plotted to give the spectral acquisition measurements seen in the four graphs of Fig. 13(c).

From this figure we can see that this setup can take spectral measurements at high speeds. The first measurement taken from 0–0.25 ms timeframe makes a spectral measurement while the red laser is off and a peak only occurs at 593 nm for the yellow laser. The second measurement taken from 0.5–0.75 ms timeframe makes a spectral measurement while the red laser is on and generates two peaks at 593 and 633 nm corresponding to the two laser inputs. The third and fourth measurements again catch the red laser at its OFF- and ON-state, respectively. This data shows that spectral measurement with only one single point detector can be taken within a 250 μs timeframe twice every millisecond.

The advantage of this setup is that the spectral measurements are fast, and also the single point detector can be extremely sensitive. Detectors such as a PMT are much more sensitive than a standard CCD array and are the optimal detectors for weak signal detection. Our recent study [33] has carefully characterized a minimum detectable signal power on the order of a few picowatts in this spectroscopy method. Also, discussed in this paper is the spectral resolution, where it is determined that monochromatic peaks are distinguishable if they differ by 5–9 nm in wavelength for the studied spectral range of $\lambda = 585$ to 645 nm. While a commercial benchtop spectrometer maintains a larger range and higher resolution, our simple setup has a better combination of speed and sensitivity, warranting its use for many of the applications mentioned above.

V. CONCLUSION

In summary, the developed innovative hybrid microsystem demonstrates the new possibilities from the on-chip integration of elastomeric polymer (PDMS) onto silicon by soft lithographic replica molding and assembly. The hybrid microdevice incorporating a soft polymer nanograting structure is shown to be excellent at easily testing the polymer’s mechanical response for a large variety of stress conditions and serves as a vehicle.
Fig. 12. Second experimental setup for testing spectroscopy capabilities. Red and yellow lasers are combined with a beam splitter to create a multiwavelength signal. A PMT covered by a slit replaces the PSD.

Fig. 13. Spectroscopy measurements and the corresponding data taken with our device. (a) Wavelength incident onto the PMT slit calculated from the PSD data. (b) PMT detection for the two-wavelength signal as the device is being actuated. (c) Spectroscopy plots taken by comparing PMT intensity to the incident wavelength for the range between 585 and 645 nm. Plots accurately show two peaks at the corresponding laser wavelengths. The plots for different times capture the ON/OFF-states of the red laser pulsed at 1 kHz.

for microscale material characterization. The integrated PDMS structure can yield a high level of microscale actuated strain (> 10%) at large actuation bandwidth (2 kHz). Combining the elasticity, transparency, and soft lithographic nanopatterning of PDMS with silicon MEMS technology, our on-chip hybrid material integration approach leads to the development of a new photospectroscopic technique with both a high detection limit and a short time window (250 µs). With the ability to capture the time-varying spectral information of an extremely weak optical signal using a single-point detector, the demonstrated photospectroscopic technique may be applied to quantitative studies of dynamic biological phenomena that employ quantum dot flow cytometry [3], [4], [6], spectrophotometry [34], and even single molecule fluorescence spectroscopy [7], [35] within a microfluidic setting. To achieve this, our future work will integrate the hybrid MEMS tunable grating device in the optics of a fluorescence microscope, which can collect and collimate the fluorescent emissions from targeted molecules in a microfluidic channel.

REFERENCES


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