High-speed deformation of soft lithographic nanograting patterns for ultrasensitive optical spectroscopy

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We demonstrate a spectroscopy technique that implements a high-speed tunable grating to take spectroscopy measurements with a single, extremely sensitive photodetector. The tunable grating consists of a transparent elastomer microbridge soft lithographically patterned and assembled onto silicon microactuators. We show the ability to track the optical spectrum of time-varying multiwavelength signals with a 500 μs time resolution and sensitivity capable of detecting optical powers near 36 pW. Such a level of sensitivity is suitable for detecting the spectral fluorescence of low concentration dyes, microbeads, or quantum dots. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2842415]

Optical spectroscopy has become valuable in microsystem applications for the simultaneous detection of multiple fluorescent markers in biological samples.[1,2] More studies are finding that wavelength discrimination of multiple fluorescent color signatures is essential for multiplexed biological sensing, imaging, and diagnostics.[3–5] Several microsystems have been designed for this task and implement photodiode arrays[6,7] or charge-coupled device cameras[8–11] to detect a range of wavelengths needed for spectral measurements. One problem, however, is that the inherently small sample volumes in microsystems can lead to low intensity fluorescent emissions difficult to detect.

Our work describes an approach to spectroscopy that allows for high speed and exceptionally sensitive spectral measurements with minimal optical equipment requirements. With the application of an innovative tunable grating through microelectromechanical system (MEMS) technology, spectral measurements can be taken in a submillisecond time window with the sensitivity of a single point detector such as a photomultiplier tube (PMT).

The devised tunable grating seen in Fig. 1 consists of an elastic microbridge with a nanoimprinted grating pattern on the top surface. The microbridge is fabricated from the transparent polymer polydimethylsiloxane (PDMS) and attached to MEMS silicon comb drive electrostatic actuators. Applying a voltage difference across the comb drive generates a force that mechanically stretches the microbridge, altering the grating spacing on the top surface. Fabrication of the device involves soft lithography of a nanoimprinted grating and silicon bulk micromachining.[12] The grating period elastically stretches from 700 to 780 nm for a maximum strain and tunability of over 11%. The MEMS device is a vast improvement from our original concept device[13] that lacked an appreciable strain range and dynamic measurement capabilities.

We implemented the device into an optical experimental setup (Fig. 2) capable of directing laser light to pass through the grating at various wavelengths, intensities, and frequencies. A voltage actuation of 20–160 V at 2 kHz was applied to the MEMS actuators. In our first experiment (Fig. 2, setup 1), a position sensitive diode (C4674, Hamamatsu Photonics) tracked the changing first order diffraction angle of a monochromatic source and hence the change in grating period, plotted in Fig. 3(a). With the tunable grating period characterized, spectroscopic measurements were subsequently taken by placing a highly sensitive detector at a specific diffraction angle and comparing the detector's measurements with the wavelength incident onto the detector's slit opening. A PMT (H5784-20, Hamamatsu Photonics) was implemented as the highly sensitive detector. The incident wavelength onto the PMT during device actuation was calculated with the grating period data and standard grating equation.[13] To simulate a collimated multiwavelength signal with an unknown spectrum, red ($\lambda = 632.8$ nm, 25-LHP-991-249, Melles Griot) and yellow ($\lambda = 593.5$ nm, Rigel-2, Laserglow Technologies) lasers were both incident onto the underside of

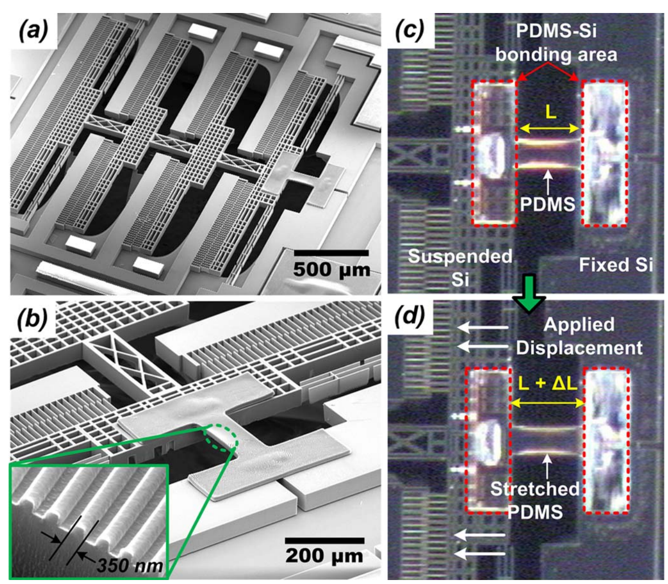

FIG. 1. (Color online) Images of the MEMS tunable grating. (a) SEM image of entire MEMS device. (b) A closer view of the elastic PDMS bridge attached to the silicon comb drive. The inset is a magnified view of the grating pattern on the bridge. (c) Optical image of PDMS bridge at initial position. (d) Image of the bridge strained by MEMS actuator.

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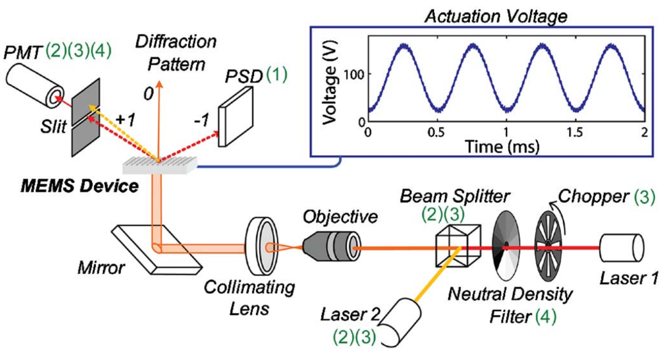

FIG. 2. (Color online) The test bed to conduct four separate optical experiments: setup (1) for dynamic response measurement setup, setup (2) for spectral acquisition measurement setup, setup (3) for variable signal measurement setup, and setup (4) for maximum sensitivity measurement.

the transmission grating (Fig. 2, setup 2). The PMT detection signal [Fig. 3(b)] was directly compared to the incident wavelength data to determine the intensity at each wavelength. The wavelength intensities were plotted [Fig. 3(c)] to give the spectral acquisition measurement within that particular 250 μs time frame.

To demonstrate the speed of the spectral measurements, the red laser was pulsed at 1 kHz using an optical chopper (300CD, Scitec Instruments) while the yellow laser remained constant (Fig. 2, setup 3). Figure 4(a) shows the input signal of the two lasers measured separately with a photodiode. The spectral acquisitions were taken twice every millisecond and plotted versus time. The spectral measurements were fast

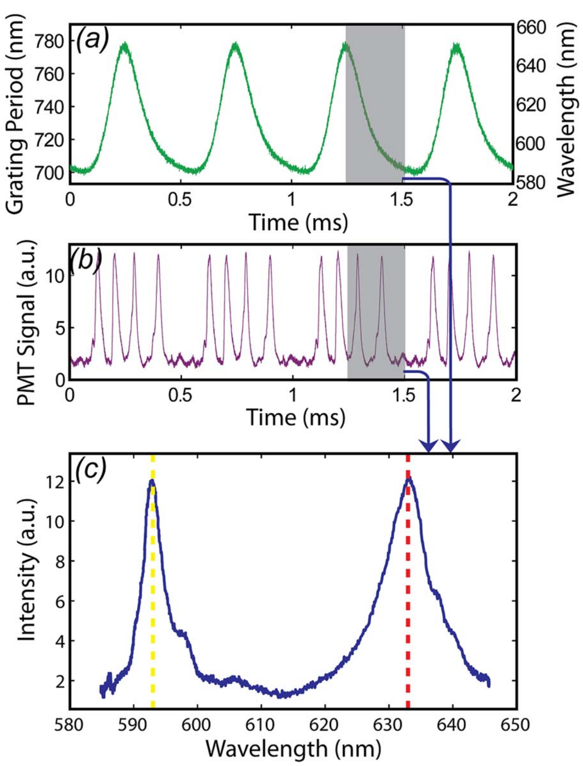

FIG. 3. (Color online) Results from the change in grating period and spectroscopy measurements. (a) The time variation of the grating period calculated from the change in diffraction angle. Right y axis shows the calculated incident wavelength onto the PMT detector. (b) PMT detection as the device is being actuated. (c) A spectral acquisition taken with one sweep of the device for the two-wavelength signal. Dotted lines mark the wavelengths of lasers being detected.

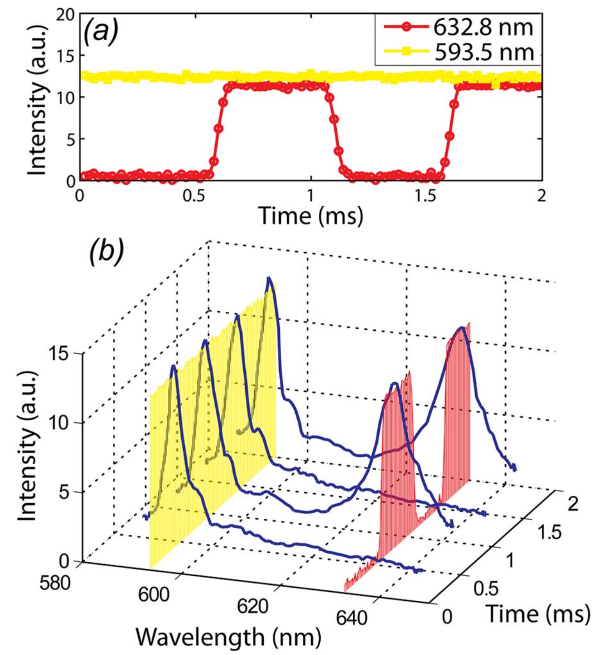

FIG. 4. (Color online) Spectral measurements for multiwavelength signal inputs varying with time. (a) Input signals of the red and yellow lasers measured separately. Yellow is constant while red is pulsed at 1 kHz. (b) Spectral acquisition measurements of input signal plotted versus time.

enough to capture the real-time switching of the red laser [Fig. 4(b)]. As the laser was pulsed, each of the lasers' on/off states was still detectable.

For these spectral measurements, the resolving power is calculated as $R=\lambda/\Delta\lambda$, where $\Delta\lambda$ is the full width half maximum taken at wavelength λ, and it is assumed that the laser source is perfectly monochromatic. From our results we obtained a R of 119 at 593 nm and 79 at 633 nm. The difference in resolution may be due to the grating tuning, which changes the number of grating grooves the incident beam diameter passes through. For the lower R value of 79, monochromatic peaks in the visible spectrum are resolvable if their wavelengths differ by approximately 5–9 nm. The theoretical spectral resolution limit of $R=mN$, where m is the order of diffraction and N is the number of grating grooves, is 285.7 for our grating. The current optical setup is purposely kept as simple as possible, but to increase our spectral resolution toward this limit, a focusing element can be inserted after the grating.[14]

To prove the high sensitivity, we calculated and measured the minimum detectable signal (MDS). With the PMT's gain at maximum, the minimum detectable output is determined by the mean plus one standard deviation of the PMT output voltage noise floor,[15] which includes dark counts of the PMT, stray light, and other noise sources of the system. The radiant flux that would produce this output value is calculated using the PMT sensitivity and grating efficiency. According to specification data, the PMT sensitivity at the maximum gain is 78 V/nW. Using GSOLVER software (Grating Solver Development Co.), we determined the theoretical grating efficiency to be 13%–16% for the first order diffraction for the 595–655 nm wavelength range of interest in this experiment, with negligible variation as the grating period changes from 700 to 780 nm. The efficiency of the grating was multiplied by the PMT sensitivity and the minimum detectable voltage output to calculate a MDS of

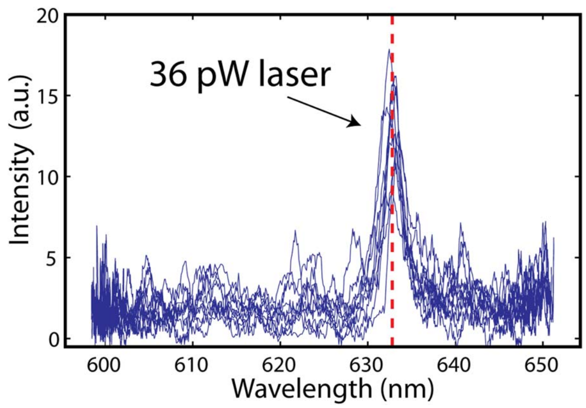

FIG. 5. (Color online) Results from the maximum sensitivity measurements. The dotted line indicates the detected laser wavelength at 632.8 nm.

6.49 pW. To demonstrate a detectable signal close to this power level, we dramatically reduced the red laser's intensity using neutral density filters (NT54-082, Edmund Optics) and took spectral measurements with the red laser peaks just observable (Fig. 2, setup 4). Figure 5 graphs ten spectral sweeps and shows the red laser peak clearly identifiable in all 10, with the signal average at the laser wavelength being 7.6 dB higher than the noise level. An optical power meter (PDA-750, Terahertz Technologies Inc.) was used to measure the power of the incident laser signal before the grating. A value of 36 pW was measured, which is higher than the calculated MDS value but reaches the lowest level of the incident signal power practically measurable with the power meter. This level of sensitivity is difficult to achieve without the use of a sensitive single point detector or very long integration times of pixel based detectors.[16] In addition, the sensitivity can further be increased by imprinting a grating profile with a higher grating efficiency.

In summary, the innovative hybrid microsystem developed demonstrates the possibilities involving high speed, highly sensitive optical spectroscopy. The hybrid microdevice incorporating a soft polymer nano grating structure can yield a high level of microscale actuated strain ($>11\%$) at large actuation bandwidth (2 kHz). Our on-chip hybrid material integration approach leads to the development of a photospectroscopic technique for detecting collimated light with both a high detection limit ($\sim$36 pW) and a short time resolution (500 μs). With a future optical setup to capture and collimate the time-varying spectral information of an extremely weak fluorescent source, the demonstrated photospectroscopic technique holds the promise to enable quantitative studies involving the spectral acquisition of very weak single molecular level photoemissions in biological systems.

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