Enhancing Light-Induced Thermal Actuation with Gold Nanoplates

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ABSTRACT

Development of microscale actuating technologies has been critical for interacting with elements at the cellular level. Small-scale actuators and switches have potential in areas such as microscale pumping and particle manipulation. Thermal actuation has been combined with unique geometries to create large deflections with high force relative to electrostatically driven systems. However, many thermally based techniques require a physical connection for power and operate outside the temperature range conducive for biological studies and medical applications. The work presented here describes the theory, design, and experimental response of an out-of-plane microactuator that reacts with enhanced absorbtion of near-infrared light by patterning a wavelength-specific absorbent gold nanoplate (GNPlate) film onto the microstructure. Near IR wavelength light is able to harmlessly permeate living tissue, and high stress mismatch in the bilayer geometry allows for large actuation under biologically acceptable conditions.

Keywords: microactuators, thermal actuation, gold nanoplates, near infrared, plasmonic heating

1 INTRODUCTION

Bioengineering has benefitted greatly from advances in microelectromechanical systems (MEMS). Small-scale actuators and switches have potential in areas such as microscale pumping and cell manipulation. Powering these micro devices is often a challenge because of the invasive wiring needed. Tissue-permeant near infrared (nIR) light has been used in theraputic applications [1, 2] and provides a noninvasive energy transfer method for heating gold GNPlate with enhanced nIR absorbtion for cancer cell ablation. Depositing these particles onto a MEMS device could provide new optically induced functionality if the absorption behavior remains the same.

The ability to use GNPlate-aided optical absorbtion to provide thermal energy for "pop-up" structures [3] has not been previously investigated. The most common MEMS heating mechanism is direct current (Joule) heating, but this requires wiring leads to the active region which is undesirable in biomedical applications. An optically induced temperature gradient using wavelength-selective printable or spinnable coatings would provide a versatile method of wireless and non-invasive thermal actuation. This project aims to provide fundamental understanding of the particle and surface interaction that is required for developing bioengineering applications based on a hybrid of nIR resonant gold GNPlates and MEMS structures.

For this article, the pop-up structure is fabricated from a patterned metal-oxide bilayer.The material strain is created during fabrication and is heavily temperature dependent. As the temperature increases, the material strain is reduced and the curved structure will lie flat in the plane. In contrast to thermal actuating principles that require wired conductive components for joule heating, the devices shown here are wirelessly powered by nIR light by patterning a wavelength-specific absorbent GNPlate film onto the microstructure.

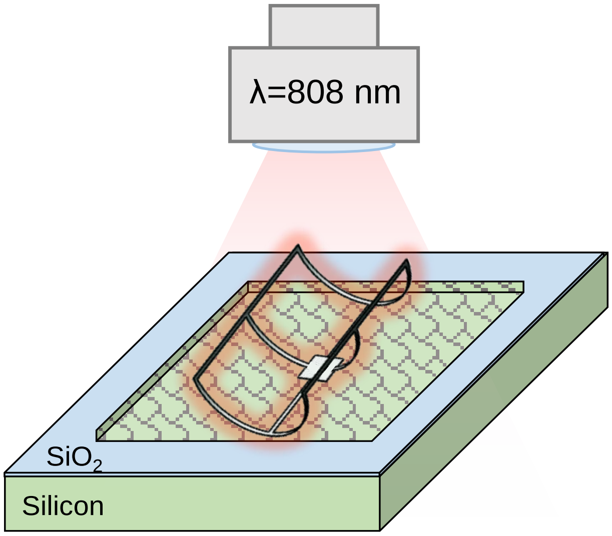

Figure 1: Illustration of the system being investigated in this experiment.

A previous investigation showed that a polymer film with suspended gold nanoparticles will provide enhanced NIR absorption compared to a bare platinum surface at a single wavelength [3]. A following study presented surface heating trials with two wavelengths (808nm and 915nm) of tuned GNPlates in order to characterize and compare the heating of matching wavelength GNPlates, non-matched GNPlates, and bare platinum surfaces [4]. The work presented here integrates this heating technology with thermally actuated pop-up MEMS structures, leading to the possiblity of wirelessly applying a mechanical stimulus at the cellular level.

2 FABRICATION

Composite structures with bistable geometry are the mechanical basis for this work. Strain between layers of SiO_2 and platinum is responsible for the out-of-plane orientation and rapid temperature response [5]. Wavelength tuned GNPlates are integrated into a polymer film and applied during fabrication.

2.1 Gold Nanoplate Synthesis

Gold nanoplates with precisely controlled near infrared (NIR) absorption are synthesized by one-step reaction of chloroauric acid and sodium thiosulfate in the presence of a cellulose membrane. The nIR absorption wavelengths and average particle size increase with increasing molar ratio of $HAuCl_4/Na_2S_2O_3$. The gold salt used is hydrogen tetrachloroaurate (III) trihydrate 99.99% ($HAuCl_4 \cdot 3H_2O$) purchased from Alfa Aesar. A 1.72 mM solution is prepared with DI water and protected from light with aluminum foil. A 32.6 ml volume of the 1.72 mM gold salt solution is combined with 7.4 ml of a 3 mM sodium thiosulfate pentahydrate solution ($Na_2S_2O_3 \cdot 5H_2O$; purchased from Sigma-Aldrich) to perform the reactions for synthesizing the nIR GNPlates.

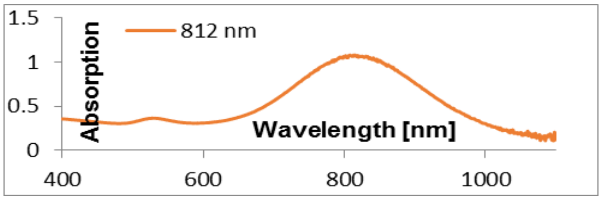

Figure 2: Absorbance spectrum of the gold nanoparticles synthesized for this study with a peak at 812 nm.

A 12 kDa MWCO membrane (Flat Width = 43 mm) from Sigma-Aldrich is cut to the desired length for 812 nm GNPlates, then one end of the tube is closed with a weighted dialysis clip and filled with the gold salt solution. The $Na_2S_2O_3$ solution is then added into the tube via pipette followed by mixing of the solution within the membrane by manually stirring with the tip of the pipette and aspirating with the pipette pump. Subsequently, air is removed from the tubing and the other end of the membrane is clipped. The sealed tube is placed in an 8 L beaker with 7 L of DI water and a stir bar at the bottom of the beaker rotating at 200 RPM and allowed to react for 1 hour. Particle batches, composed of various pseudo-spheres and anisotropic nanostructures, are dispersed in 8% PVP by weight solution to make the polymer GNPlate mixture with an absorption peak at 812 nm, as shown in Figure 2.

2.2 Cleanroom Processing

The devices for this project are fabricated with a two mask cleanroom process. A standard 4 inch silicon wafer acts as the substrate for material deposition. The bilayer required for creating the pop-up geometry is created by first thermally growing a 500 nm SiO_2 layer on the substrate. Photoresist is then deposited and patterned to mask the surface for the metal deposition. Next, a 10 nm/90 nm titanium/platinum (Ti/Pt) layer is deposited by sputtering, the features are defined by lift-off of the previous patterned photoresist.

A second photomask is used to define where the SiO_2 will be etched to make windows to the silicon. The exposed photoresist and patterned metal act to mask areas of the SiO_2 while the wafer is exposed to a reactive ion etch to create open windows to the substrate. Before releasing the devices, spin coating is used to disperse the polyvinylpyrrolidone (PVP) GNPlate solution on the substrates. A pipette is used to place a 35 μL droplet on the center of a device, and then the solution is dispersed by spinning at 4000 rpm for 10 seconds. The device is then baked on a 115°C hotplate for 2 minutes to stabilize the film. Typical thickness of a PVP GNP film is approximately 100 nm.

With evenly distributed particles on the surface, some of the residual PVP must be removed to allow the final release step. The devices are placed in a very low percent oxygen plasma for 30 seconds to remove the residual film that would otherwise prevent the Si substrate from being exposed to the etching gas. Finally, the previously etched oxide openings allow an isotropic XeF_2 etch of the silicon that is not hindered by the PVP film, and that releases the strained bilayer from the surface as seen in Figure 4.

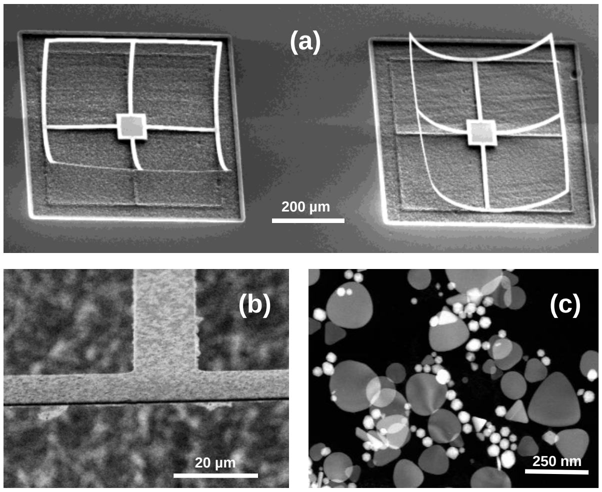

Figure 4: SEM image of the fabricated devices (a), closer view to show the depisited GNPs (b), and a TEM of the gold nanoparticles (c).

3 EXPERIMENTAL SETUP

An experiment was designed to characterize variation in the mechanical response of a popped-up microstructure when resonant GNPlates are applied to the surface. When exposed to a specific wavelength of light, each particle acts as a plasmonic heater [6]. This property of the GNPlates is based on a phenomenon called surface plasmon resonance. The absorption and electric field within the particle are controlled by the particle geometry which allows the film to be tuned to adjust the absorption of the substrate in a specific wavelength range, such as the 812 nm resonant GNPlates used here.

3.1 Laser Control and Device Monitoring

The GNPlates were tuned to this wavelength to complement the avaliable 808 nm diode laser. A schematic of the experimental setup is shown in Figure 5. The laser is controlled by a 490 Hz pulse width modulated (PWM) signal (5 watt at 100% power), generated by an Arduino Duemilanove with an ATmega328P microcontroller (a). The duty cycle of the waveform is varied to adjust the laser power (b). The form of the surface structure while under the laser is monitored in an Olympus microscope with a JenOptik ProgRes CF camera attachment (c).

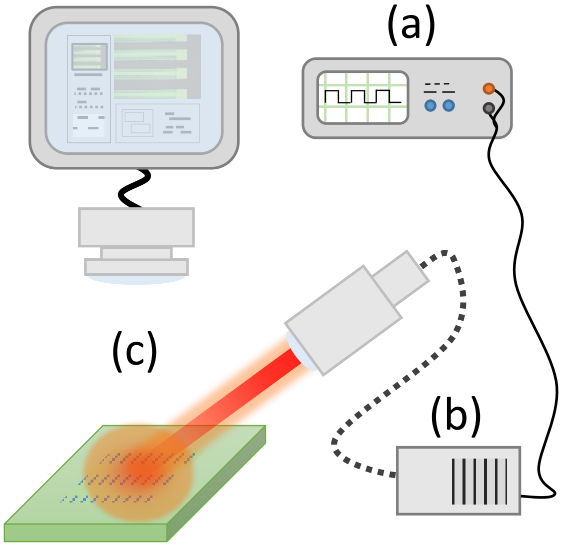

Figure 5: Schematic of the setup used for data collection in this system.

Samples were fabricated on a single wafer, and half were coated in GNPlates. Six duty cycle levels were chosen to test the device: 0, 10, 15, 20, 25, and 30 percent “power on” time. This sequence was randomized and collected from a pair of devices at each level with GNPlate coated and bare surfaces. Data was captured in the form of a 250 image sequence taken at ~3 FPS (250 images in 90 seconds). Matlab was used to process the sequences and monitor the reflection.

3.2 Segmenting Reflection in the Images

Image processing techniques were used to extract the reflected area and estimate the windowpane structure’s radius of curvature. In Figure 6, raw images of a device’s reflection are shown at the unheated state (a), an intermediate heated state (b), and the maximum possible thermal deflection (c). To do the segmentation, an image is split into its RGB components, such as the (R), (G), (B) below which are the components of (b). The pixels in the (G) component are catagorized by an intensity threshold (I > 155) as seen in (d) in orange.

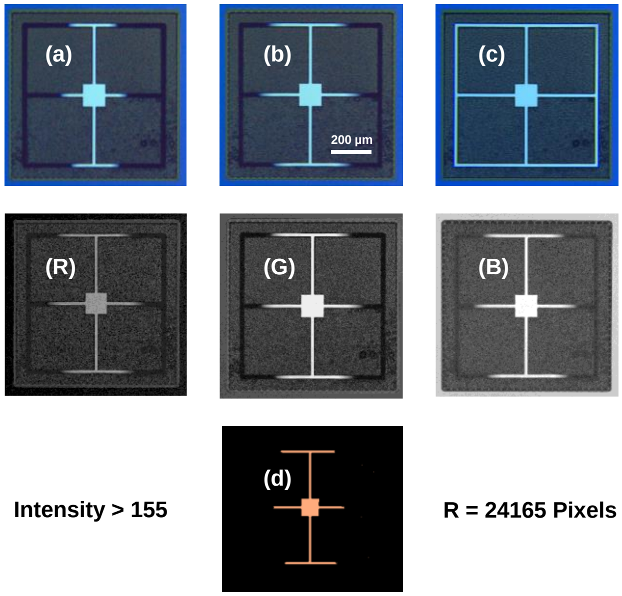

Figure 6: Reflection profiles of a pop-up windowpane at different stages of actuation (a, b, c). The RGB components of a reflection image (R, G, B). The segmented reflection used for a pixel count (d).

A simple pixel count is taken, and this metric is proportional to the deflection of the device, which is proportional to the light-induced temperature. This technique was applied to each sequence of 250 images at each duty cycle and sample combination (24 total sequences).

4 RESULTS

At a glance, the resulting data indicated that adding a GNP coating will improve the light absorbtion of the pop-up bilayer. Figure 7 shows averages of the duplicate trials of GNP-coated and bare samples at each duty cycle over 90 seconds. The dashed lines represent bare sample averages, and solid lines have incorperated GNPs. Each color pair is at a specific duty cycle, as labeled in the legend. As expected, higher applied power resulted in larger deflection, which is seen as a higher reflectivity in Figure 7. The Y-axis is normalized to the starting value of the zero-duty cycle trace in each averaged data set. The difference in reflectance near t=0 is due to variation between each device.

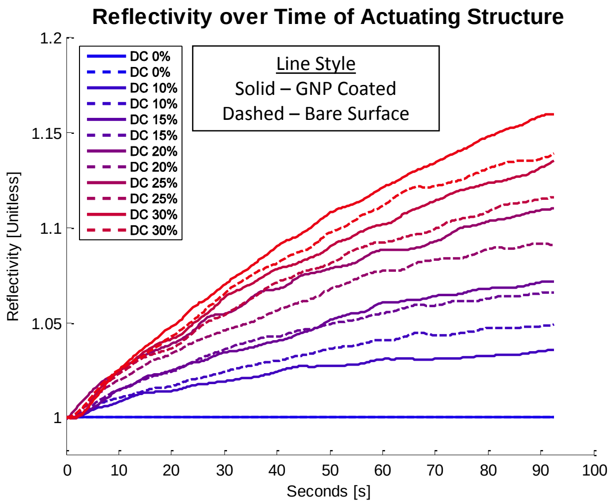

Figure 7: Reflectivity averaged data at different duty cycles (DC) for GNP coated and bare surfaces.

An analysis of variance (ANOVA) to generate statistics on the factors present in this study, with included interactions and independent devices from set to set. The presence of GNPs on a device at any duty cycle was found to be a significant factor of the resulting reflectivity ($p = 0.0042$), and therefore deflection.

5 DISCUSSION

The reflection was taken at initial and maximum deflections for each device, and a curve was fit between the two pixel counts. Using that estimation, a conversion between reflection and radius of curvature has been established. The level after 90 seconds has been collected at each duty cycle (the end points in Figure 7) and is displayed in Figure 8. This very clearly shows that there is an increased effect seen on the GNPlate-coated devices, shown in red.

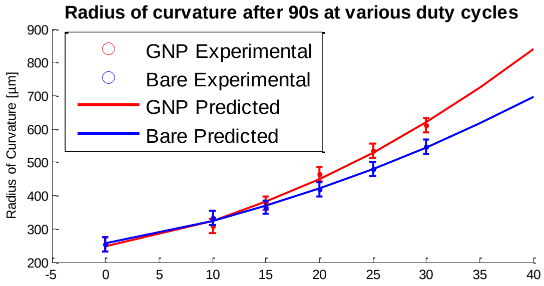

Figure 8: Fitted curves displaying the correlation between light and deflection of the structure with error bars depicting 95% confidence interval of the fit.

Since the duty cycle is directly laser power being applied, the average efficiency increase can be determined by taking the difference between the lines at each duty cycle level at a certain time (at 90s for this calculation), and making a linear regression. The slope of that line is 5.2, which is an estimate of the average percent effiency increase that is added by the GNPlates. This figure is in the vicinity of previously reported efficiency calculations on a planar surface [3].

6 CONCLUSION

We have shown that this hybrid of near infrared absorbent gold nanoparticles and MEMS fabrication technology leads to a surface with enhanced absorbtion. This project adds to the foundation of knowledge required for designing light-powered microactuators for exploring cellular response to mechanical stimuli, and performing studies that increase our understanding of tissue response to mechanical stresses.

ACKNOWLEDGEMENT

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