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Monitoring of ultraviolet pulse rate dependent photomechanical actuation in carbon nanotubes using fiber Bragg gratings

B. N. Shivananju,1 Ashish Suri,1 S. Asokan,1,2,3 and Abha Misra1,2,a)
1Department of Instrumentation and Applied Physics, Indian Institute of Science, Bangalore, India
2Applied Photonics Initiative, Indian Institute of Science, Bangalore, India
3Robert Bosch Centre for Cyber Physical Systems, Indian Institute of Science, Bangalore, India

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In this Letter, we present a non-contact method of controlling and monitoring photomechanical actuation in carbon nanotubes (CNT) by exposing it to ultra-violet radiation at different pulse rates (10 to 200 Hz). This is accomplished by imparting a reversible photo induced strain (5–330 με) on CNT coated fibre Bragg gratings; CNT undergoes an internal reversible structural change due to cyclic photon absorption that leads to the development of mechanical strain, which in turn allows reversible switching of the Bragg wavelength. The results also reveal an interesting pulse rate dependent rise and fall times of photomechanical actuation in CNT. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4860965]

Carbon nanotubes (CNT) are reported to be photosensitive due to near black body characteristics; these are responsive to a wide range of optical wavelengths from ultraviolet (UV) to infrared (IR) radiations (0.2 to 200 μm).1 Zhang and Iijima have shown that single walled carbon nanotubes (SWCNT) can undergo photomechanical actuation when exposed to visible light using different light sources.2 Thereafter, many researchers have explored the photomechanical actuation in pristine CNT, using IR or visible wavelength3,4 as well as its composites with polymers.5,6 These studies have revealed that the photomechanical actuation in CNT is dependent on intensity, power, and polarization of the light.7,8 Moreover, alignment and orientation of CNT also influence the photomechanical actuation.7,8 It has been suggested that the interplay between photo-elastic, electrostatic, polaronic, and thermal interactions give rise to the overall photomechanical responses in CNT.2,5,7,8

In literature, the photomechanical actuation in bulk CNT has been demonstrated mostly using mechanical and complex techniques with a resolution of about 1 μm.5,7 However, in the present work, a higher measurement accuracy (~picometer) has been achieved for the photo actuation in comparison with the earlier techniques.5,7 Here, a fiber Bragg grating (FBG) interrogator system with a resolution of 1 pm (picometer) is used to monitor the photomechanical actuation in CNT by measuring the shift in the Bragg wavelength. In a recent related report by the authors,9 the photomechanical actuation in CNT has been used to tune and reversibly switch the Bragg wavelength by exposing the CNT coated FBG (CNT-FBG) system to a broad band radiation.9 This Letter reports monitoring of UV pulse rate dependent photomechanical actuation in CNT. There is no such report in literature on the effect of UV pulse rate on photomechanical actuation in CNT by using FBG.

The phase mask method is widely used to fabricate FBG sensors;10–14 In this method, a UV beam (KrF excimer laser) of 3 mJ energy and 248 nm wavelength passes through the phase mask over a length of 3 mm to form an interference pattern in the core of a photo sensitive fiber that has core and clad diameters of 4.2 and 125 μm, respectively (Fibercore, SM1500).15 This process results in photo imprinting of a refractive index modulation (Bragg grating) in the fiber core. The grating periodicity produced with the phase mask of 1064 nm pitch is approximately 532 nm, giving a baseline Bragg wavelength around 1550 nm with a bandwidth of 80 nm.

Multi-walled carbon nanotubes (MWCNT) are grown directly on to the FBG sensor by using chemical vapor deposition. This process involves the thermal decomposition of a carbon source, toluene in the presence of ferrocene, which acts as a catalyst, at a high temperature of 780 °C under an inert gas atmosphere. The two precursors are mixed by 0.02 g/ml. The solution is carried into a reaction zone after pre-heating to its vaporization temperature using argon as the carrier gas at a flowing rate of 800 sccm (standard cubic centimeter per minute). A 50 ml solution is vaporized for a complete reaction. The FBG is used directly as a substrate to grow the vertically aligned CNT.15,16

Figure 1(a) shows a high-resolution scanning electron micrograph (SEM) of CNT-FBG system, where MWCNT are directly grown on the circular FBG surface (FBG cladding is shown by an arrow) along its radial direction. The diameter of CNT in the mat is measured between 10 and 50 nm and an overall length is ~70 μm; the inset in Figure 1(a) shows a uniform coating of CNT on to the overall length of the FBG. Figure 1(b) shows a high-resolution image of the as-grown CNT, where a vertical orientation of all CNT in the mat can be observed.

Photomechanical actuation study on CNT-FBG is carried out under an exposure of UV light (248 nm, using an UV excimer laser), as shown schematically in Figure 1(c). A tunable UV laser is used with an energy variation from 1 to 4 mJ with a resolution of 1 mJ; the pulse rate or chopping frequency is tuned between 1 and 200 Hz with a resolution of 1 Hz. The UV light is exposed perpendicular to the axis of
CNT-FBG system as can be seen in Figure 1(c). A resulting shift in the Bragg wavelength of CNT-FBG system is measured using a FBG interrogator (Micron Optics SM130) with a resolution of 1 pm at a sampling rate of 1 kHz.

When a broad band light (1510–1590 nm) is launched inside the core of a FBG, the contribution of the reflected light from each of the grating planes add constructively in the backward direction for a particular Bragg wavelength ($\lambda_B$), as given below

$$\lambda_B = \frac{2n_{eff}}{K};$$

where $K$ is the grating period and $n_{eff}$ is the effective refractive index of the fiber core. Any external perturbation such as strain, temperature, etc., will alter the grating period ($\Lambda$) and effective refractive index ($n_{eff}$) of the grating, which in turn results in shift in the reflected Bragg wavelength ($\Delta \lambda_B$).

By interrogating the shift in Bragg wavelength, the external perturbation at the grating site can be quantified.

When the CNT-FBG system is exposed to UV radiation along its radial direction, a shift is observed in the Bragg wavelength, which is due to the strain induced by the photo-mechanical actuation in CNT. This induced strain alters the Bragg wavelength ($\lambda_B$) according to

$$\Delta \lambda_B = \lambda_B (1 - P_e) \varepsilon_Z,$$

where $P_e$ is an effective strain-optic coefficient, $\varepsilon_Z$ is the photo-induced strain, $\lambda_B$ is the initial Bragg wavelength, and $\Delta \lambda_B$ is the shift in the Bragg wavelength due to photo-induced strain. Above relation provides a direct relation between shift in Bragg wavelength and the magnitude of the photomechanical actuation in CNT to provide an optical approach for studying photomechanical properties of CNT.

Figure 2 shows the shift in Bragg wavelength with the switching time. A reversible switching in the Bragg wavelength is seen when the incident UV radiation is periodically turned on and off for five cycles. This cyclic response of the shift in Bragg wavelength is observed due to the photo-elastic nature of CNT system as mentioned in the earlier studies.

An average shift of ~370 pm is observed when CNT-FBG system is exposed to 4 mJ of UV radiation at a pulse rate of 200 Hz. Fluctuation in Bragg wavelength can be seen despite the incident energy has been kept constant at 4 mJ, which is due to the variability ($\pm 0.5 \text{mJ}$) in the laser energy.
after turning off the UV exposure, the Bragg wavelength recovers back to its initial value.

Figures 3(a) and 3(b) show the response or rise time ($\tau_{\text{rise}} = t_{90\%} - t_{10\%}$) and recovery or fall time ($\tau_{\text{fall}} = t_{10\%} - t_{90\%}$) of CNT-FBG system, respectively, where $t_{90\%}$ and $t_{10\%}$ are the time taken by the system to reach 90% and 10% shifts in the Bragg wavelength. The rise time and fall time of the CNT-FBG system for photomechanical actuation is found to be $\sim 3.84$ s and $\sim 4.46$ s when it is exposed to 4 mJ at 200 Hz; this indicates that the time taken by the CNT-FBG system for recovering back to the initial position is slightly higher than the rise time.

In addition, the effect of UV pulse rate on photomechanical actuation in CNT-FBG system has been investigated. Figures 4(a) and 4(b) show the photomechanical actuation of CNT at different pulse rates of 10–40 Hz and 50–200 Hz, respectively. Figure 4(a) depicts that the Bragg wavelength shifts to $\sim 12$ pm when UV laser (10 Hz, 2 mJ) source is switched on for a constant period of 1 min. During this period of UV exposure, the Bragg wavelength remains constant (stable); once the UV laser source is turned off, the Bragg wavelength returns to its initial value, as similar to the behavior observed in Figure 2. Upon increasing the pulse rate of UV laser to 20, 30, and 40 Hz, an increase in the Bragg wavelength has been observed to 24, 30, and 40 pm, respectively, which shows a pulse rate dependence on reversible switching of CNT due to photo elastic behavior of the CNT-FBG system.

Further increasing the UV source energy to 4 mJ, keeping the same pulse rates (10 to 40 Hz in steps of 10 Hz), the Bragg wavelength shifts by more than two times as compared to the exposure with 2 mJ. Maximum shifts of 94 and 40 pm have been observed at 4 and 2 mJ, respectively, for a maximum pulse rate of 40 Hz. These results can directly be attributed to the enhanced photomechanical actuation in CNT by increasing energy as well as pulse rate of the exposure. Thus, the present results reveal that photomechanical actuation in CNT can be controlled not only by controlling the intensity of the light exposure (as reported earlier) but also with the pulse rate.

Figure 4(b) shows the shift in the Bragg wavelength at higher pulse rates (50 to 200 Hz) in steps of 50 Hz; the shift in the Bragg wavelength doubles with the increase in the energy from 2 to 4 mJ as in the earlier case of lower pulse rates (10 to 40 Hz).
126, and 147 pm at 2 mJ of UV power and 122, 209, 295, and 370 pm at 4 mJ of UV exposure at four pulse rates of 50, 100, 150, and 200 Hz, respectively. Since the pulse rates are directly related to the radiation power, hence a higher actuation in CNT resulted in the larger shift in the Bragg wavelength.

In order to ascertain that the observed shift is due to the CNT-FBG system and not due to FBG alone, bare FBGs have also been exposed to UV light at both lower (10 to 40 Hz) as well as higher pulse rates (50 to 200 Hz) using 4 mJ radiation energy. The results are depicted in Figures 4(c) and 4(d). The bare FBGs also respond to the switching of the UV radiation source; a shift of 30 pm has been observed at 4 mJ incident energy with a pulse rate of 200 Hz, which is about 12 times lesser as compared to the Bragg wavelength shift observed in CNT-FBG system (~370 pm at 4 mJ and 200 Hz pulse rate). This observed shift in the Bragg wavelength for the bare FBG can be attributed to the thermal effects (around 3°C rise in temperature) induced upon UV exposure. In addition, an unstable response from FBG is observed, which is shown by a continuous increase in base line with the pulse rates. The base line in bare FBG increases linearly with pulse rates (10–200 Hz). On the other hand, in the CNT-FBG system, the base line remains nearly constant, with the increase in the pulse rate. The observed irreversible shift in the Bragg wavelength of bare FBG can be related to the fatigue induced by high energy or rise in temperature associated with UV exposure.

Figures 5(a) and 5(b) show a linear relation in shift of the Bragg wavelength with the pulse rates for both lower (10–40 Hz) and higher pulse rates (50–200 Hz) at 2 and 4 mJ energies. This linear characteristic of CNT-FBG system can be exploited in the photomechanical actuation of CNT in the picometer scale, which can find applications in tunable sensors. However, it is to be noted that an increase in the pulse rate induces more noises in CNT-FBG system, as indicated by the error bars.

The observed linear shift in the Bragg wavelength with the pulse rate can directly be translated into the photo-induced strain in CNT upon UV exposure. Figures 5(c) and 5(d) reveal the magnitude of photomechanical strain undergone by CNT at different UV laser pulse rates, namely, 10–40 Hz and 50–200 Hz, respectively. The photo induced strain in CNT can be calculated by dividing the Bragg wavelength by 1.2 pm (as the sensitivity of the FBG sensors used in this study is 1.2 pm/με). The obtained strains are 5–87 με at 10–200 Hz for 2 mJ and 13–274 με at 10–200 Hz for 4 mJ. It can be observed that the magnitude of photomechanical strain also doubles at 4 mJ as compared to 2 mJ energy of the exposure, which is shown to follow a linear relation with the pulse rate. Based on these observations, it is suggested that CNT-FBG system can be used for real time monitoring of photomechanical (photo-induced) strain in nano-scale materials, which is found precise than other reported techniques that are mostly mechanical and complex in nature.

Figure 6(a) shows the response of CNT-FBG system for five cycles of UV exposure at four different pulse rates (50 to 200 Hz), which reveals the stability of the system against multiple numbers of exposures. It can be seen that there is a persistent reversible switching in the Bragg wavelength due to photo elastic nature of CNT over a repeated exposure of UV radiation with a negligible drift in the base line. Figure 6(b) shows the photomechanical induced strain (0.085 to 0.330 με) in CNT that is calculated over the same number of exposure cycles at different pulse rates (50 to 200 Hz). The photomechanical induced strain at constant pulse rate remains same in magnitude for all cycles of exposure; however, it increases in magnitude with the increase in pulse rate.

In addition, the pulse rate dependence on response time (rise time) and recovery time (fall time) of photomechanical
actuation in CNT-FBG has been estimated in (Figures 6(c) and 6(d), respectively). The rise time of CNT-FBG system for photomechanical actuation is found to be ~2, 3, 3.5, and 3.9 s for 50 Hz, 100 Hz, 150 Hz, and 200 Hz, respectively, as depicted in Figure 6(c); the photomechanical response of CNT-FBG at 200 Hz pulse rate is higher than that at 50 Hz pulse rate. The fall time of photomechanical actuation in CNT-FBG system is found to be ~3, 3.3, 4, and 4.4 s at 50, 100, 150, and 200 Hz, respectively, as shown in Figure 6(d); the photomechanical recovery time of CNT-FBG at 200 Hz pulse rate is higher than 50 Hz pulse rate. Higher rise time and fall time at 200 Hz are attributed to the higher photoinduced strain in CNT-FBG due to higher power associated with this pulse rate (200 Hz).

This Letter brings out a method of controlling and monitoring photomechanical actuation or photo induced strain (5–330 Å) in CNT by exposing it to UV radiation at different pulse rates (10 to 200 Hz); the FBG is utilized for a real time monitoring of the photo induced strain in CNT under UV exposure; the present technique is more precise (1 pm resolution) than any other existing ones (1 µm resolution), which are mostly mechanical and complex in nature. An investigation on pulse rate dependent response time (rise time) and recovery time (fall time) of photomechanical is also presented.