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Photodriven, Flexural–Torsional Oscillation of Glassy Azobenzene Liquid Crystal Polymer Networks

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Abstract

Cantilevers composed of glassy, photoresponsive liquid crystalline polymer networks (LCNs) are shown to oscillate at high frequency (~50 Hz) and large amplitude when exposed to light from a 442 nm coherent wave (CW) laser. Added dimensionality to previously reported in-plane oscillations is enabled by adjusting the orientation of the nematic director to the long axis of the cantilever yielding in-plane bending accompanied by out-of-plane twisting (flexural–torsional oscillation). The fundamental photoresponse of this class of glassy azobenzene liquid crystal polymer networks (azo-LCN) is further probed by examining the influence of cantilever aspect ratio, laser intensity, and temperature. The frequency of photodirected oscillations is strongly correlated to the length of the cantilever while the amplitude and threshold laser intensity for oscillation is strongly correlated to temperature.

1. Introduction

Actuators are systems in which energy is converted from an input stimulus into motion.[1] Both material performance and system design are critical elements in efficiently transducing energy into mechanical work. Materials employed as monolithic, kinematic elements that respond to an energy stimulus by changing shape or form can be utilized for actuation and can simplify the design of a system, potentially eliminating any need for response amplifiers. Soft materials are excellent transducers of energy, typified by low energy requirements and large displacements.

Dynamic responses to stimuli (stimuli-responsive) as observed in a shape memory polymer (SMP)[2] are a prime example of transduction in which a temperature input yields large scale mechanical deformation. Similarly, electroactive polymers (EAPs) are capable of muscle-like power densities and have been examined for applicability in robotics.[3]

In addition to SMPs and EAPs, another class of adaptive materials that has received considerable attention is liquid crystal polymer glasses and elastomers ("liquid crystal polymer networks", LCNs). LCNs are soft materials known to transduce energy from a range of input stimuli including temperature,[4] electric field,[5, 6] magnetic field,[7, 8] and light.[9-12] LCN materials can yield amplified responses compared to amorphous or crystalline polymeric counterparts due to the order of the system. For example, thermotropic phase transitions in elastomeric LCN can accomplish significant work upon heating through the nematic to isotropic phase transition.[13]

A topic of considerable interest over the last decade has been photoresponsive LCN, materials which can be wirelessly actuated with light.[14] Light offers novel control over the magnitude and directionality of the response of a system evident in a number of works that use the orientation of light polarization to direct the bending response of thin films and cantilevers.[11, 15-20] In all of the reports to date, the photosensitivity of the system is enabled through the utilization of the azobenzene chromophore which predominately is covalently bound into the network but at times[12] employed as a guest-host. The response of azo-based LCNs (azo-LCNs) has primarily been static, evident as uniaxial contraction of a film[9, 10, 21] or bending of a cantilever.[15, 16, 19, 20] Recently, oscillatory responses have been reported in azo-LCNs exposed to 442 (helium cadmium, HeCd) or 457–514 nm (multi-line Argon-ion) laser light with frequencies ranging from 10–270 Hz.[17, 22] The oscillations previously reported occur in-plane to the long axis of the cantilever.

In the work presented here, we revisit the oscillatory behavior previously observed in monodomain azo-LCN[17, 22] in a new class of glassy materials with a comparatively lower azobenzene concentration. For the first time, flexural–torsional oscillations composed of three-dimensional movement encompassing both in-plane bending and out-of-plane twisting are reported, enabled by aligning the nematic director of the monodomain azo-LCN at intermediate angles to the long axis of the cantilever. Furthermore, the flexural–torsional oscillations of these materials are distinguished by the use of comparably higher modulus, glassy monodomain azo-LCN which doubles the attainable frequency for a given cantilever geometry. The flexural– torsional oscillations presented here emulate the flapping motion of insects that undergo three-dimensional movement encompassing bend, twist, and sweep to generate efficient and highly agile flight.[23, 24]

2. Results and Discussion

Glassy, high modulus azobenzene liquid crystal polymer networks (azo-LCN) were formed by photoinitiated polymerization of the crosslinking monomers RM257 (79 wt%) and 4–4′-bis[6-(acryoloxy)hexyloxy]-azobenzene (2azo, 20 wt%) with 1 wt% of the inorganic photoinitiator Irgacure 784 (chemical structures shown in **Figure 1**a). The samples were cured at 75 °C, just above the crystalline to nematic transition for the monomers. The result is a uniaxially aligned "monodomain", referred to here as MD-20CL. The geometry of the cantilever is illustrated in Figure 1b. The long axis of the cantilever will be referred to as *x*, the nematic director of MD-20CL as N, and the orientation of the electric field direction of light (polarization) as *E*. The thermomechanical properties of MD-20CL are shown in Figure 1c and 1d. As evident in Figure 1c and Figure 1d, the peak of tan *δ* occurs near 47°. The modulus of MD-20CL is anisotropic due to the preferential orientation of the liquid crystal molecules along the rubbing direction, with a storage modulus (*E*') of approximately 1 GPa when *N*//x and 0.6 GPa when *N*⊥x.

Figure 1

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a) Chemical structures of RM257 and 2azo. b) Illustration of cantilever geometry and light polarization. Thermo-mechanical properties (tan δ (□), storage modulus (*E*', ○), and loss modulus (*E*'', Δ) of MD-20CL with the nematic director c) parallel and d) perpendicular to the 6 mm × 1 mm gauge.

Subjecting MD-20CL cantilevers to 1.0 W cm^{-2} of 442 nm exposure yields oscillatory responses. As shown in **Figure 2**a, the alignment of the nematic director (*N*) to the cantilever can strongly influence the dimensionality of the oscillations. When *N* is cut so that it is parallel to the long axis (*x*) of the cantilever, in-plane oscillation similar to that previously reported[17, 22] is observed (Figure 2a-i). When *N* is aligned such that it is along the short axis of the cantilever (*N*⊥*x*), the material exhibits asymmetric bidirectional bending[16] (Figure 2a-iv) when exposed to linearly polarized light (E//*x* or *E*⊥*x*) but does not oscillate at the range of intensities available in the irradiation system. However, when *N* is aligned at intermediate angles to the long axis of the cantilever, flexural–torsional oscillatory behavior is observed. Evident in images in Figure 2a-ii, when *N* is aligned approximately 15° to *x* the oscillation is typified by large amplitude with slight twisting. When *N* is aligned approximately 45° to *x* (Figure 2a-iii), the cantilever oscillates at comparably less amplitude but with a significant increase in twisting motion. The flexural–torsional oscillations observed in the MD-20CL cantilever with intermediate alignment of *N* at 15° to *x* are further examined in Figure 2b, which images the upstroke and downstroke of the oscillating cantilever from above. Apparent in Figure 2b is twisting of the cantilever (particularly at the tip) through the course of the stroke.

Figure 2

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a) Polarized optical micrograph (POM) of MD-20CL and photo-mechanical response of cantilevers with nematic director cut i) 0°, ii) 15°, iii) 45°, and iv) 90° to the long axis of the cantilever. b) Top view of multidimensional oscillation of MD-20CL cantilever with nematic director cut 15° to the long axis. 442 nm exposure at 1.0 W/cm^2.

The flexural–torsional response is also apparent in lower amplitude, static bending that occurs when MD-20CL cantilevers are subjected to lower intensity 442 nm light. Shown in **Figure 3** is the photomechanical response of cantilevers cut with N aligned 0°, 30°, 45°, and 65° to *x* when exposed to 200 mW cm^{-2} of 442 nm light polarized parallel to *x* (*E*//*x*). In-plane bending is observed when *N* is parallel to *x* (0°). As the alignment of *N* is rotated with respect to the long axis of the cantilever, in-plane bending as well as out-of-plane twisting occurs. The magnitude of the out-of-plane twisting increases as the angle between *N* and *x* increases, up to a maximum that occurs at 65° for the cantilever aspect ratio used here. As shown in Figure 3, when the cantilever is cut such that *N* is –65° to *x*, the direction of twisting reverses. The photoinduced deformation is retained (shape memory) in the absence of light.[25]

Figure 3

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In plane bending and out of plane twisting observed in MD-20CL cantilevers with the nematic director aligned 0°, 30°, 45°, 65°, and –65° to the cantilever long axis (x) when exposed to 200 mW cm^{-2} (442 nm) polarized parallel to x.

To ascertain whether the flexural–torsional oscillation can be induced with the polarization direction of the 442 nm laser, **Figure 4** examines the photoresponse of cantilevers with *N*//*x* subjected light polarized 0–70° to *x*. Figure 4 plots the amplitude of the oscillation of MD-20CL at three laser intensities as a function of polarization angle. As expected, increasing the laser intensity increases the amplitude of the oscillation. However, as evident in the inset images (for 1.2 W cm^{-2} data), the oscillations at intermediate polarizations ranging from 15–70° for all three intensities presented in Figure 4 yield only in-plane oscillation.

Figure 4

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Oscillation amplitude as a function of polarization angle for MD-20CL cantilevers with the nematic director along the long axis at 1 W cm^{-2} (▪) 1.1 W cm^{-2} (□), and 1.2 W cm^{-2} (•).

The data presented in Figure 2–4 indicate that the flexural–torsional oscillation in MD-20CL cantilevers is strongly related to the orientation of the nematic director. The photo-directed bending of the cantilever is caused by an absorption gradient in the azo-LCN, which absorbs most of the light near the surface resulting in a nonuniform strain through the sample thickness. The absorption gradient within MD-20CL can be described to a first approximation by Beer's law (although recent reports document deviation from Beer's law in similar materials.26) The absorption coefficient of MD-20CL is 1304 cm^{-1} at 442 nm, which means that light of this wavelength is extinguished within 7.5 μm from the exposed surface. In the terminology of27 the *w*/*d* of these materials is 2 where *w* refers to the thickness of MD-20CL and *d* is the optical penetration depth estimated from Beer's law. Although this predicts that light should be completely extinguished within the MD-20CL, approximately 1–5 mW cm^{-2} can be transmitted through the film in the intensity regime utilized here (0.8–1.2 W cm^{-2}), explained previously by Warner27, 28 and Terentjev.26 The absorption gradient is mirrored by a strain gradient, which causes the cantilever to bend towards the light source. Increasing laser intensity increases the magnitude of the bending by increasing the photogenerated strain, eventually generating a large enough moment of inertia for the cantilever to deflect in and out of the CW laser beam resulting in oscillation. Evident here as well as in our prior work,16 the contractile strain is largest along the nematic director in monodomain azo-LCN and the magnitude of the strain is influenced by the orientation of the polarization of the 442 nm light. In addition to bending, we also observe out-of-plane twisting (torsion) when the nematic director of MD-20CL is cut at intermediate angles to the long axis of the cantilever. We propose that the out-of-plane twisting results from a photogenerated shear gradient through the thickness of the MD-20CL cantilever. Offsetting the nematic director from the cantilever long axis also serves to offset the direction of photogenerated contractile strain from the primary geometric axes of the cantilever. The exposed surface now contracts to a greater extent along one diagonal of the cantilever than the other, resulting in shear. Similar to the photogenerated strain gradient that causes bending, the photogenerated shear is non-uniform through the thickness (mirroring the absorption gradient through the thickness of the azo-LCN). To accommodate the shear gradient, the cantilever twists. The direction of the twisting is dictated by the orientation of N. This is apparent in Figure 3 when N is +65° to *x* the cantilever twists to the left and when N is -65° to *x* the cantilever twists to the right. In the geometry examined in Figure 3, the maximum twisting is observed when the nematic director of the MD-20CL polymer is aligned at 65° to the long axis of the cantilever.

Photodriven oscillations of monodomain azo-LCN in chemistry entirely of azobenzene mesogens have been shown to oscillate from 10–270 Hz, strongly depending on the aspect ratio of the cantilever.[17], [22] Given that the MD-20CL samples examined here have significantly reduced azobenzene concentration as well as disparate thermomechanical properties, the remaining portion of this work focuses on the characterization of the fundamental structure-property of the oscillation of these glassy materials as well as the role of temperature. To avoid complication in analysis due to flexural–torsional responses, the data presented in **Figure** 5–7 examines in-plane oscillations in samples with the nematic director aligned parallel to the long axis of the cantilever (*N*//*x*). Figure 5 plots the frequency of the oscillations as a function of laser intensity. The threshold laser intensity to induce oscillations in MD-20CL cantilevers at room temperature is 1 W cm^{-2}. Increasing the laser intensity from 1–1.5 W cm^{-2} does not alter the frequency of the oscillation. Comparably, the previously examined oscillations in the previously examined azo-LCN chemistry occurred with as little as 0.2 W cm^{-2} for 20 μm thick samples.[17] The five-fold increase in threshold laser intensity necessary to induce oscillations in these materials with respect to the previous chemistry is likely related to the considerable reduction in azobenzene concentration and differences in thermomechanical properties, which requires more input energy to deflect the cantilever past 90° and initiate the oscillation. Increasing the intensity, as shown in Figure 5b, increases the amplitude of the oscillation of the MD-20CL cantilevers. As laser intensity increases from 1 to 1.5 W cm^{-2}, the amplitude of the oscillation increases from 20° to 90°.

Figure 5

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a) Frequency and b) amplitude of in plane oscillation of a MD-20CL cantilever as a function of laser intensity at room temperature. 442 nm exposure linearly polarized parallel to the long axis of the cantilever.

The oscillation frequency of a cantilever is known to be highly dependent on the aspect ratio (*x*/*y*). The influence of aspect ratio on the oscillation of MD-20CL was examined by decreasing the length of a cantilever with a constant width of 1 mm. As shown in Figure 6, the cantilever oscillates at 45 Hz for *x*/*y* of 5. Decreasing *x*/*y* increases the frequency to as much as 120 Hz at *x*/*y* of 3. As has been reported,[22] the resonant frequency of a cantilever scales with the inverse of the length squared which holds true in these materials as well. Interestingly, the frequency of the oscillation of these glassy, high modulus monodomain azo-LCN based on MD-20CL is two times greater for a given aspect ratio (5 mm × 1 mm) than the previously examined azo-LCN chemistry entirely composed of azobenzene mesogens.[17], [22] A number of factors are likely contributing to this increase, including an increase in modulus as well as an alteration of the penetration profile of the 442 nm light in the sample thickness.

Figure 6

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Oscillation frequency as a function of aspect ratio (*x/y*) for MD-20CL cantilevers subjected to 1.1 W cm^{-2} 442 nm exposure polarized parallel to the long axis of the cantilver.

Given the comparably large intensities in this work and the potential for photothermal effects, the photodriven oscillation of MD-20CL were examined over a range of laser intensities and temperatures (Figure 7). Figure 7a plots the oscillation frequency as a function of temperature. Increasing the temperature reduces the threshold laser intensity for oscillation. For example 1.0 W cm^{-2} or 0.8 W cm^{-2} light can induce oscillations at temperatures of 50° and 80° respectively. Notably, the frequency of the oscillations for the 5 mm × 1 mm cantilever is approximately 45 Hz, regardless of temperature. The oscillations are observed when the sample is initially below T_g (at 22 °C), near T_g (at 50 °C), and above T_g (at 75 °C). However, as shown in Figure 7b increasing temperature at a given laser intensity results in larger amplitude oscillations. The results in Figure 7 indicate that at the laser intensities examined here temperature is an important factor and indicate that photothermal heating is a potential contributor to the oscillation of these materials.[29]

Figure 7

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a) Frequency and b) amplitude of photodriven oscillation of MD-20CL cantilever as a function of temperature for laser intensities of 1.2 W cm^{-2} (•), 1.0 W cm^{-2} (□), and 0.8 W cm^{-2} (▪).

3. Conclusions

Flexural–torsional oscillations are observed in cantilevers composed of glassy, monodomain azobenzene liquid crystal polymer network (azo-LCN) when the nematic director is aligned at intermediate angles to the long axis of the cantilever. The flexural–torsional response of these materials is evidenced in both in-plane bending and out-of-plane twisting. The strong absorption of 442 nm light generates nonuniform strain through the sample thickness, causing bending. When the nematic director is aligned at intermediate angles to the long axis of the cantilever, a gradient in shear also forms that causes twisting of the cantilever. The fundamental oscillatory response of the comparably lower-azobenzene concentration materials examined here was examined as a function of laser intensity, cantilever aspect ratio, and temperature. It is found that amplitude of the oscillations increase with laser intensity and temperature. As predicted by cantilever beam mechanics, the frequency of the oscillation is strongly influenced by the aspect ratio of the cantilever, with oscillation frequencies of nearly 120 Hz observed in cantilevers with 3 mm length and 1 mm width.

4. Experimental Section

Synthesis of Azobenzene Liquid Crystalline Networks: Liquid crystal polymer networks containing azobenzene (azo-LCN) were synthesized by copolymerizing RM257 (Merck) and 4–4′-bis[6-(acryoloxy)hexyloxy]-azobenzene (2azo) (BEAM Co.). The polymerization was initiated by 1 wt% of the inorganic photoinitiator Irgacure 784 (I-784) (Ciba). The chemical structures of these molecules are shown in Figure 1a. A mixture of these solid monomers containing 20 wt% 2azo were melted and mixed at 100 °C and drawn by capillary action into 15 μm thick Elvamide-coated rubbed glass cells. For photocuring, the sample was cooled to 75 °C and subjected to 60 mW cm^{-2} of 532 nm laser light for 15 min to form a monodomain azo-LCN referred to here as MD-20CL. After photocuring, the MD-20CL film was removed from the glass substrates and washed in methanol to remove residual Elvamide alignment material.

Characterization Methods: The monodomain structure of the azo-LCNs was confirmed by polarized optical microscopy (POM, Nikon) with a heating accessory (Mettler), (Figure 2). Thermomechanical properties of the azo-LCN films were examined in cantilevers of 6 mm × 1 mm × 15 μm (width, length, and thickness, respectively). Storage modulus (E'), loss modulus (E''), and loss tangent (tan δ) were determined by dynamic mechanical analysis (DMA) (RSA III, TA Instruments) operating at a strain of 0.5% and frequency of 1 Hz with a heating rate of 2.5 °C min^{-1} over the temperature range of 20 °C to 200 °C. Glass transition temperatures (T_g) are reported from the maximum of the tan δ curve.

Photodriven Oscillation of Azo-LCN Cantilevers: The polarization controlled oscillation of the MD-20CL cantilevers was directed with a linearly polarized 442 nm line of a 130 mW helium-cadmium (HeCd, Kimmon) laser. Note that 442 nm light is absorbed by both the trans and cis isomers of azobenzene. The HeCd laser beam was expanded and collimated with a spherical lens and the polarization direction of the laser was controlled with a Fresnel rhomb (Newport). To achieve the intensities necessary to induce oscillation, the laser beam was focused and collimated to an approximately 4 mm diameter (optical setup was previously reported[17]). The focused beam was aligned such that the bottom 3 mm of the 5 mm × 1 mm cantilevers were exposed. As such, the tip of the cantilever during the highest point of the upstroke and the lowest point of the downstroke were not exposed. The photomechanical bending, twisting and oscillation of azo-LCN cantilevers were characterized by a camera operating here at a frame rate of 30 Hz. The oscillation frequency was determined optically with a photodiode and an oscilloscope. The amplitude of oscillation was measured from the images captured with the camera. The effect of temperature on photomechanical response of these materials was investigated by mounting the azo-LCN cantilevers in a Peltier heating device. At each temperature, the azo-LCN cantilever was allowed to equilibrate at the set temperature before measurement.

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