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Photomechanical Response of Pre-strained Azobenzene-Functionalized Polyimide Materials

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Abstract

Recently, we have reported that azobenzene-functionalized polyimide materials exhibit a substantial increase in photogenerated stress in comparison to azobenzene-functionalized acrylate-based liquid crystal polymer networks. Here, we show that pre-straining the azo-CP2-20 material further increases the magnitude of the photomechanical response visualized as bending in the cantilever geometry as well as in direct measurements of photogenerated stress. Additionally, we also report on the ability to optically fix both optically and mechanically generated shapes in azo-CP2-20. The optically fixable shape memory of azo-CP2-20 is enabled by the introduction of excess free volume during thermal processing as the material was pre-strained.



1. Introduction

The mechanical output of photoresponsive polymeric materials and composites is a complex interplay between light absorption (regulated by intensity and wavelength of light and concentration of absorbing species in the material system), photochemical or photothermal response of material or composite (regulated by polymer network morphology, wavelength of irradiation, guest additives), thermomechanical properties of the material, and the geometry of the sample.<u>1-4</u> The active response of the material system can be described by the mature and widely understood language of mechanics. The thin film<u>5</u> and cantilever<u>6</u>, <u>7</u> geometries have been widely exploited as a means to visualize and measure photomechanical effects in polymeric materials. Although the bending angles (radius of curvature) can be large, the photogenerated strain response of glassy polymeric materials is considerably smaller (0.1–2%) than what has been reported in liquid-crystal elastomers (≈20%).<u>2</u>, <u>8-12</u> It is important to note that for the cantilever to bend, light must be attenuated (e.g., absorbed) nonuniformly across the sample thickness to yield a strain gradient.

The wavelength of the exposing irradiation influences both the extent of absorption (e.g., the penetration depth of light into the cantilever—critically important to mechanics) as well as the photochemical mechanism in azobenzene-based material systems. Most commonly, UV light has been employed to trigger photomechanical effects. Absorption of UV light causes the azobenzene unit to isomerize from the *trans* to *cis* conformation. The isomerization of azobenzene from *trans* to *cis* is accompanied by a reduction in the molecular length from 9 to 5.5 Å. The reduction in molecular length of azobenzene generates a molecular level strain response. In azobenzene-functionalized liquid-crystal elastomers, the conformational change of azobenzene from the *trans* to *cis* isomer upon irradiation with UV light also results in a significant decrease in the liquid-crystal order parameter, which amplifies the strain by as much as two orders of magnitude.<u>13-15</u> The photomechanical effects generated by the *trans-cis*

mechanism (e.g., UV light) are metastable and restore to the original form at a rate proportional to the photochemical kinetics of *cis-trans* isomerization in the dark (hours to days). Faster reconfiguration of the material back to the original form can be enabled by subsequent irradiation to higher wavelength light (or heating) to induce the *cis-trans* isomerization. In low-concentration azobenzene material systems, the bend of the cantilever can be strongly dependent on irradiation time to UV.<u>12</u>

Our prior efforts have employed light of "blue-green" light with wavelength(s) ranging from 440–514 nm (typically 442 nm).9, 11, 12, 16-22 Patterned blue-green irradiation has been employed in azobenzene polymer thin films to generate both volume and surface relief gratings, formed through intensity and polarization holography.23, 24 In our work examining both azobenzene-functionalized liquid-crystal polymer networks and azobenzenefunctionalized polyimides-blue-green irradiation allows for polarization controlled forward/neutral/reverse bending that in nearly all cases exhibits shape fixing behavior (shape memory). Irradiating a mechanically deformed sample with blue-green light allows for optical fixing of a complex shape at temperatures far below the glass transition temperature. 25 Bluegreen light induces a related but distinctive photochemical mechanism referred to as trans-cistrans reorientation. This mechanism is observed upon irradiation of azobenzene material systems with linearly polarized light of a wavelength that is nearly equivalently absorbed by the trans and cis isomers (typically induced with wavelengths in the blue-green region of the electromagnetic spectrum). As such, to continuing irradiation—the azobenzene chromophores interchange between conformational states. Due to the rotational freedom of the azo bond as well as the dichroic absorbance of both *trans* and *cis* azobenzene, blue-green irradiation results in a statistical buildup of trans azobenzene reoriented normal to the incident linear polarization. This can be confirmed with polarized UV–Vis spectroscopy as photoinduced dichroism.<u>12</u>, <u>26-29</u> Importantly, the photoinduced dichroism in materials exhibiting optically fixable shape memory is stable over many days and potentially years—confirming that the photoinduced reconfiguration of the azobenzene units is trapped within the glass and the means by which the strain is fixed.

Regardless of the photochemical mechanism, the properties of the material regulate the kinetics and magnitude of the mechanical response. Properties of primary importance include the concentration of absorbing species, <u>10</u> polymer network morphology (amorphous, semicrystalline, and liquid crystalline), <u>13</u>, <u>21</u>, <u>22</u> and thermomechanical properties (glassy or rubbery state, modulus). <u>2</u>, <u>10</u>, <u>11</u>, <u>30</u> Within the subset of azobenzene-functionalized photoresponsive polymeric materials, the efficiency of the transduction from light into work is primarily regulated by the efficiency of the isomerization/reorientation of azobenzene. The isomerization of azobenzene in liquid media has been extensively studied, and known to be dependent on a number of factors but principally on the functionalization of the azobenzene

chromophore<u>31</u> (e.g., conventional, amino-azobenzene, or pseudo-stilbene) and polarity of the medium.<u>32</u> The extent of azobenzene isomerization (efficiency) is considerably lessened by embedding this photochromic moiety in a polymeric material—evident in the work of Paik and Morawetz,<u>33</u> which contrasts the kinetics and photostationary state concentration azobenzene photoisomerization in dilute solution, plasticized, and bulk forms.

A few recent reports examine photomechanical effects in glassy azobenzene-functionalized polymeric materials.21, 22 The examination presented here extends upon this recent work by studying photomechanical effects in pre-strained amorphous, azobenzene-functionalized polyimide material. The influence of pre-strain on the magnitude of the photomechanical response is measured by the deflection of thin cantilevers as well as the photogenerated stress of samples in tension. In both geometries, aligning the material by pre-strain substantially increases the response and thus, increases the efficiency of converting input photons to output mechanical work. Accompanying the pre-strain is the ability to optically fix both optically and mechanically generated shapes, due to the introduction of excess free volume into the material during thermal processing.

2. Experimental Section

2.1. Synthesis of Crosslinked Azobenzene Polyimide Network "Azo-CP2-20"

The experimental details for the synthesis of the crosslinker and "azo-CP2-20" have been previously described.<u>21</u>

2.2. Subjecting Azo-CP2-20 to Pre-strain

The azo-CP2-20 samples were heated to 275 °C (50 °C above T_g) on a hot plate. After 5 min at this elevated temperature, the materials were stretched by tweezers to the desired length immediately followed by immersion in liquid nitrogen to arrest the glass and fix the programmed pre-strain. It should be noted that the thickness of the films decreased from 20 µm to 17–18 µm (measured by micrometer) upon stretching. The strain to failure for these materials was approximately 100%.

2.3. Characterization of Azo-CP2-20

The amorphous nature and strain-induced birefringence of the azo-polyimide network were confirmed by polarized optical microscopy (POM, Nikon). The absorption of the azo-CP2-20 materials was examined with a Cary 5000 UV–Vis spectrometer. A polarizer was inserted in the sample beam and the orientation of the polarization of the probe light was rotated to measure the polarized absorption dependence. The photogenerated stress of the azo-CP2-20 materials

was determined in specimens with gauge length of 6 mm \times 1 mm \times 20 μ m (width, length, and thickness, respectively) measured in tensile grips of a dynamic mechanical analysis (DMA) (RSA III, TA Instruments) at room temperature.

The photomechanical response was also measured by measuring the deflection of a cantilever composed of the azo-CP2-20 material. Bending was directed with linearly polarized 442 nm of a 130 mW helium-cadmium (HeCd, Kimmon) laser. The HeCd laser beam was expanded and collimated with a spherical lens, uniformly exposing the entirety of the cantilever with an intensity of 80 mW cm⁻² unless otherwise noted. The polarization direction of the laser was controlled with a Fresnel rhomb (Newport). The optical setup has been illustrated and described in detail elsewhere.9, <u>11</u> Notably, exposure of both the as-prepared and pre-strained azo-CP2-20 to light of this wavelength does not influence the amorphous nature of the network. Irradiating conventional CP2 (e.g., without azobenzene functionalization) does not induce deflection. The photomechanical bending of azo-CP2-20 cantilevers was monitored with a charge coupled device (CCD). The bending angle was analyzed with a program that determined the tip displacement angle between the mounting point of the cantilever and the outside edge of the tip of cantilever.

3. Results and Discussion

The chemical structure of the amorphous, crosslinked, azobenzene-functionalized CP2 polyimide is illustrated in Figure 1. We have previously reported the photomechanical response of this material upon irradiation with blue-green light.21 The observed photomechanical response of this material is similar to that reported in glassy, polydomain azobenzenefunctionalized liquid crystal polymer networks (azo-LCN).<u>11</u>, <u>12</u>, <u>16</u> In this work, we focus our examination on increasing the magnitude of the photomechanical response by subjecting the azo-CP2-20 sample to pre-strain. Evident in Figure <u>1</u>b, birefringence (evident in the polarized optical micrographs) accompanies the pre-strain indicating alignment of the imide units along the lateral direction of the strain.<u>34</u> The strain-induced birefringence is lost upon heating the material through the glass transition (T_g). Notably, as plotted in Figure <u>1</u>c, pre-straining the azo-CP2-20 sample also aligns the azobenzene crosslinking units along the direction of the strain. Figure 1c is a plot of the magnitude of the absorbance of the azobenzene chromophore at 355 nm (trans conformation, $\pi - \pi^*$) measured with polarized UV–Vis spectroscopy. The 70% prestrained sample is contrasted to the "as-prepared" azo-CP2-20 (AP-azo-CP2-20) sample, which has polarization-independent absorption (e.g., circular) expected of an amorphous polymer. Straining azo-CP2-20 induces a dichroic absorbance (e.g. ellipsoidal), evident in a dichroic ratio of 1.313. We previously have reported that the azo-CP2-20 sample has a T_g of 246 °C and a storage modulus of 2.4 GPa at 30 °C.21

Figure 1

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a) Chemical structure of the azobenzene-functionalized polyimide material examined here-"azo-CP2-20" composed with 20 mol% azobenzene crosslinker. b) Polarized optical micrographs of an azo-CP2-20 sample i) as prepared, ii) after 10% strain, iii) after 35% strain, iv) after 70% strain, and v) after heating the strained azo-CP2-20 sample through the glass transition temperature (T_g). c) Summary of the magnitude of the absorbance of the azo-CP2-20 sample at 355 nm as a function of the orientation of the probe light of a UV–Vis spectrometer to the sample. The as prepared, amorphous azo-CP2-20 has uniform absorbance (•) before stretching. Subjecting the azo-CP2-20 sample to 70% pre-strain aligns the azobenzene chromophores resulting in a dichroic absorbance (\circ).

The photomechanical response both during and after irradiation for the "as-prepared" and 70% pre-strained azo-CP2-20 samples are contrasted in Figure 2, visualized in the cantilever geometry (5 mm × 1 mm × \approx 20 µm). As evident in our prior report,<u>21</u> at an appropriate aspect ratio the AP-azo-CP2-20 exhibits polarization-controlled bidirectional bending evident in Figure 2a-ii and Figure 2a-iv. Adjusting the orientation of the linear polarization of the 442 nm irradiation source to the cantilever geometry allows the strain gradient to be switched from contractile to expansive. However, due to the strong anisotropy of azobenzene within the 70% pre-strained azo-CP2-20 sample irradiation with 442 nm results in a considerable increase in the magnitude of the bend but also exhibits a unidirectional response—similar to that reported in monodomain azo-LCN materials. 9 Perhaps most notably, the response of the materials in the dark are starkly different in that the AP-azo-CP2-20 is "shape recovering" while the prestrained azo-CP2-20 sample is "shape memory" in that it retains the optically generated strain profile. The ability to generate "shape memory" responses in the pre-strained material systems is not directly correlated to the pre-strain of the material but rather due to the thermal processing employed to pre-strain the material, which serves to generate a large amount of local free volume.<u>35</u> The bent shape depicted in Figure <u>2</u>b-iii has been maintained for more than 3 months time with no observable reduction in angle.

Figure 2

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a) Bidirectional bending of a cantilever composed of azo-CP2-20 (as prepared). i) Before irradiation, ii, iv) after irradiation to 80 mW cm⁻² 442 nm linearly polarized such that the electric field vector is parallel (as in ii) or perpendicular (as in iv) to the long axis (x) of the

cantilever, iii, v) upon removal of the irradiation the cantilever slowly returns to the original, vertical shape (shape recovery). b) A 70% pre-strained azo-CP2-20 sample is irradiated with 80 mW cm⁻² 442 nm linearly polarized such that the electric field vector is parallel to the long axis of the cantilever. i) Before irradiation, ii) during irradiation, and iii) 70 h after irradiation the sample retains the optically generated strain (shape memory).

The dependence of the photomechanical response on the extent of pre-strain of the azo-CP2-20 samples is examined in Figure <u>3</u>. Once again, the photomechanical response was visualized in cantilevers of 5 mm × 1 mm × \approx 20 µm. The as-prepared azo-CP2-20 sample (0% strain) exhibits bidirectional bending, while the 70% pre-strained azo-CP2-20 exhibits polarizationindependent unidirectional bending. At intermediate pre-strains, the sample increasingly begins to exhibit asymmetric bidirectional bending, with the transition beginning around 25% pre-strain. Interestingly, the difference between the magnitudes of the bend between the polarizations is effectively constant from 0%–45% pre-strain.

Figure 3

Open in figure viewer Maximum bending angle measured after 1 h of irradiation to 80 mW cm⁻² of linearly polarized 442 nm irradiation polarized both parallel (E//x) and perpendicular (E//x) to the long axis of cantilevers composed of azo-CP2-20 subjected to 0%–70% pre-strain. Between 25%–35% pre-strain, the sample begins to exhibit asymmetrical bending to irradiation of the two polarization conditions here before exhibiting no dependence on polarization at 70% pre-strain.

We previously have reported on the ability to "fix" optically generated strain in glassy azo-LCN materials.25 Irradiation of a cantilever subjected to mechanical deformation allows for the shape to be fixed. Similar to these previous results, we show in Figure 4 the ability to optically fix a mechanically generated complex shape. As illustrated in the images in Figure 4 and b, the material does not exhibit shape memory in the absence of light nor in the case of the asprepared sample. Evident in Figure 4c, the sample subjected to pre-strain is capable of shape fixing upon irradiation with linearly polarized 442 nm light. Once again, the ability of the sample to exhibit optically fixable shape memory is correlated to the thermal processing of the materials and the introduction of added free volume, rather than directly correlated to the pre-strain.36

Figure 4

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Illustration of optically fixable shape memory cycle and dependence on pre-strain. a) An asprepared azo-CP2-20 sample was mechanically deformed in the absence of irradiation, no shape memory was observed. b) An as-prepared azo-CP2-20 sample was mechanically deformed and subjected to linearly polarized 442 nm irradiation, no shape memory was observed. c) A 70% pre-strained and rapidly quenched azo-CP2-20 sample was mechanically deformed and subjected to linearly polarized 442 nm irradiation. The sample retained the shape of the complex deformation, confirming that mechanically generated shape can also be fixed with light.

The magnitude of the stress generated by blue-green irradiation of azobenzene-functionalized polyimides²² far exceeds that generated in a glassy azo-LCN.<u>36</u> The influence of pre-strain on the magnitude of the stress response is presented in Figure 5. Once again, a 70% pre-strained azo-CP2-20 sample is contrasted to the as-prepared azo-CP2-20 material. Upon irradiation with 100 mW cm⁻² of 488 nm light linearly polarized so that the electric field vector is parallel to the tensile gauge both samples exhibit contractile stress of 0.6 (as prepared) and 1.2 MPa (prestrained). Notably, the magnitude of the stress is doubled by pre-straining the azo-CP2-20 material. As evident in Figure 1c, pre-straining the azo-CP2-20 material aligns the azobenzene chromophore along the direction of the strain. Alignment of the azobenzene units by pre-strain contributes to the magnitude of the stress response in two ways. First, the absorbance of the pre-strained azo-CP2-20 sample increases significantly when the linear polarization of the 442 nm light is parallel to the aligned azobenzene chromophores (analogous to a monodomain azo-LCN<u>9</u>, <u>17</u>, <u>19</u>, <u>20</u>). This increases the "efficiency" of absorbance of a polarized flux of photons to transduce into mechanical work (stress). Second and perhaps more importantly, the directionality of the photogenerated stress is concentrated into the same direction (e.g., along the gauge length) due to the alignment of the azobenzene units. Pre-strain of the material thus enhances the cooperative motion necessary to transduce light into work evident here in a twofold increase in photogenerated stress.

Figure 5

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Determination of photogenerated stress in (•) as-prepared azo-CP2-20 sample and (\circ) in 70% pre-strained azo-CP2-20 sample upon irradiation with 100 mW cm⁻² 488 nm light polarized such that the electric field vector was parallel to the gauge in dynamic mechanical analyzer. The samples were subjected to an 3 × 10⁻⁵% strain in tensile grips.

4. Conclusion

A glassy, azobenzene-functionalized polyimide material (azo-CP2-20) was subjected to thermally processing to examine the influence of pre-strain on the photomechanical response. Pre-straining the azo-CP2-20 material serves to align both the imide units (evident as stressinduced birefringence) as well as the azobenzene chromophores (evident as dichroic absorption). Due to trapped free volume within the thermally processed materials, shape memory behaviors are demonstrated—including the ability to fix optically and mechanically generated strain in pre-strained azo-CP2-20 samples. Perhaps most importantly, the already exceptional magnitude of the photogenerated stress measured in the as-prepared azo-CP2-20 materials is doubled by pre-strain, which serves to align the azobenzene units in the material, which improves the coordination of the light-to-work transduction.

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