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PAPER

## Dependence of photoinduced bending behavior of diarylethene crystals on irradiation wavelength of ultraviolet light

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Received 00th January 20xx,  
Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

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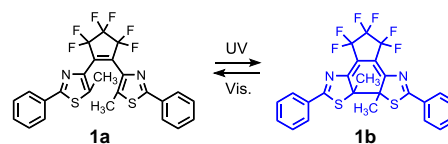
The dependence of the photoinduced bending behavior of diarylethene crystals on the ultraviolet light irradiation wavelength was investigated. When irradiated with 365 nm light, a crystal of 1,2-bis(5-methyl-2-phenyl-4-thiazolyl)perfluorocyclopentene (**1a**) bends toward the incident light. On the other hand, when irradiated with 380 nm light, the crystal of **1a** first bends away from the light source and then bends toward the incident light. To explain this bending behavior, we propose a comprehensive mechanism based on the depth of the photochromic reaction from the crystal surface. This mechanism is successfully supported by the change of cell parameters associated with the photochromic reaction upon irradiation with 380 nm light, which was determined by *in situ* X-ray crystallographic analysis.

### Introduction

Materials that can change their shape in response to external stimuli are being extensively explored for applications in molecular robotics and artificial muscles.<sup>1,2</sup> In particular, research on photomechanical crystals has attracted much attention because they not only have the potential to be used as photoactuators, but they also have well-defined crystal structures that enable the actuation mechanism to be easily elucidated. Research on photomechanical crystals can thus provide an enormous wealth of information for future applications.<sup>3–20</sup>

Diarylethenes are well known as photochromic compounds that reversibly change their chemical and physical properties upon alternating irradiation with ultraviolet (UV) and visible light, and which can undergo photochromic reactions even in the crystalline phase as well as in a solution.<sup>21,22</sup> Diarylethene derivatives have been expected for application in optical memories,<sup>23</sup> optical switches,<sup>24</sup> optoelectrical devices,<sup>25</sup> and photoresponsive surfaces.<sup>26–30</sup> Diarylethene derivatives are also one of the best studied compounds that exhibit photomechanical response in the crystalline phase. Diarylethene crystals exhibit various photomechanical responses such as contraction,<sup>31,32</sup> expansion,<sup>33</sup> bending,<sup>31,33–38</sup> twisting,<sup>39</sup> and separation.<sup>40</sup> Especially, bending is a typical motion in the photomechanical response. The mechanical work of the bending behavior in practical applications such as

gearwheel rotation and the lifting of heavy weight has been reported by Morimoto, Irie, and coworkers.<sup>35,36</sup> Recently, we have also reported photoreversible current ON/OFF switching using gold-coated diarylethene crystals.<sup>38</sup> In addition, as a fundamental research, we have reported on the crystal thickness dependence of the photoinduced bending behavior of diarylethene crystals using the bimetal model.<sup>33,37</sup> The bending speed is slower as the crystal becomes thicker. The bending speed is affected by the reaction depth from the surface. Naumov et al. and Bardeen et al. have independently reported on the mathematical treatment of bending motion for other photochromic crystals in terms of the gradients between the reactant and the product.<sup>41,42</sup> In our previous work, it was clarified that the depth of the photochromic reaction from the crystal surface in diarylethenes plays an important role in the photoinduced bending behavior. From this viewpoint, it is considered that more detailed information on the photoinduced bending behavior can be obtained if the depth of the photochromic reaction from the crystal surface can be controlled.



Here we report the dependence of the photoinduced crystal bending behavior of diarylethene crystals on UV light irradiation wavelength. We newly found that the crystal of 1,2-bis(5-methyl-2-phenyl-4-thiazolyl)perfluorocyclopentene (**1a**) shows the different photoinduced crystal bending behavior by changing wavelength of the incident UV light. When irradiated with 365 nm light, the crystal bends toward the incident light. On the other hand, when irradiated with 380 nm light, the crystal first bends away from the light source and then bends toward the incident light. It is ascribed to the difference in the reaction depth of diarylethenes from crystal surface by

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† Electronic Supplementary Information (ESI) available: Crystal shape and molecular packing diagrams of **1a** (Fig. S1), evidence of no thermal effect of the bending behavior (Fig. S2), absorption spectrum of crystal **1a** (Fig. S3), the photoinduced bending behavior of crystal **1a** (Figs. S4 and S5), a change of the cell parameters upon irradiation with 380 nm light (Fig. S6, Tables S1 and S2), and video of the photoinduced bending behavior (Videos S1, S2, and S3). See DOI: 10.1039/x0xx00000x

changing wavelength of the incident UV light. We propose a comprehensive mechanism to explain the bending behavior in this paper. The mechanism is successfully supported by the crystal thickness dependence of the photoinduced bending behavior and the change of cell parameters in unit cell associated with the photochromic reaction upon irradiation with 380 nm light.

## Experimental Section

### General

All solvents used were purified by distillation prior to use. Absorption spectra were measured in *n*-hexane with a JASCO V-560 absorption spectrophotometer. Diffuse reflectance spectra were also measured using the same spectrophotometer equipped with a JASCO ISV-469 integrating sphere.

### Materials

Diarylethene **1a** was synthesized according to a procedure in the literature.<sup>43</sup> Rod-like crystals of **1a** were prepared by recrystallization from *n*-hexane.

### Photomechanical Behavior

The edge of a rod-like crystal was fixed to a glass capillary, and a fluorescent material was painted on the glass capillary. The fluorescence of the glass capillary indicates the onset time of UV irradiation. The photoinduced bending behavior of **1a** crystals was observed using a Keyence VHX-500 digital microscope. UV irradiation was performed using a Keyence UV-LED UV-400/UV-50H (365 nm light) and UV-10 (380 nm light). The light intensity on the crystal surface was 13 mW cm<sup>-2</sup>. Visible light irradiation was performed using a halogen lamp (100 W).

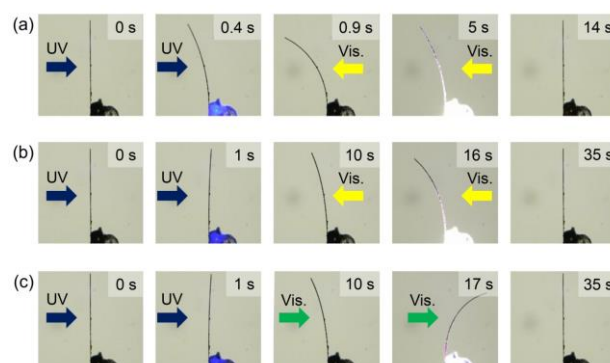
### Change in Cell Parameters

The change in cell parameters of the crystal of **1a** was measured at room temperature using a Rigaku VariMax with Saturn CCD single crystal X-ray diffraction instrument (50 kV, 24 mA) with MoK $\alpha$  radiation ( $\lambda = 0.71073$  Å). The crystal-to-detector distance was 5.5 cm. The data collection, data reduction, and cell refinement were performed with the Assign Unit Cell mode using Rigaku Crystal Clear software. Each diffraction image covered 0.5° in  $\omega$  for 24 s exposure time. The maximum and minimum theta angles of reflections used to measure the unit cell were 27.5 and 2°, respectively. X-ray crystallographic analysis was conducted upon irradiation to crystal **1a** with 380 nm light to follow the change in the cell parameters. The number of reflections used for determination of the cell parameters in each step was 281 (resolution limits: 7.17123 to 0.871013 Å), 299 (resolution limits: 6.9244 to 0.913383 Å), 339 (resolution limits: 7.26774 to 0.928345 Å), 444 (resolution limits: 7.20532 to 0.980169 Å), and 248 (resolution limits: 9.13645 to 1.03237 Å). X-ray crystallographic analysis of crystal **1a** without UV light irradiation was also conducted as a control experiment. The number of reflections

used for determination of the cell parameters in each step was 330 (resolution limits: 6.92206 to 0.78633 Å), 335 (resolution limits: 6.92876 to 0.787275 Å), 342 (resolution limits: 9.03406 to 0.786409 Å), 382 (resolution limits: 6.90882 to 0.776983 Å), and 350 (resolution limits: 6.90844 to 0.810076 Å).

## Results and discussion

### Photoinduced bending behavior of crystal **1a** upon irradiation with 365 and 380 nm light

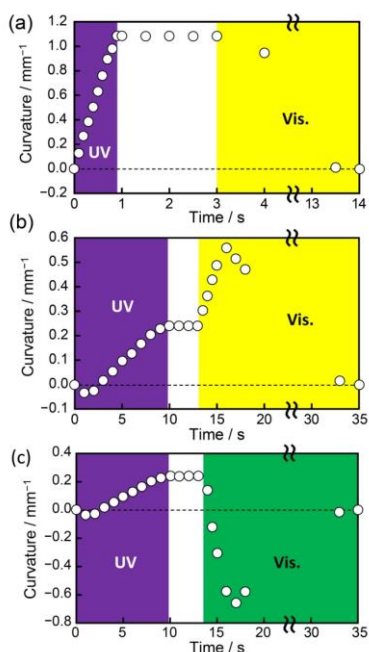


**Fig. 1** Photoinduced bending behavior of crystal **1a** (4.3  $\mu\text{m}$  thick). (a) The crystal was irradiated with 365 nm light from the left side and then irradiated with visible light from the right side. (b) The crystal was irradiated with 380 nm light from the left side and then irradiated with visible light from the right side. (c) The crystal was irradiated with 380 nm light from the left side and then irradiated with visible light from the left side.

Fig. 1 shows the photoinduced bending behavior of crystal **1a**. The crystal shape, face indices, and molecular packing of crystal **1a** are shown in Fig. S1.<sup>38</sup> Crystal **1a** belongs to the monoclinic system and the space group  $P2_1/n$  ( $a = 7.236(2)$  Å,  $b = 25.752(8)$  Å,  $c = 12.611(4)$  Å,  $\beta = 102.432(5)^\circ$  at 123 K).<sup>31</sup> The crystal of **1a** has four molecules in the unit cell and one molecule in the asymmetric unit. Therefore, only a conformation exists in the crystal. When the rod-like crystal was irradiated with 365 nm light onto the (010) face from the left side, the crystal bent toward the incident light. Upon irradiation with visible light from the right side, the crystal returned straight through the same bending trajectory, as shown in Fig. 1a and Video S1. This bending behavior is similar to that presented in previous works.<sup>31,34,36,38</sup> On the other hand, when the rod-like crystal was irradiated with 380 nm light onto the same face from the left side, the crystal first bent away from the light source and then bent toward the incident light after further irradiation. Bending away from the light source is not due to the thermal effect of the irradiation because the bent crystal could maintain its shape even after the light was turned off, as shown in Fig. S2. This unique bending behavior was not observed upon irradiation with 365 nm light, even when the irradiation power was changed. Moreover, upon irradiation to the bent crystal with visible light from the right side, the crystal first bent further away from the light source and then became straight after further irradiation,

as shown in Fig. 1b and Video S2. In contrast, upon irradiation to the bent crystal with visible light from the left side, the crystal returned back and then bent away from the light source. Finally, the crystal returned to the original straight shape upon further irradiation with visible light, as shown in Fig. 1c and Video S3.

Fig. 2 shows the change of curvature relative to the irradiation time. Bending to the left side is defined as a positive. When the crystal was irradiated with 365 nm light from the left side and then irradiated with visible light from the right side, the curvature increased only during UV light irradiation and decreased only during visible light irradiation (Fig. 2a). On the other hand, when the crystal was irradiated with 380 nm light from the left side and then irradiated with visible light from the right side, the change in the curvature became complicated. At first, the curvature became negative and then increased up to a positive region after further irradiation with 380 nm light. Upon irradiation with visible light, the curvature increased again and then gradually decreased to zero (Fig. 2b). In contrast, upon irradiation to the bent crystal with visible light from the left side, the curvature decreased to the negative region and then gradually increased up to zero (Fig. 2c).

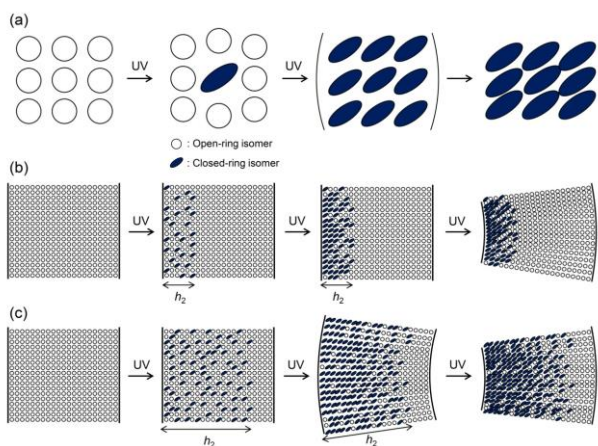


**Fig. 2** Change in the curvature of the crystal of **1a** ( $4.3 \mu\text{m}$  thick) relative to the irradiation time. (a) The crystal was irradiated with 365 nm light from the left side and then irradiated with visible light from the right side. (b) The crystal was irradiated with 380 nm light from the left side and then irradiated with visible light from the right side. (c) The crystal was irradiated with 380 nm light from the left side and then irradiated with visible light from the left side.

### Comprehensive mechanism to explain the bending behavior

To explain all of the observed bending behavior, a mechanism for photoinduced crystal bending is proposed, as illustrated in Fig. 3. When the crystal is irradiated with UV light, photoisomerization from the open-ring isomer to the closed-ring isomer of diarylethene molecules occurs at first randomly. In this condition, repulsion between the open-ring isomers and the photogenerated closed-ring isomers may induce expansion of the photoreacted layer. As the photoconversion of diarylethene molecules increases, the repulsion disappears and Van der Waals interaction between the closed-ring isomers occurs, which induces contraction of the photoreacted layer (Fig. 3a). We have previously reported that the photoinduced bending behavior of diarylethenes can be well explained by Timoshenko's bimetal model.<sup>33,37,38</sup> In the model, it has been clarified that the reaction depth of photoisomerization from the crystal surface ( $h_2$ ) plays a very important role in the crystal bending behavior. When  $h_2$  is very small relative to the crystal thickness, the crystal cannot bend to a significant extent. However, the crystal can bend to a large extent when  $h_2$  is approximately half of the crystal thickness. When irradiated with 365 nm light, photoisomerization of diarylethene molecules occurs only at the crystal surface because of the high absorbance of the dense crystal. This means that  $h_2$  is very small relative to the crystal thickness and the conversion of the photoreacted layer increases quickly. Therefore, initial bending away from the light source is not observed and bending toward the incident light occurs immediately (Fig. 3b). Upon irradiation with visible light, photocycloreversion from the closed-ring isomer to the open-ring isomer occurs simultaneously in the photoreacted layer because  $h_2$  is very small. Therefore, the bent crystal returns to the original straight shape through the same trajectory of bending. In contrast, when irradiated with 380 nm light, photoisomerization of diarylethene molecules can occur deeply within the crystal because diarylethene **1a** has low absorption at 380 nm compared with that at 365 nm, as shown in Fig. S3. This means that  $h_2$  is close to the crystal thickness and conversion of the photoreacted layer proceeds slowly. Therefore, initial bending away from the light source can be observed clearly and subsequent bending toward the incident light occurs slowly (Fig. 3c). Upon irradiation with visible light, the photocycloreversion reaction proceeds only on the irradiated side in the photoreacted layer of the crystal. This results in expansion from the contracted state to the initial state only at the irradiated side. Therefore, when the crystal is irradiated with visible light from the right side, further bending to the left side can occur, as shown in Fig. S4. When the crystal is irradiated with visible light from the left side, then further bending to the right side can be observed.

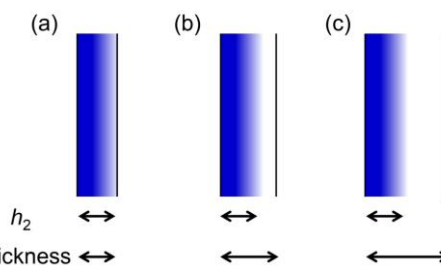




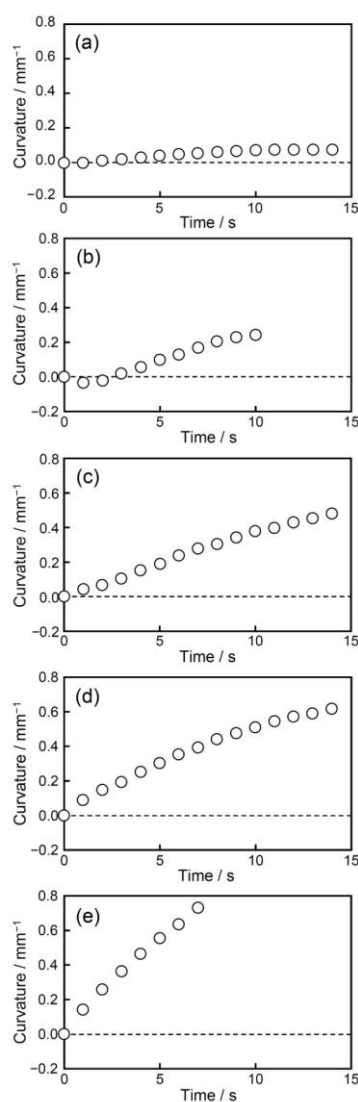
**Fig. 3** Schematic illustration of the mechanism for photoinduced crystal bending. (a) Photoisomerization from the open-ring isomer to the closed-ring isomer in the crystal. (b) Model for bending of the crystal when the crystal was irradiated with 365 nm light. (c) Model for bending of the crystal when the crystal was irradiated with 380 nm light.

#### Crystal thickness dependence of the photoinduced bending behavior

To verify the proposed mechanism, the dependence of the photoinduced bending behavior on the crystal thickness upon irradiation with 380 nm light was investigated. The photoinduced bending behavior is considered to depend on the crystal thickness because the ratio of  $h_2$  to the crystal thickness is changed, as shown in Fig. 4. When the crystal thickness is very thin, UV light penetrates the crystal. As a result, the crystal cannot bend, because photoisomerization of the diarylethene can occur homogeneously throughout the entire crystal (Fig. 4a). When the crystal thickness is slightly thicker than  $h_2$ , the crystal first bends away from the light source, and then bends toward the incident light (Fig. 4b). When the crystal thickness is considerably thicker, the crystal immediately bends toward the incident light (Fig. 4c). Fig. 5 shows the change in the curvature of crystals with various thicknesses upon irradiation with 380 nm light. Pictures of the bending behavior are shown in Fig. S5. When the crystal thickness is 2.9  $\mu\text{m}$ , the crystal cannot bend to a significant extent, because the crystal thickness is very close to  $h_2$ , as shown in Fig. 4a. When the crystal thickness is 4.3  $\mu\text{m}$ , the crystal bends away from the light source at the early stage of photoirradiation and then bends toward the incident light (Fig. 5b). This is ascribed to the appropriate thickness of the crystal, relative to  $h_2$ , to bend away from the light source, as shown in Fig. 4b. In contrast, when the crystal is thicker than 5.5  $\mu\text{m}$ , the crystal bends toward the incident light (Fig. 5c-e), because the crystal thickness is considerably larger relative to  $h_2$ , as shown in Fig. 4c. Thus, all of the photoinduced bending behavior can be explained by the comprehensive mechanism we have proposed.



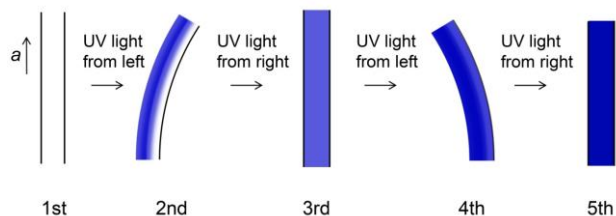
**Fig. 4** Change in the ratio of  $h_2$  to the crystal thickness upon irradiation with 380 nm light for various crystal thickness; (a) close to  $h_2$ , (b) slightly thicker than  $h_2$ , and (c) considerably thicker than  $h_2$ .



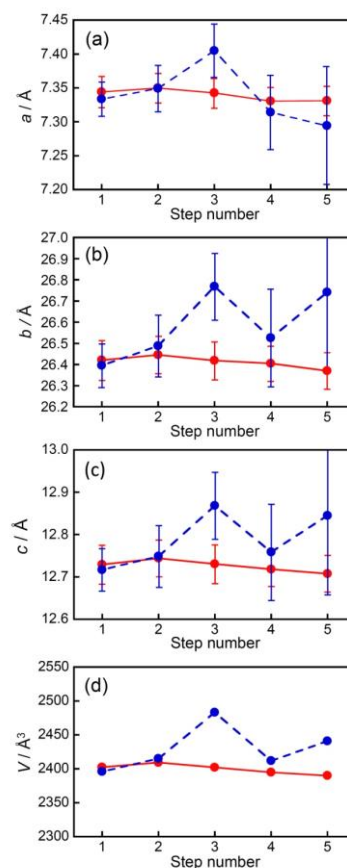
**Fig. 5** Change in the curvature of the crystal **1a** relative to the irradiation time with 380 nm light for crystal thicknesses of (a) 2.9, (b) 4.3, (c) 5.5, (d) 7.6, and (e) 10.3  $\mu\text{m}$ .

### In situ X-ray crystallographic analysis

The change in the cell parameters of the unit cell associated with the photochromic reaction was examined under irradiation with 380 nm light using single crystal X-ray crystallographic analysis. Crystal **1a** has a monoclinic crystal system with the space group  $P2_1/n$ . Fig. 6 illustrates the crystal at each step of the cell parameter measurement under irradiation with 380 nm light. The cell parameters of crystal **1a** were first measured prior to UV irradiation (1st step). The crystal was then irradiated with 380 nm light from the left side. The crystal bent away from the incident light (2nd step). When the bent crystal was irradiated with 380 nm light from the right side, the crystal became straight shape (3rd step). Further irradiation with 380 nm light from the left side caused the crystal to bend toward the incident light (4th step). The bent crystal then became straight upon continued irradiation with 380 nm light from the right side (5th step). The cell parameters at each step are summarized in Tables S1 and S2. Fig. 7 and Fig. S6 show the change in the cell parameters of the unit cell upon irradiation with 380 nm light. The blue and red circles indicate the cell parameters at each step under irradiation with 380 nm light and without UV light irradiation, respectively. While the cell parameters did not change significantly without UV light irradiation, they clearly changed when the crystal was irradiated with 380 nm light. The cell lengths of the  $a$ -,  $b$ -, and  $c$ -axes increased up until the 3rd step and then decreased. This result indicates that the crystal first expanded in all directions and then the  $a$ -axis contracted. The long edge of the crystal is parallel to the  $a$ -axis, as shown in Fig. S1. Therefore, when irradiated with 380 nm light, the crystal first bends away from the light source and then bends toward the incident light. Thus, the single crystal X-ray crystallographic analysis also supports the proposed mechanism.



**Fig. 6** Schematic illustration of the crystal shape at each step during the cell index measurement.



**Fig. 7** Change in the cell parameters of the unit cell relative to the step of irradiation with 380 nm light; (a)  $a$ -axis, (b)  $b$ -axis, (c)  $c$ -axis, and (d) volume. The blue and red circles indicate the cell parameters at each step upon irradiation with 380 nm light and that without UV light irradiation, respectively.

### Conclusion

In conclusion, the dependence of the photoinduced crystal bending behavior of diarylethene crystals was investigated with respect to the UV light irradiation wavelength. Crystal **1a** exhibited different bending behavior with different UV light wavelengths. When irradiated with 365 nm light, the crystal bends toward the incident light. In contrast, when irradiated with 380 nm light, the crystal first bends away from the light source and then bends toward the incident light. We have proposed a comprehensive mechanism to explain their bending behavior based on the depth of the photochromic reaction of diarylethenes from the crystal surface. The proposed mechanism is well supported by the experimental results with respect to the crystal thickness and the change in the cell parameters of the unit cell associated with the photochromic reaction.

## Acknowledgements

This work was partly supported by a Grant-in-Aid for Scientific Research on Innovative Areas "Photosynergetics" (No. 26107013) from the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan. D.K. appreciates Research Fellowships of the Japan Society for the Promotion of Science (JSPS) for Young Scientists.

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