



Reusable dismantlable adhesion interfaces induced by photodimerization and thermo/photocleavage reactions

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Abstract

Controlling adhesion strength during and after the use of bonded materials is crucial. Dismantlable adhesives play important roles in material recycling. However, typical dismantlable adhesives rely on the bulk properties of the adhesive, and specific control of dismantling behavior is challenging. Here, we successfully demonstrated the reusability of a dismantlable adhesion interface system, in which a cleavable molecular layer forms at the adhesion interface through reversible dimerization and cleavage reactions of anthracene. The adhesion peel test was conducted repeatedly by forming a cleavable layer on the substrate surface of the specimens. Strong bonding in the initial state and easy dismantling after stimulation were achieved even in the reused layer. Our strategy for constructing a dismantlable adhesion interface holds promise in material recycling.

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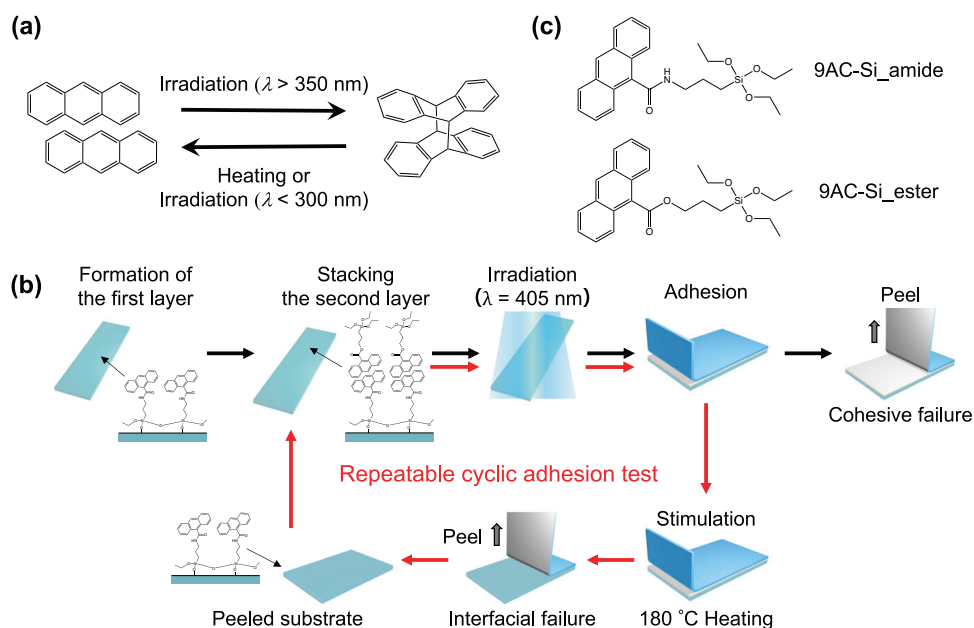
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Introduction

Adhesive bonding is essential for fabricating multimaterials comprising metals, ceramics, and plastics, and adhesion science and technology have become increasingly important in interdisciplinary fields [1]. Dismantlable adhesive systems are smart in the material recycling field that enable strong bonding during utilization and rapid reduction in adhesion strength on demand [2, 3]. The design of dismantlable adhesive materials requires a change in adhesive properties in response to external stimuli [4–6]. Various types of stimuli, including heat [7–10], light [11–17], electric fields [18], magnetic fields [19], and chemical treatment [20, 21], have been employed in dismantlable adhesives. In particular, repeated adhesion has been achieved with heat- and light-based dismantlable adhesives [10, 13–17], which is a strong point for material recycling. However, these dismantling processes mainly deform the adhesive in bulk, making control of the adhesion strength between the bonding and the debonding state quite difficult. In addition, this debonding process causes another problem: a long reaction time is needed to switch the adhesion strength.

A different approach to controlling adhesive strength is to focus on the adhesive interface. This approach, based on the fact that the interface state is also important in the adhesion mechanism [22, 23], is expected to solve the abovementioned problems. Recent reports have indicated that the presence of organic layers at the interface affects

Fig. 1 **a** Dimerization and cleavage of anthracene molecules. **b** Schematic of the preparation of molecular layers and the repeated adhesion test. The specimen peeled after stimulation was easily dismantled at the cleaved molecular layer, and the molecules on the substrate surface were reutilized for the next adhesion test. **c** Chemical structures of the compounds used in this study



adhesion properties [24, 25]. In addition, in the field of hydrogels, adhesive strength can be modified by focusing on bonding at the interface [26–28]. However, the concept of selectively controlling the state of the adhesive interface by applying stimuli to impart dismantlability has not been reported owing to the difficulty of precisely controlling the adhesion interface.

We have developed a dismantling system focusing on the change in chemical bonding at the adhesive interface [29–32]. This system utilizes a cleavable molecular layer based on the thermal/photocleavage reactions of anthracene molecules under external stimuli (Fig. 1a). The adhesion strength is controlled at the adhesion interface through changes in the bonding state in response to heat or light. In the dimeric state, the chemical bonds of anthracene contribute to the adhesive strength, similar to conventional primers. However, after the chemical bonds are cleaved by stimulation, separation occurs in the molecular layer at the adhesion interface, and the adhesion strength decreases. Previously, we utilized predimerized molecular layers because we focused only on the dismantling of adhesion [31]. We also successfully demonstrated a reversible chemical bond change based on the cycloaddition reaction of anthracene in the laminated molecular layer [29]. In addition, we found that anthracene monomers remained on the substrate surface after peeling [31].

Here, we studied the repeatable dismantling of an adhesion interface based on the reversible chemical bond changes in the molecular layer of anthracene. To investigate the effect of the chemical bonds in the molecular layer on adhesion strength, adhesion peel tests were conducted before and after applying a stimulus to induce cleavage reactions. Then, a second layer of anthracene molecules was

laminated to the peeled substrate and photodimerized. The adhesion peel test was then performed to evaluate whether the anthracene components present on the substrate surface after peeling could be reutilized (Fig. 1b). In this study, by investigating the repeated utilization of dismantlable molecular layers, we examined both the versatility of this material system and the importance of the role of chemical bonding at the interface in adhesion behavior.

Experiment

Materials

Figure 1c shows the chemical structures of the materials utilized in this study. All chemicals were purchased from Tokyo Chemical Industry Co. (Tokyo, Japan) or Fujifilm Wako Pure Chemical Co. (Osaka, Japan). Unless otherwise stated, they were utilized without further purification. N-(9-Anthracene carbonyl)aminopropyltriethoxysilane (9AC-Si_amide) and 9-anthracene carboxylic acid-3-(triethoxysilyl)propyl ester (9AC-Si_ester) were synthesized following previously reported methods [29].

Cleaning substrates

Glass substrates and quartz substrates were purchased from AS ONE Corporation (Osaka, Japan). Both substrates were cleaned with a 2.0 wt% neutral detergent (Cica Clean LX-II, purchased from Kanto Chemical Co., Inc., Tokyo, Japan) in water via sonication. Subsequently, the same procedure was conducted in pure deionized water (Milli-Q grade, 18 M Ω -cm). The cleaned substrates were immersed in an alkali

detergent (KOH:ethanol = 1:10) for 1 day. After that, the substrates were ultrasonically rinsed four times in deionized water for 20 min. Prior to use, the cleaned substrates were dried under vacuum for 1 h to remove residual absorbed water.

Preparation of the molecular layer

The formation of molecular layers was performed according to a procedure detailed in a previous report [29]. To form the first layer, cleaned and completely dried substrates were immersed in a 9AC-Si_{amide} solution (4 mmol/L, dry CH₂Cl₂:dry toluene = 1:1) for 1 h at 25 °C. Then, the substrates were slowly removed from the solution and heated at 110 °C for 30 min. The resultant substrates were rinsed with chloroform and dried under ambient conditions. Subsequently, a 9AC-Si_{ester} solution (0.5 wt%, chloroform) was cast to form the second molecular layer on the substrates on which the first molecular layer had already been formed. After drying at 40 °C for 5 min, the substrates were photoirradiated for 20 min with a light-emitting diode (AC8375-405, $\lambda = 405$ nm, 5 mW/cm²; CCS, Inc., Kyoto, Japan). The resultant substrates were rinsed with chloroform and thoroughly dried under ambient conditions. The efficiency of the conversion of anthracene molecules to photodimers was estimated by $(1 - A_{405}/2A_1) \times 100$, where A_{405} and A_1 refer to the absorbance of the anthryl groups at 366 nm after photoirradiation with 405 nm light and of the initial layer, respectively.

Thermal cleavage and photocleavage

The substrate with the fabricated molecular layer was heated on a hot plate (MS-H280-Pro, DLAB, Beijing, China) to thermally cleave the chemical bonds of the anthracene dimers. The photocleavage reaction was induced by UV light irradiation at 280 nm with a light-emitting diode (AC8375-280, 10 mW/cm²; CCS, Inc., Kyoto, Japan). The efficiency of the cleavage reaction was estimated by $(A_H/2A_1) \times 100$ or $(A_{280}/2A_1) \times 100$, where A_H and A_{280} refer to the absorbance of the anthryl groups at 366 nm after heating at 180 °C for 1 min and photoirradiation with 280 nm light for 1 min, respectively.

Measurement

UV-vis spectroscopy and fluorescence spectrometry were performed as described in a previous report [32] with a V-670 spectrometer (JASCO Co., Tokyo, Japan) and an FP-8550 spectrometer (JASCO Co., Tokyo, Japan).

The adhesion peel tests were performed at 25 °C with a tensile test machine (A&D Co., Ltd. STB-1225L, RTI-1310) equipped with an accessory for 90° peel tests (A&D Co., Ltd. J-PZ10-1kN) and a 1 kN loading cell. The test specimens

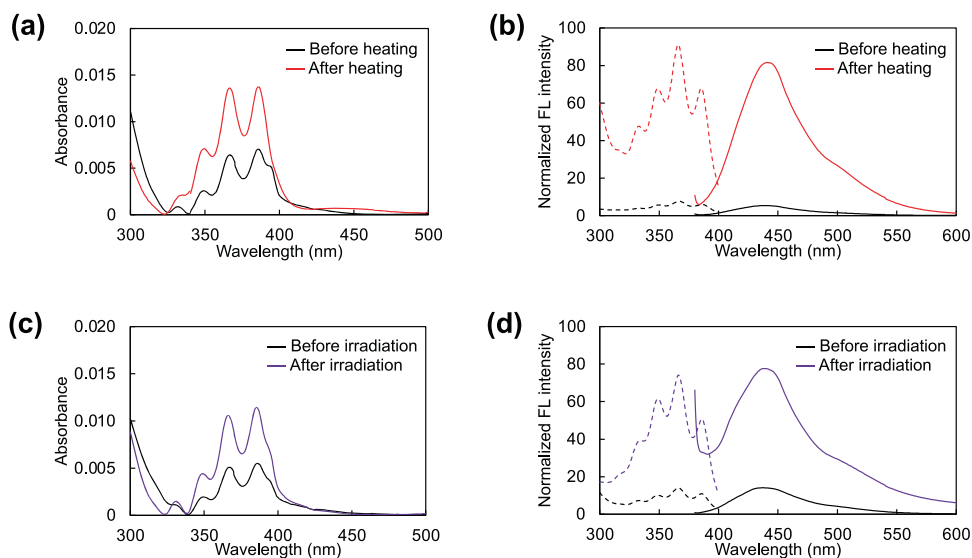
were prepared with glass or quartz substrates (75 × 25 mm²) with or without the deposited molecular layer. The process of preparing specimens for adhesion peel tests was described in a previous report [31]. A hydrophilic polyester film (9901 P, thickness of 100 μm; 3 M Japan Ltd., Tokyo, Japan) was adhered to the substrate using an adhesive (TB1530-150; ThreeBond Co., Ltd., Tokyo, Japan). Subsequently, the adhered specimens were maintained at 25 °C in an environment with a humidity >50% for 24 h. Before the adhesion peel test, a stimulus to induce cleavage reactions, such as heating or light irradiation, was applied from the substrate side. Quartz substrates were selected as the sample for light irradiation. The specimens for the second and third tests were prepared by casting a 9AC-Si_{ester} solution to form the second layer on the peeled substrate, performing photodimerization, and rinsing with chloroform before bonding to a polyester film. The peel rate for the 90° peel test was 50 mm/min. The adhered area was set to 30 × 10 mm², and the thickness of the adhesive layer was set to 100 μm. The unit of peel strength was N/10 mm, based on the width of the specimens. The reported values are averages of five measurements with standard deviations.

Results and discussion

Confirmation of the cleavage reaction

The cleavage conditions of the molecular layers were evaluated. The composition of the molecular layer was designed based on our previous research [29] for repeated use as a first and second layer. An anthracene molecule with amide groups (9AC-Si_{amide}) and an ester group (9AC-Si_{ester}) were selected because this configuration was found to perform reversible and repeatable bond formation and cleavage reactions. The molecular layer of these photodimers was formed by photoirradiation with 405 nm light. As demonstrated in Fig. 2a, the band's absorbance in the 300–400 nm range of the spectrum, attributed to the π - π^* transition, increased after heating at 180 °C for 1 min. These heating conditions are the same as those previously reported for thermal cleavage reactions [32]. Anthracene photodimers show no absorbance at 300–400 nm owing to the absence of a conjugated system [33]. Thus, the increase in the absorption peak indicates the formation of monomers by the heating process, indicating the progress of the thermal cleavage reaction. In addition, from the ratio to the absorption spectra of the first layer (Supplementary Fig. S1), the efficiencies of the photodimerization and cleavage reactions were calculated to be 70% and 72%, respectively. These values are in reasonable agreement with other research results [34, 35]. The fluorescence characteristics of the molecular layer before and after heating were

Fig. 2 **a** UV–vis absorption spectra and **b** fluorescence excitation (left, dashed line) and emission (right, solid line) spectra of the molecular layer before and after heating at 180 °C for 1 min. **c** UV–vis absorption spectra and **d** fluorescence excitation (left, dashed line) and emission (right, solid line) spectra of the molecular layer before and after irradiation with 280 nm light for 1 min. The excitation and emission wavelengths were 365 nm and 420 nm, respectively



also analyzed. Both the fluorescence emission and excitation spectra indicated an increase in fluorescence intensity upon stimulation (Fig. 2b). However, the shapes of these spectra deviated from mirror-image symmetry: the excitation spectrum showed several peaks characteristic of anthracene monomers, whereas the emission spectrum showed a broad peak with a maximum at a wavelength of 440 nm. The fluorescence peak of anthracene molecules differed owing to surrounding conditions, such as the concentration and state of matter, and excitation below and above 420 nm led to emission from the monomer and excimer anthracene, respectively [36–38]. As previously reported, the molecular layer formed an excimer after the stacking of a second layer (9AC-Si_ester) [29]. Thus, even after thermal cleavage, the photodimer molecules did not become isolated in a completely monomeric state, and fluorescent emission occurred in the long-wavelength region. In addition, the broadness of the fluorescence peak suggested that the emission must involve multiple molecules interacting with one another. Photocleavage was measured similarly, and peaks characteristic of the anthracene monomer or excimer were observed in both the absorption and fluorescence spectra (Fig. 2c, d). These results proved that the thermal and photocleavage reactions of this molecular layer proceeded after as little as 1 min of stimulation. However, the reaction efficiency of photocleavage was only 57%, which was lower than that of thermal cleavage. It can be assumed that the generation of byproducts by irradiation with 280 nm light affects the reaction efficiency [39].

Dismantling of the adhesion test specimen with the molecular layer

To investigate the change in adhesive strength as a function of the bonding state of the molecular layers, a 90° peel test

was performed. The test method was the same as that used in a previous study [31], and the peel strength was evaluated by measuring the force needed to pull off the adhered film from the glass substrate. The average peel strength was measured in the plateau region in the tensile force graph (Supplementary Fig. S2) and compared according to the type of applied stimulus and the presence or absence of the molecular layer. For the control experiment, a specimen without a molecular layer was used. When peeling without applied stimuli, the peel strength higher when a molecular layer was present (Fig. 3a). In the case of a substrate with a molecular layer, the peeled specimen showed the same failure mode as the previous specimen, in which the adhesive residue remained on the peeled substrate surface (Fig. 3b). Compared to that obtained in our previous study [31], the adhesive strength was approximately 1.4 times greater. The most likely reason for this difference is that the method of preparing the adhesive specimens was changed to perform repeated tests. The adhesive thickness of the specimens was slightly greater than before, which increased the peel strength owing to the increased cohesion of the adhesive. However, even at this peel strength, a cohesive failure mode was observed in the specimen with the molecular layer. Cohesive and/or interfacial failure occurs according to the balance between the cohesiveness of the adhesive and the adhesive strength at the interface [40]. Therefore, owing to the molecular layer formation, the adhesive strength at the interface clearly exceeds the 14 N measured here.

Next, we investigated the effect of the cleavage of anthracene dimers in the molecular layer on the peel strength. The same heating and light irradiation conditions were applied. The peel strength measurement using a control test specimen without the molecular layer confirmed that these external stimuli had no effect. In contrast, for the

Fig. 3 **a** Average peel strengths of the first adhesion test in the 10–25 mm extension range under different conditions. Error bars represent the standard deviation. **b** Photographs of specimens peeled after processing under different conditions

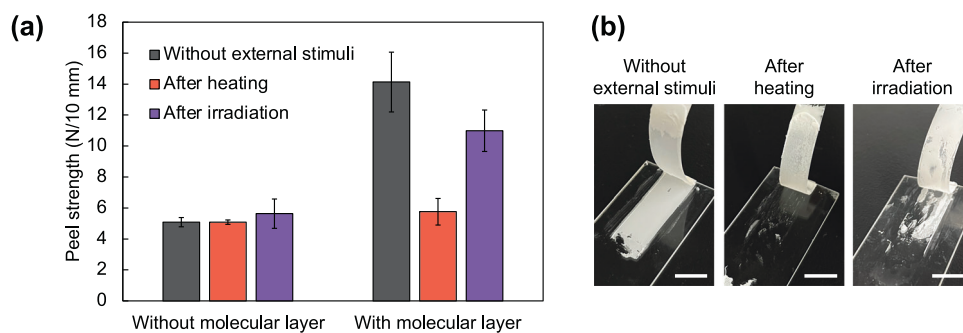
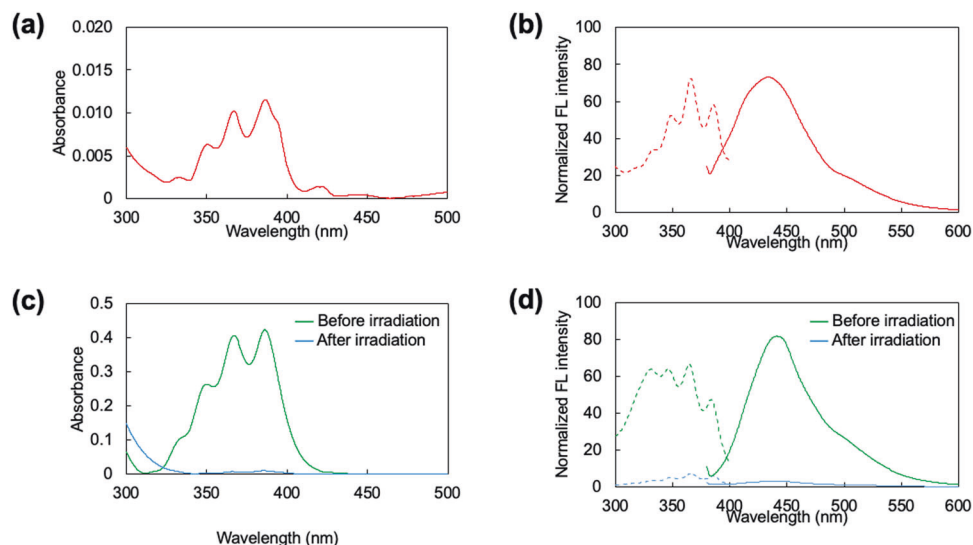


Fig. 4 **a** UV–vis absorption spectra and **b** fluorescence excitation (left, dashed line) and emission (right, solid line) spectra of the peeled specimen substrate after heating at 180 °C for 1 min. **c** UV–vis absorption spectra and **d** fluorescence excitation (left, dashed line) and emission (right, solid line) spectra of the second stacked layer before and after irradiation with 405 nm light for 20 min. The excitation and emission wavelengths were 365 nm and 420 nm, respectively

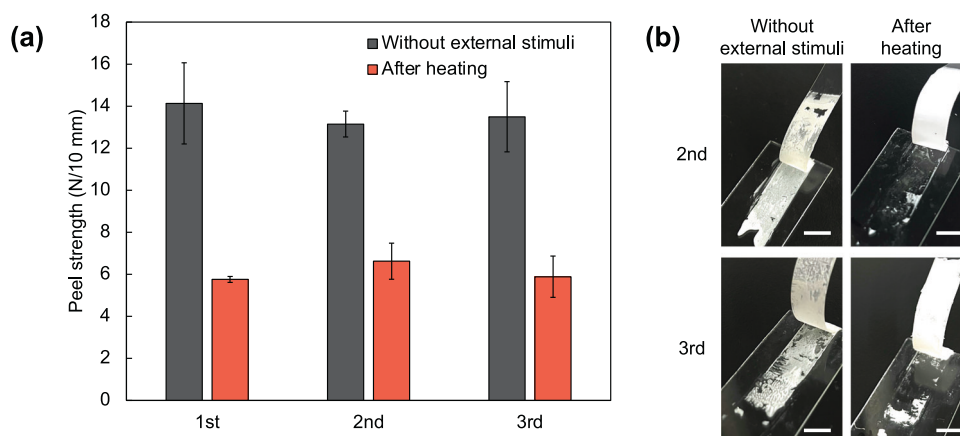


substrate with the molecular layer, the peel strength decreased by 59% in the sample peeled after heating at 180 °C for 1 min compared to that without stimulation (Fig. 3a). The peel strength shown here was 1.4 times greater than that of the molecular layer formed utilizing presynthesized photodimers. This can be attributed to the relatively low packing density of the molecular layer because the reaction efficiency of photodimerization was not 100%. However, the failure mode changed from cohesive to interfacial failure, as shown in Fig. 3b. If peeling occurs in the molecular layer, anthracene monomers generated by thermal cleavage will be present. UV–vis absorption spectroscopy of the peeled glass substrate after heating showed a characteristic absorption peak corresponding to the anthryl groups (Fig. 4a). This indicates that a molecular layer composed of anthracene molecules remained on the peeled glass substrate. The absorbance of the peeled glass substrate was 108% of that of the first molecular layer. Considering that the reaction efficiencies did not reach 100% in either the photodimerization or thermal cleavage reactions, the detected anthracene peak was not only from the thermal cleavage of the molecular layer but also from the laminated molecules remaining on the substrate surface or from mechanically cleaved

photodimers. The fluorescence spectrum was measured to investigate the state of the anthracene molecules on the peeled substrate surface, and a broad fluorescence peak was observed (Fig. 4b). Compared with that in the fluorescence spectrum of the thermally cleaved molecular layer (Fig. 2b), the maximum peak was blueshifted by 7 nm (440 nm to 433 nm). Intermolecular interactions based on the geometry of anthracene molecules change the wavelength of the fluorescence peak [37]. Because the fluorescence emission appears at shorter wavelengths with fewer interactions, the blueshift of the fluorescence peak indicates that the molecular state of the substrate surface after peeling is more monomeric than that after thermal cleavage of the dimerized molecules on the substrate surface alone. Notably, mechanical peeling occurs in the molecular layers due to the cleavage of the anthracene dimers. However, a shoulder band was observed at 510 nm, which originates from the emission of the anthracene excimer [41]. This indicated that even after peeling, the layer is not completely monolayer, and excess deposited component of the second layer still remains.

The effect of the photocleavage reaction on the dismantling system was also examined by irradiation with 280 nm light. The average peel strength was 22% lower in

Fig. 5 a Average peel strengths obtained in repeated adhesion peel tests in the 10–25 mm extension range under different conditions (without any stimulus and after heating). Error bars represent the standard deviation. **b** Photographs of specimens in the second and third tests that were peeled without any stimulus or after heating



the experimental group than in the unstimulated group (Fig. 3a). As illustrated in Fig. 3b, compared with that of the specimens without external stimuli, the percentage of the area of interfacial failure increased; however, part of the adhesive remained on the substrate surface. This behavior was also observed in our previous study [31], suggesting a decreased efficiency of the cleavage reaction by photoirradiation. Although light irradiation induced adhesion disassembly, the effect is weaker than that of the thermal stimulus. Therefore, in repeated peeling tests, we utilized heat as the stimulus, which resulted in interfacial peeling on almost all the surfaces.

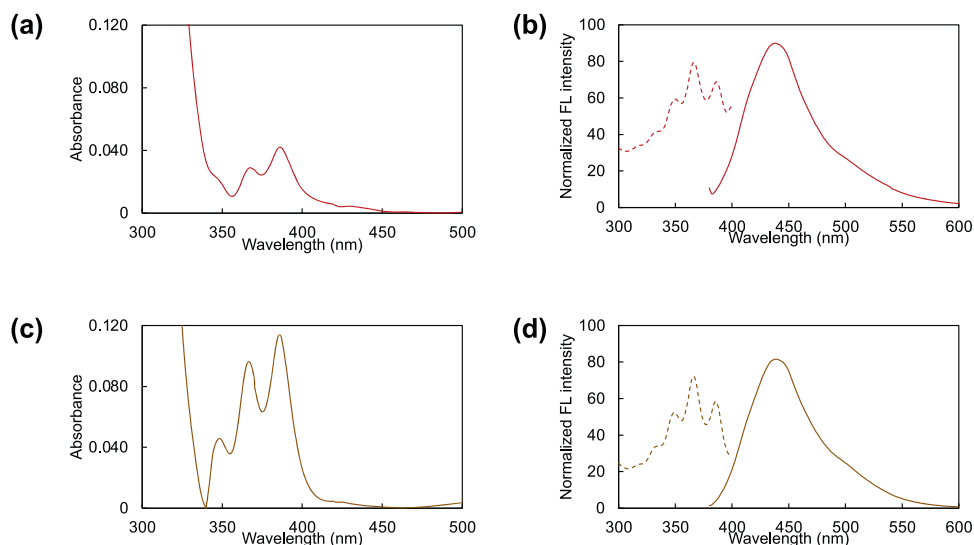
Effect of the molecular layer on repeated adhesion tests

The reusability of the interfacial dismantling system was investigated through three cycles of peeling tests with the reattachment of specimens. Figure 1b shows a schematic of the repeated cyclic adhesion test procedure. For repeated adhesion peel tests, 9AC-Si₂ester was adsorbed on the substrate of the peeled specimen after heating, and a molecular layer was formed by photodimerization. The UV–vis absorption spectra of the substrate showed a large increase in absorbance after the second layer of 9AC-Si₂ester was applied (Fig. 4c). As demonstrated in Fig. 4d, we measured the fluorescence spectra of this substrate via this procedure. After the addition of the second layer, a broad emission peak appeared at a maximum of 441 nm, indicating an 8-nm redshift compared to that of the specimen peeled after heating (433 nm). This indicates that stacking layers increases the intermolecular interactions between anthracene monomers. In addition, a shoulder band at approximately 510 nm was observed, corresponding to the formation of an anthracene excimer, which indicates that the neighboring anthracene molecules are close enough to form a dimer photochemically. In fact, 20 min of photoirradiation with 405 nm light drastically decreased the

intensities of all the absorption peaks in the 350–400-nm range, the fluorescence emission peak, and the fluorescence excitation peak. This indicates that photodimerization occurs through the molecular layer adsorbed on the substrate of the peeled specimen.

Figure 5 shows the results of the repeated peel tests. The second test was conducted with the specimen peeled after heating in the first test. The third test was performed with the specimen peeled after heating in the first and second tests. These samples were peeled again after the molecular layer was formed and bonded. Two specimens were compared here: after bonding the substrate to the molecular layer, one was peeled without heating, and the other was peeled after heating again at 180 °C for 1 min. From the obtained tensile force plots (Supplementary Fig. S3), the average peel strength was calculated (Fig. 5a). First, we compared the peeling results without heating. The second and third results indicated that the peel strength was close to that of the first one, and the failure mode was cohesive peeling (Fig. 5b). This suggests that the molecular layer can function repeatedly as a primer. The peel test after heating showed much lower peel strengths in the second and third tests than in the absence of stimulation. Compared to the peel strength of the specimen without stimulus during each test, the peel strength decreased by 50% for the second test and 56% for the third test (Fig. 5a). The failure mode was generally interface peeling (Fig. 5b), confirming that the cleavage reaction affected the peeling test. A comparison of the failure modes in repeated adhesive peel tests indicated that the percentage of adhesive residue on the peeled specimen differed with and without stimulation at all steps (Supplementary Fig. S4). In the first test, the amount of adhesive residue on the substrate peeled after heating was less than 5%; in the second and third tests, the residual area ratio of the adhesives increased, ranging from 10.6% to 33.5%. However, after peeling without heating, the amount of adhesive residue was more than 75%, indicating that a clear difference occurred depending on the stimulation.

Fig. 6 (a, c) UV–vis absorption spectra and (b, d) fluorescence excitation (left, dashed line) and emission (right, solid line) spectra of the peeled specimen substrate after heating at 180 °C for 1 min in the second (a, b) and third (c, d) peel tests. The excitation and emission wavelengths were 365 nm and 420 nm, respectively



UV–vis and fluorescence spectra of the peeled specimens revealed that anthracene monomer remained on the substrate in both cases (Fig. 6). However, the absorbance was higher than the theoretical value calculated from the reaction efficiency of thermal cleavage. In addition, the absorbance increased after the second and third tests. Considering these facts, this heightened increase in absorbance is due to the gradual deposition of molecular layer components on the substrate surface, forming multiple layers through repeated adhesion peeling tests. Indeed, the fluorescence emission spectra showed a maximum peak at 438 nm in the second and third tests. This redshift was greater than that in the first test, indicating an increase in the intermolecular interactions between anthracene molecules. However, the deposition of the molecular layer detected here did not affect the ability of this system to increase both the adhesion strength in the initial state as a primer and the dismantling ability after heating.

Conclusion

A repeatably dismantlable adhesion interface system was achieved by utilizing a compound that enables chemical bond formation between molecular layers. The molecular layers were cleavable under both heat and light irradiation; however, heating was more suitable for repeated adhesion tests, as determined from the failure mode of the specimens. Anthracene molecules were present on the substrate after peeling, and the adhesion test was successfully repeated more than three times. Here, we confirmed that the molecular layer, which had been cleaved and peeled off, could be reused as a primer in the dismantling system. We believe that these findings provide new insight for the development of dismantlable adhesion technology.

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Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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