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ORIGINAL ARTICLE

On-demand easy peeling of acrylic adhesives containing ionic liquids through a microwave irradiation stimulus

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Abstract

We prepared microwave-responsive "on-demand-peeling" adhesives simply by mixing second-generation acrylic adhesives with ionic liquids. A rapid response (<30 s for adhesive failure) to microwave irradiation was achieved using a microwave oven. This "on-demand-peeling" was attributed to local heating of the ionic liquid in the adhesive by microwave irradiation. In addition, the adhesive strength was not influenced by the ionic liquid additives without microwave irradiation. We revealed that the local heating and the plasticizing effect of the ionic liquid were the cause of the "on-demand-peeling". Our preparation method for these microwave-responsive adhesives is simple, conventional, and attractive for the fundamental and industrial fields.

Introduction

Adhesion is a crucial technology in the industrial processing of final products. From the perspective of product safety and credibility, high adhesive strength and durability are significant attractive properties. Excellent adhesive properties are accessible, and the process of applying adhesives is simple. Therefore, adhesion is widely applicable in our daily life as well as in a wide range of industrial fields such as food packaging, sports gear, laminated films, electronic devices, electronic appliances, automobiles, aircrafts, and buildings [1]. Second-generation acrylic (SGA) adhesives have been widely accepted due to their simple rapid processing through the static mixing of two components in disposable cartridges [2, 3]. The adhesive strength of SGA adhesives is reliable and high for industrial applications, while SGA adhesives have disadvantages in terms of the recycling and dismantling of adhered products.

For environmental reasons, the recycling of industrial products and ecofriendly components is currently in high demand, and adhesives are no exception. In addition, stimuli-responsive "on-demand-peeling" adhesives are desirable to enable the rapid, neat dismantling of products after the application of stimuli [4–21]. Various stimuli for "on-demand-peeling" adhesives have been reported, e.g., UV or visible light [5–13], heat [14–18], electricity [19, 20], or a magnetic force [21]. Light and heat are conventional in daily life, and accidental dismantling triggered by these stimuli may occur. In the case of a light stimulus, optical transmittance is required for the substrates and adhesives. A heat stimulus can also cause damage to the adhered products. Electricity and a magnetic force are unusual as daily stimuli but are limited to the adhered substrates. Moreover, stimuli-responsive adhesives are synthesized through multiple steps, which leads to significant disadvantages when applied in the industrial field. Therefore, "on-demand-peeling" adhesives activated by an unusual, simple non-damaging stimulus from a conventional apparatus is desirable.

Ionic liquids (ILs) have attracted much attention in the synthetic, electronic, and biological fields [22–25]. Various ILs have been synthesized with a combination of organic anions and cations. The well-known functional properties of ILs are non-volatility, non-flammability, high ionic density, ion conductivity, high electric permittivity, reusability as "green" solvents, and solubility for cellulose [26–32]. It is also reported that ILs with high electric permittivity are heated by irradiation of microwaves [33–35]. This heating

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of ILs originates from molecular rearrangements and collisions in the dipolar polarization mechanism and conductive mechanism [36, 37]. Microwave-assisted local heating has been the subject of research on media in organic reactions and nanoparticle-coated ILs [38–40].

Here, we focused on the microwave-assisted heating of ILs and microwave-responsive "on-demand-peeling" adhesives. When the size of the adhered specimen is large, heating the whole specimen is difficult, whereas selective local heating in a microwave oven can limit the heated region of the adhesives. Thus, selective heating minimizes damage to the products. To heat adhesives via irradiation with microwaves, ILs were added to SGA adhesives without any synthetic process. Under this microwave irradiation, the lap shear strength was evaluated.

Experimental

Materials

SGA adhesive (HARDLOC NS-700S20) was kindly provided by Denka Co., Ltd. 1-Butyl-3-methylimidazolium thiocyanate (BMIM-SCN), trihexyltetradecylphosphonium chloride (THTDP-Cl), and 1-butyl-1-methylpyrrolidinium bis(trifluoromethanesulfonyl)imide (BMPY-TFSI) were purchased from Sigma-Aldrich. 1-Butyl-3-methylimidazolium hexafluorophosphate (BMIM-PF_6), 1-butyl-3-methylimidazolium tetrafluoroborate (BMIM-BF_4), 1-butyl-3-methylimidazolium iodide (BMIM-I), and 1-butyl-3-methylimidazolium bromide (BMIM-Br) were purchased from TCI Co., Ltd. Tributylmethylammonium bis(trifluoromethanesulfonyl)imide (TBMA-TFSI) and 1-ethyl-3-methylimidazolium thiocyanate (EMIM-SCN) were purchased from Wako Pure Chemical Industries, Ltd. 1-Allyl-3-butylimidazolium bis(trifluoromethanesulfonyl)imide (ABIM-TFSI) was purchased from Kanto Chemical Co., Inc. Polyimide film (Kapton® 500H/500V) was purchased from the Du Pont-Toray Co., Ltd. All chemicals were used as received without further purification.

Microwave-assisted heating of ILs

Microwaves were irradiated onto ILs in glass capillaries at 2.45 GHz and 500 W for 30 s in a microwave oven (DR-Y22, TWIN BIRD). BMIM-SCN was employed as the IL in the adhesive samples for the microwave-assisted heating test because it can be rapidly heated.

Preparation of adhered samples of SGA adhesives containing BMIM-SCN (IL-SGA)

BMIM-SCN was added to SGA adhesives with varying content, and adhesives containing BMIM-SCN (IL-SGAs) were prepared. The concentrations of BMIM-SCN were 0, 5, 7, 10, 15, 16.5, and 20 wt% vs. adhesives. After the IL-SGAs were applied to polyimide films, other non-applied polyimide films were overlapped with the application side of the polyimide films. The adhered areas were 15 mm × 15 mm for lap shear tests and 10 mm × 5 mm for in situ lap shear tests. The thicknesses of the applied adhesives were ~20 μm. After curing for one day, single-lap joint specimens were prepared.

Fourier transform infrared spectra measurements

Fourier transform infrared (FT-IR) spectra for ILs were obtained using an FT-IR spectrophotometer (Spectrum GX FT-IR System I-KS, Perkin Elmer). The FT-IR spectra of IL-SGAs with various IL contents were measured on a microscopic FT-IR spectrophotometer (AutoImage System, Perkin Elmer) in reflection mode.

In situ lap shear tests

In situ lap shear tests were performed in the microwave or in a heating oven (up to 200 °C). The samples were loaded with 4.9 N tensile stress until debonding occurred.

Lap shear tests

The lap shear strength was measured using an Autograph AG-X plus (Shimadzu Co) with a 1 kN load cell using a cross-head speed of 50 mm/min. The lap shear strengths of the single-lap joints before and after microwave irradiation at 2.45 GHz and 500 W for 2.5 min were also measured. These experiments were performed for at least five different specimens prepared under the same conditions.

Measurement of in situ temperature of IL-SGA adhesives under microwave irradiation

The time dependence of the temperature of IL-SGAs in a microwave oven was obtained using a fiber optic thermometer (FL-2000, Anritsu Meter Co., Ltd.). The temperature sensor of a thermometer was placed between samples (25 mm × 5 mm) with cured IL-SGA films.

Adhesive failure occurred in one, three, three, six, and six of the seven samples with 2.5, 5.0, 7.5, 10.0, and 12.5 wt% IL content, respectively. In the case of adhesives with more than 12.5 wt% IL contents, failure was observed in all samples. For IL content above 12.5 wt%, all samples failed under microwave irradiation within shorter times.

Dynamic mechanical analysis measurements

Dynamic mechanical analysis (DMA) was performed using a dynamic mechanical analyzer (DVA-220S, ITK Co., Ltd).

The heating rate and frequency were 6 °C/min and 10 Hz, respectively. Specimens with dimensions of 30 mm × 5 mm were prepared by curing and trimming the adhesives. The thickness was ~600 μm.

Results and discussion

Before preparing the SGA adhesives, including those with ILs, the heating of ILs alone with microwave irradiation was evaluated to choose proper ILs for investigating effective responsivity to microwave irradiation. Various ILs in glass capillaries were irradiated with microwaves in the microwave apparatus. The chemical structures of the ILs are shown in Fig. 1. Table 1 shows the heating response of the IL and IL-SGA adhesives to microwaves. Some ILs, namely, BMPY-TFSI, ABIM-TFSI, EMIM-SCN, BMIM-SCN, BMIM-PF_6, BMIM-BF_4, and BMIM-I, were heated under microwave irradiation to a temperature so hot that we could not touch them, while others showed no response or only a slight response to microwaves. The heated ILs were added to SGA, and the IL-SGA adhesives were prepared. The SGA adhesives including only BMIM-SCN and BMIM-I were rapidly heated under microwave irradiation. However, the IL-SGA adhesives including BMIM-I could not be cured and were thus inadequate as an adhesive. Therefore, we employed BMIM-SCN as an IL and prepared IL-SGA adhesives to investigate “on-demand peeling” properties as triggered by microwave irradiation. The weight ratios of BMIM-SCN to the SGA adhesive ranged from 0 to 20 wt%.

After applying the IL-SGA adhesives on PI substrates and adhering the PI substrates, the adhesives were cured for 1 day at room temperature. Figure. 2 shows the FT-IR spectra of the cured IL-SGA adhesives. Bands originating from the thiocyanate and alkyl groups of ILs were observed at 2053 cm^{-1} and 2900 cm^{-1}, respectively [41]. SGA adhesives showed IR bands of the carboxyl groups of the ester at 1723 cm^{-1} and the alkyl groups at ~2900 cm^{-1}. In the FT-IR spectra of the IL-SGA adhesives, bands originating from both the SGA adhesives and ILs were observed. In addition, the absorption of the band originating from the thiocyanate group increased with the amount of IL in the IL-SGA adhesives. Therefore, in the curing processing of the adhesives, the chemical structure of the IL was retained without reaction with the precursors of the SGA adhesives.

To investigate the lap shear strength of the IL-SGA adhesives under microwave irradiation, the samples adhered with the IL-SGA adhesives were loaded with weights of 4.9 N in the microwave oven, as shown in Fig. 3a. The power and frequency of the microwaves were 500 W and 2.45 GHz, respectively. The time required for the weight to slip down was used as the holding time to evaluate the microwave irradiation effects. Figure. 3b shows the effect of the IL contents in the adhesives on the holding time. In the case of the SGA adhesives alone, no adhesion failure occurred after more than 5 min of microwave irradiation. The addition of ILs led to decreased holding time during in situ lap shear tests under microwave irradiation within 5 min. With IL content below 12.5 wt%, several adhered specimens withstood microwave irradiation for 5 min. In the case of the IL-SGA adhesive including 20 wt% IL, the holding time was <30 s. The adhesive failures for IL-SGA adhesives occurred at the interfaces between the adhesives and the PI substrates. These results suggested that the SGA adhesives were modified into microwave-responsive “on-demand-peeling” adhesives via loading with ILs.

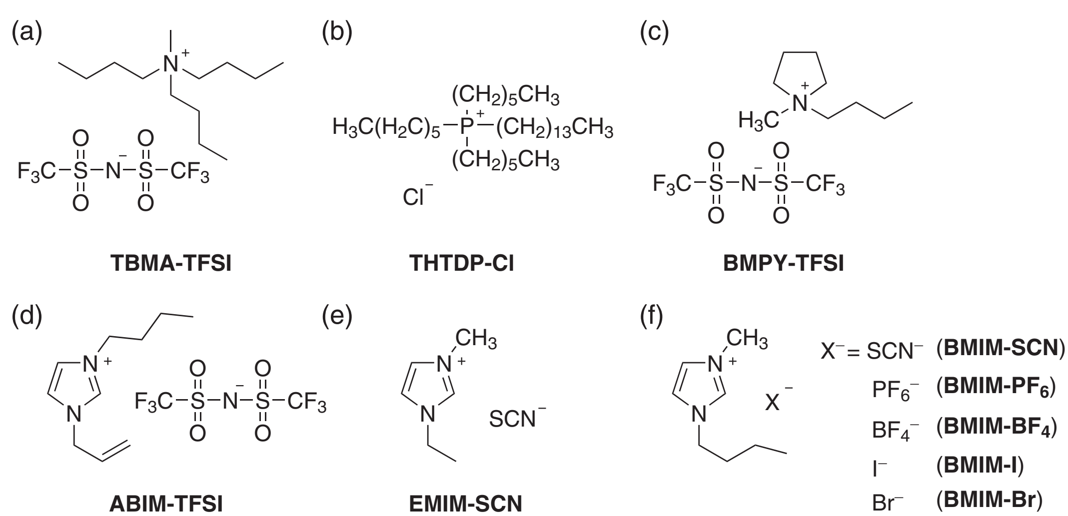

Fig. 1 Chemical structures of microwave-irradiated ILs

Table 1 Heating response of ILs and IL-SGA adhesives to microwave irradiation[a]

	ILs alone	ILs+SGA adhesive
TBMA-TFSI	None	–
THTDP-Cl	None	–
BMPY-TFSI	Heated[b]	None
ABIM-TFSI	Heated[b]	None
EMIM-SCN	Heated[b]	Slightly heated[c]
BMIM-SCN	Heated[b]	Heated[b]
BMIM-PF_6	Heated[b]	None
BMIM-BF_4	Heated[b]	Slightly heated[c]
BMIM-I	Heated[b]	Heated[b]
BMIM-Br	None	–

[a]After 30 s of microwave irradiation

[b]ILs or IL-SGAs were heated hotter than could be touched

[c]ILs or IL-SGAs were warm to the touch

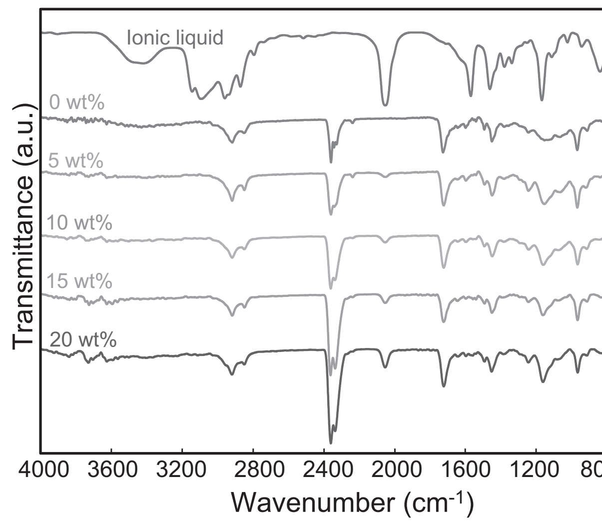

Fig. 2 FT-IR spectra of an IL, SGA adhesive, and IL-SGA adhesives containing 5, 10, 15, and 20 wt% of the IL

To investigate the microwave-assisted heating effects in the IL-SGA adhesives, we evaluated the temperature of the IL-SGA adhesives in situ under microwave irradiation with an optical fiber thermometer. The microwave-irradiation effect on the temperature of the IL-SGA adhesives is shown in Fig. 4. In the case of adhered specimens without ILs as well as even the fiber thermometer alone without any samples, slight increases of temperature were observed. In contrast, higher IL contents led to more rapid heating of the IL-SGA adhesives by microwave irradiation. The temperature of the IL-SGA adhesives with 20 wt% IL under microwave irradiation increased more than 250 °C. The oscillations in temperature caused by microwave-assisted heating were attributed to the heterogeneous microwave irradiation in the conventional microwave apparatus.

The lap shear strength of the PI/IL-SGA adhesive/PI specimens with various IL contents were evaluated at room temperature before and after microwave irradiation. The lap shear strengths are shown in Fig. 5. The lap shear strengths of all IL-SGA adhesives were maintained even after microwave irradiation compared with the strength before irradiation. This suggests that microwave irradiation would have no effect on the chemical structure and morphology of the SGA adhesives and serves as a non-destructive stimulus for the SGA adhesives. Moreover, the lap shear strengths were almost completely retained, regardless of the IL contents in the IL-SGA adhesives. The shear strength values were ~0.9 MPa. Typically, additives with a low mechanical modulus, such as ILs, behave as plasticizers in polymer resins. However, the adhesive properties of IL-SGA adhesives were constant, even with the addition of ILs. Therefore, in these microwave-responsive "on-demand-peeling" adhesives, adhesion failure was achieved only through microwave-assisted heating of ILs, without a decrease in the adhesive properties or decomposition of the SGA adhesives.

To investigate the effect of temperature on the adhesive strength and other mechanical properties, we performed lap shear tests at various temperatures as well as DMA measurements. In the lap shear tests at various temperatures, we evaluated the holding temperature at which the weight slipped down, in a similar system to that used in the in situ lap shear tests under microwave irradiation. Figure S1 in Supporting information shows the relationship between the holding temperature in the heating oven (without microwave irradiation) and the IL content in the SGA. In the case of only the SGA adhesives, even at temperatures of greater than 200 °C, adhesion between the PI substrates and SGA adhesives persisted. The addition of ILs led to a decrease in the holding temperature. The decrease in irradiation time required for the weight to slip down was also attributed to the lower holding temperature. However, the holding temperature of the IL-SGA adhesives including 20 wt% IL was above 60 °C, and the adhesion was maintained solidly at room temperature.

Figure S2 in Supporting information shows the temperature dependence of the storage modulus and mechanical tan δ of SGA with ILs. The storage modulus and glass transition temperature (T_g) of the IL-SGA adhesives decreased with the addition of ILs. At 25 °C, the storage modulus of the IL-SGA adhesives decreased from 1.12×10^3 to 11.9 MPa with the addition of ILs. The T_g of the IL-SGA adhesives also changed from 66 to 12 °C, as shown in Table S1 in Supporting information. This suggested that the ILs in the IL-SGA adhesives behave as plasticizers. The decrease in the holding temperature in the heating oven was attributed to this plasticization effect via the addition of IL to the SGA adhesives. In contrast, the amount of IL in SGA

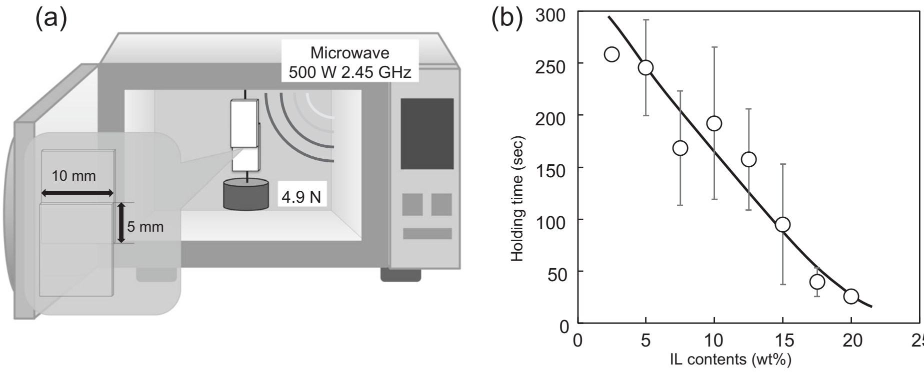

Fig. 3 **a** In situ lap shear test system in a microwave oven. **b** Effect of IL content on the holding time in the microwave oven

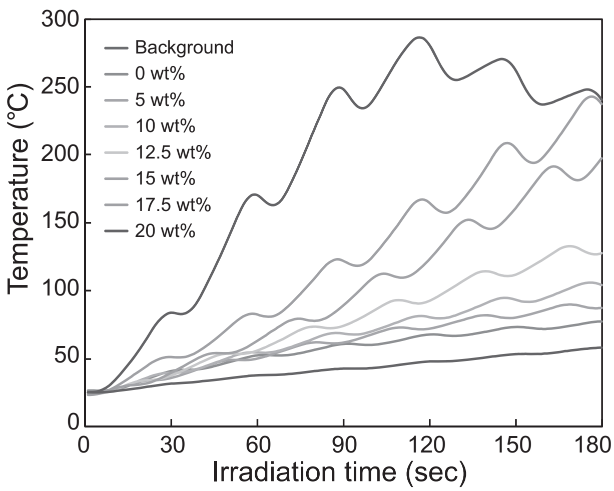

Fig. 4 Irradiation time dependence of the temperature of SGA adhesives with varying IL content in a microwave oven

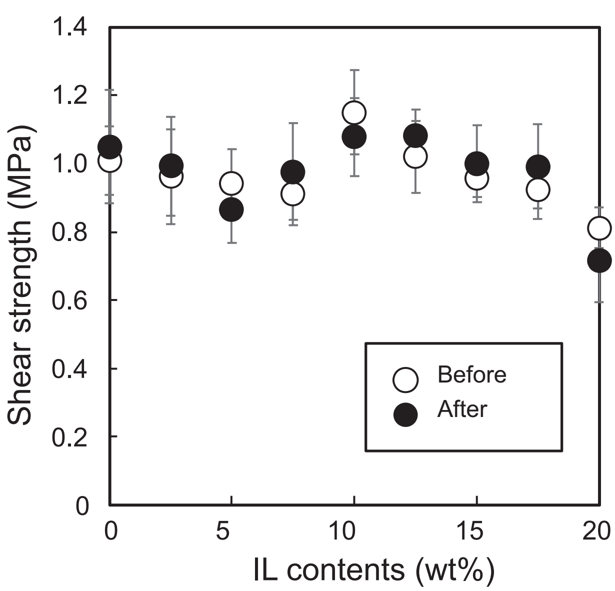

Fig. 5 Lap shear strength of SGA adhesives with varying IL contents at room temperature before and after microwave irradiation

had almost no effect on the adhesion strength at room temperature because the fracture mode of the adhered samples was interfacial peeling. Therefore, the IL-SGA adhesives, including 20 wt% ILs responded rapidly to microwave irradiation, and the adhesive strength under microwave irradiation decreased drastically.

Conclusions

We established an experimental system and evaluated the "on-demand easy peeling" of acrylic adhesives including ILs by microwave irradiation. The combination of anions and cations had large effects on the microwave response of the adhesives with ILs. As little as 30 s of microwave irradiation led to a rapid response and adhesive failure. The adhesive strengths of the adhesives with ionic liquid content was unchanged without microwave irradiation. Moreover, the local elevation of temperature under microwave irradiation was observed directly. The temperature of the adhesive increased to 250 °C under microwave irradiation. The unusual microwave stimulus prevents accidental, undesired dismantling of the stimuli-responsive adhesives. In addition, we suggest that the method of preparation of these "on-demand easy peeling" adhesives with ILs is simple and convenient.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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