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# Adhesion performance and recovery of acrylic pressure-sensitive adhesives thermally crosslinked with styrene–isoprene–styrene elastomer blends for flexible display applications



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# ABSTRACT

Fixing layers in flexible displays requires acrylic pressure-sensitive adhesives (PSAs) that can withstand deformation and folding stress. In this study, the effects of a styrene–isoprene–styrene (SIS) elastomer on the adhesion performance and recovery of an acrylic PSA were investigated. In simple blends, the peel strength and probe tack of a PSA sheet decreased slightly as the SIS content increased owing to the elastomer's soft nature. In contrast, the lap shear stress increased slightly owing to entanglement of the SIS polymer chains. In stress relaxation tests, the effect of SIS on PSA recovery was limited by weak bonding. Therefore, the use of thermal crosslinking to improve bonding within PSA/SIS blends was examined. Although increased crosslinking decreased the peel strength and probe tack, the adhesion properties were maintained and the lap shear stress and strain increased. Further, PSA recovery improved in stress relaxation tests, but a specific strain could not be endured above 1 phr crosslinking agent. Thus, crosslinking with an elastomer plays an active role in the adhesion performance and recovery of acrylic PSAs. However, as the PSA sheet becomes opaque in the presence of SIS, the display applications of this system are limited and further improvements are necessary.

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# Introduction

Pressure-sensitive adhesives (PSAs) are important in various fields because when used to assembly different materials, they allow the substrates to be separated easily under weak pressure without leaving a residue [1,2]. There are several types of PSAs, including acrylic, rubber, and silicon PSAs. Among these, acrylic PSAs, which contain various reactive monomers that can be modified to impart functionality, are the most widely used in industrial applications, such as splicing tapes, graphic films, display products, and medical products [3]. In general, the physical properties of acrylic PSAs are determined by the reactive monomer, such as butyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, and isobornyl acrylate [4,5]. Because PSAs are used to fix individual layers in flexible displays, they must be able to

withstand the stress and the strain generated by bending or rolling. However, sufficient deformation and recovery have not yet been achieved using PSAs alone.

Styrene–isoprene–styrene (SIS) triblock copolymers are typical thermoplastic elastomers, which exhibit both elastic behavior and thermoplastic properties [6]. Thus, the physical properties of SIS are very suitable for use in PSAs, and SIS has been used in various industrial applications, such as pressure-sensitive tapes and labels, contact adhesives, hot-melt adhesives, construction adhesives, and mastics [7–12]. SIS is manufactured by first producing a styrene chain, then adding an isoprene unit to form a diblock copolymer, and finally connecting two diblock copolymers to produce a triblock copolymer [13]. In SIS, adjacent polystyrene (PS) blocks gather together in small areas to form hard thermoplastic PS groups that are dispersed throughout a network of rubbery polyisoprene (PI). This structure imparts SIS with elasticity and the recovery properties of isoprene [14–16].

Crosslinking is one of the most effective methods for adjusting the adhesion performance, including the cohesion and creep resistance, of PSAs,. Typically, crosslinking of PSAs involves a chemical reaction between a crosslinking agent and functional

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groups on the PSA chain, such as hydroxyl groups. However, too high a crosslinking density can cause gelation, which decreases the cohesive strength and corresponding tack properties of crosslinked PSAs [17,18].

In our previous study, the effect of crosslinking on the adhesion performance and recovery of an acrylic PSA was evaluated. As the content of the crosslinking agent increased, the adhesion performance decreased and the recovery improved. However, when the content of the crosslinking agent was too high, the acrylic PSA exhibited no adhesion and a specific deformation could not be tolerated. In other words, crosslinking was very important for realizing flexible display applications, but its effect was limited [19].

In this study, an acrylic PSA was synthesized and the adhesion performance and recovery of simple acrylic PSA/SIS blends were investigated. After optimizing the SIS content, acrylic PSA/SIS blends were crosslinked to improve the bonding between the molecular chains of the two substances (Fig. 1). The adhesion properties of PSA sheets were determined via peel, tack, and lap shear tests conducted using a texture analyzer. In addition, stress relaxation tests were performed via dynamic mechanical analysis (DMA) to evaluate the recovery of the PSA.

# Materials and methods

# Materials

Acrylic reactive monomers, including 2-ethylhexyl acrylate (2-EHA, 99.0% purity, Samchun Pure Chemical, Republic of Korea), 2-hydroxyethyl methacrylate (2-HEMA, 99.0% purity, Samchun Pure Chemical), butyl acrylate (BA, 99.0% purity, Samchun Pure Chemical), and acrylic acid (AA, 99.0%) were used without further purification. An SIS elastomer (TSRC (Nantong) Industrial Ltd., China) with a styrene content of 17.5–19.0 wt.% and a melt flow index of 58.2–82.8 g/min was used. Ethyl acetate (EAc, 99.0% purity, Samchun Pure Chemical) was used as a solvent. 2,2'-azobisisoby-tryronitrile (AIBN, Junsei Chemical, Japan) was used as a thermal initiator. AK-75 (Aekyung Chemical Corp. Ltd., Republic of Korea) with a NCO content between 12.5% and 13.5% was used as a crosslinking agent.

# Synthesis of acrylic PSA

The acrylic reactive monomers (2-EHA, 2-HEMA, BA, and AA) and AIBN with EAc were mixed together in a 500 mL four-neck flask equipped with a stirrer, condenser, and thermometer, and the mixture was then heated to 80 °C under continuous mechanical stirring. The formulation for the acrylic PSA pre-polymer is listed in Table 1. During synthesis, the mixture was purged with N<sub>2</sub> gas to

Table 1
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Formulation of acrylic PSA pre-polymer.

Formulation		Acrylic PSA pre-polymer
Reactive monomer (wt.%)	2-EHA	70
	2-HEMA	10
	BA	15
	AA	5
Thermal initiator (phr)	AIBN	0.5

remove  $O_2$ , which interrupts the synthesis process. The total content of AIBN was fixed at 5 parts per hundred resin (phr) and the synthesized PSA pre-polymer was a random copolymer.

# Noncrosslinked acrylic PSA/SIS blends

To determine the optimum SIS content, various mixtures of the synthesized PSA pre-polymer and SIS were prepared. Table 2 shows the compositions of the acrylic PSA/SIS blends. To investigate the influence of the SIS elastomer alone on the properties of PSA sheets, these samples were coated on polyethylene terephthalate (PET) films without thermal cross-linking and then dried at 80 °C for 10 min to evaporate the solvent.

#### Thermally crosslinked acrylic PSA/SIS blends

Crosslinked acrylic PSA/SIS blends were prepared by mixing 100 wt.% PSA/SIS with 0.2, 0.4, 0.6, 0.8, and 1.0 phr of the crosslinking agent. Based on the results for the noncrosslinked blends, the SIS content was fixed at 5 wt.% (Table 3). The mixed samples were coated on the surface of 75  $\mu$ m thick corona-treated PET films, dried at 80 °C for 10 min, and then thermally crosslinked at 120 °C for 2 min and at 60 °C for 12 h.

# Characterization

#### Gel permeation chromatography

The molecular weights and polydispersity indices (PDIs) of the synthesized PSAs were determined using an Agilent 110

## Table 2

Composition of acrylic PSA/SIS blends without crosslinking agent.

Samples	Acrylic PSA pre-polymer (wt.%)	SIS Elastomer (wt.%)
PSA-SIS-00	100	0
PSA-SIS-05	95	5
PSA-SIS-10	90	10
PSA-SIS-15	85	15
PSA-SIS-20	80	20



Fig. 1. Schematic design of the acrylic PSA/elastomer binary system

#### Table 3

Composition of acrylic PSA/SIS belnds with crosslinking agent.

Samples	Crosslinked acrylic PSAs		SIS elastomer (wt.%)
	Acrylic PSA pre-polymer (wt.%)	Crosslinking agent (phr)	
PSA-SIS-05	95	0	5
PSA-SIS-05-02		0.2	
PSA-SIS-05-04		0.4	
PSA-SIS-05-06		0.6	
PSA-SIS-05-08		0.8	
PSA-SIS-05-10		1.0	

instrument equipped with a pump and a refractive index detector. Tetrahydrofuran was used as the eluent at a flow rate of 1 mL/min.

## Peel strength

The peel strengths of specimens with widths of 25 mm were determined using a texture analyzer (TA-T2i, Micro Stable Systems, UK). The specimens were pressed onto stainless steel (SUS) substrates by two passes of a 2 kg rubber roller and then stored at room temperature for 24 h. The peel strength magnitude (defined as the average force affecting a PSA specimen during debonding) was determined in accordance with the ASTM D3330 standard at an angle of 180°, crosshead rate of 300 mm/min, and temperature of 20 °C. The affected forces were recorded for five different runs with an average value of N/25 mm.

# Probe tack

Probe tack testing was conducted at 20 °C in accordance with the ASTM D2979 standard using the texture analyzer equipped with a 5 mm SUS cylinder probe. The testing procedure consisted of the following three stages: approaching the PSA surface, touching the PSA surface, and detaching from the PSA surface. The probe speed rate was 0.5 mm/s, the separation rate was 10 mm/s, and the contact time with the PSA surface was 1 s at a constant force of 100 g/cm<sup>2</sup>. During debonding, the measured tack values corresponded to the maximum debonding forces. The debonding forces were recorded six times with an average value of N.

#### Lap shear

Lap shear testing was conducted using the texture analyzer. The tested specimens were cut into smaller pieces with widths of 25 mm. After removal from a silicone release film, each PSA film was attached to a PET substrate (the adhesion cross-sectional area was  $25 \times 25 \text{ mm}^2$  and a 2 kg rubber roller was passed over the film surface three times). Lap shear tests were performed at a crosshead rate of 5 mm/min. The shear strain rate values were calculated using the following equation:

## Shear strain rate (%) = $\Delta L/t \times 100$

where  $\Delta L$  is the moving distance and t is the thickness of the PSA film.

PSAs for flexible displays are usually subjected to different shear strains, depending on their structure and radius of curvature. Therefore, investigating the relationship between the shear strain rate and the thickness of the applied PSA film is imperative for their future use in flexible display applications [20].

# Stress relaxation

Stress relaxation testing was conducted using a DMA apparatus (Q-800, TA Instruments, USA). The goal of this experiment was to determine the PSA characteristics and their suitability for flexible display applications by measuring the correlation between the deformation and stress over time. During the initial testing period, the investigated PSA samples were stabilized for 1 min, and then 500% strain was applied for 10 min. Subsequently, the studied specimens were allowed to recover for 5 min. The degrees of elastic recovery and residual creep strain values were obtained for the PSA samples as a function of time at different applied strains. The initial stress, final stress, and relaxation ratio values were determined from the obtained stress/time graphs [19].

## **Results and discussion**

### Gel permeation chromatography

Table 4 shows the molecular weights and PDIs of the acrylic PSA pre-polymer and SIS. The number average molecular weights of the synthesized acrylic PSA pre-polymer and SIS were approximately 40,000 and 140,000 respectively. The PDIs of the materials were 4.9 and 1.1 respectively. These results confirmed the suitability of these materials for our research design, which involves combining the long and uniform molecular chains of the elastomer to improve the restoring force of PSA sheets with the short and random molecular chains of the PSA to maintain the adhesion performance.

# Noncrosslinked acrylic PSA/SIS blends

First, to confirm the optimum content of the elastomer, the elastomer content in noncrosslinked PSA/SIS blends was varied and the adhesion performance and flexibility were investigated. In subsequent experiments, the elastomer content in the crosslinked blends was fixed based on these results.

#### Peel strength and probe tack

Fig. 2 shows the peel strength and probe tack of the acrylic PSA/ SIS blends as a function of the SIS content. First, the peel strength of the noncrosslinked acrylic PSA/SIS blends decreased as the SIS content increased. This behavior is due to the influence of the long polymer chains of SIS on the wettability of the acrylic PSA, which has relatively short polymer chains [21]. However, as the content of SIS was not high, the peel strength did not decrease significantly. Second, the probe tack of the noncrosslinked acrylic PSA/SIS blends showed a similar decrease as the SIS content increased. In general, SIS is known to be more effective than other elastomers for improving cohesion, and the cohesion is an important factor for determining the probe tack of the PSA [22]. However, in this case, the much shorter polymer chains of the acrylic PSA had a negative effect. Nevertheless, the probe tack values did not decrease significantly. These results indicated that the long molecular chains of SIS decrease the adhesion performance of the acrylic PSA, but

#### Table 4

Characterization of the acrylic PSA pre-polymer and SIS elastomer.

Materials	M <sub>n</sub> (g/mol)	M <sub>w</sub> (g/mol)	PDI
Acrylic PSA pre-polymer	39,632	192,860	4.9
SIS elastomer	136,320	151,330	1.1



Fig. 2. Peel strength and probe tack of noncrosslinked acrylic PSA/SIS blends as a function of SIS content.

the decrease is relatively small owing to the elastomer's soft nature.

## Lap shear test

Fig. 3 shows the lap shear test results for the noncrosslinked acrylic PSA/SIS blends as a function of the SIS content. As shown in Fig. 3a, the stress and strain values slightly increased as the SIS content increased. This behavior is due to the increase in the content of SIS, which has long molecular chains, thus increasing the probability of entanglement. Previous studies have found that entanglement phenomena rapidly increase as the molecular weight increases [20]. As confirmed by gel permeation chromatography, SIS has a higher molecular weight than acrylic PSA, and the extent of entanglement increased as the content of SIS increased. Fig. 3b shows the maximum stress and the strain at the maximum stress from the lap shear tests of acrylic PSA/SIS blends as a function of SIS content. The lap shear value did not vary greatly as the content of SIS increased. This behavior occurred because there is only physical entanglement between the molecular chains without chemical bonding [23].

# Stress relaxation test

Stress relaxation tests were performed to determine the strain change as a function of the SIS content (Fig. 4). Pure acrylic PSA without addition of the SIS elastomer was unable to withstand a specific strain and did not recover after removal of the strain. In contrast, SIS showed a tendency to withstand a specific strain, and although the extent of recovery was small, the system did recover. Thus, the addition of SIS with long molecular chains improves the modulus values of the acrylic PSA/SIS blends and recovery is possible. According to our previous work, recovery of noncrosslinked acrylic PSA under a specific strain is observed at number average molecular weights greater than approximately 100.000 g/mol [20]. Considering that the number average molecular weight of the acrylic PSA used in this study was approximately 40,000 g/mol, the observed recovery at a specific strain can be attributed to the entanglement phenomenon of the SIS elastomer. However, the elastic recovery and residual creep strain values did not change significantly as the SIS content increased. Owing to the lack of chemical bonding in these films manufactured from simple acrylic PSA/SIS blends, the presence of SIS did not have a significant influence on recovery.

Fig. 5 shows the stress change, which is a measurement of the stress generated under a specific strain, as a function of the SIS content. Similar to the strain change results, pure acrylic PSA without added SIS was unable to bear a very small initial stress. However, as the SIS content increased, the stress began to increase, but the change was quite small. In addition, the final stress relaxed completely, regardless of SIS addition, with a value close to 0 N. As a result, the relaxation ratios of all the samples were close to 100%. These results confirmed that the elastomer influences the recovery of the PSA, but improvement of the recovery is limited because simple blending only results in physical entanglement between the molecular chains. Thus, we tried to improve the recovery by enhancing the interactions between the SIS and PSA chains through crosslinking the acrylic PSA.

# Thermally crosslinked acrylic PSA/SIS blends

To improve the recovery of the PSA, a crosslinking system was introduced. In our previous work, when the crosslinking density of PSA increased, the adhesion performance decreased because the PSA became solid-like [19]. Therefore, the elastomer was added at a fixed content of 5 wt.%, corresponding to the highest adhesive strength observed in the simple PSA/SIS blends. Then, the adhesion performance and flexibility of crosslinked PSA/SIS systems with different contents of crosslinking agent were investigated.

## Peel strength and probe tack

Fig. 6 depicts the adhesion results according to the content of crosslinking agent in the acrylic PSA/SIS blend (fixed SIS content of 5 wt.%). In general, the adhesion performance of the PSA decreased as



Fig. 3. Lap shear test results for noncrosslinked acrylic PSA/SIS blends as a function of SIS content: (a) stress-strain curve and (b) maximum stress and strain values obtained at the maximum lap shear stress.



Fig. 4. Stress relaxation test results for noncrosslinked acrylic PSA/SIS blends as a function of SIS content: (a) strain change and (b) elastic recovery and residual creep strain.



Fig. 5. Stress relaxation test results for noncrosslinked acrylic PSA/SIS blends as a function of SIS content: (a) stress change and (b) initial stress, final stress, and relaxation ratio.



Fig. 6. Peel strength and probe tack of acrylic PSA/SIS blends as a function of crosslinking agent content.

crosslinking proceeded. Interestingly, the performance exhibited a sharp decline at 0.2 phr crosslinking agent, but no significant changes in the adhesion performance were observed when the content of the crosslinking agent was increased further. Even when the content of the crosslinking agent increased from 0.2 to 1.0 phr, the peel strength and the probe tack remained greater than 10 N and 5 N, respectively. Typically, the adhesion performance of an acrylic PSA decreases as the content of the crosslinking agent increases [19,24]. However, as we will discuss in Section "Lap shear test", the elastomer did not interfere with the crosslinking of the acrylic PSA. As the acrylic film was solidified when the crosslinking agent content was 1.0 phr or more, further measurements were impossible. According to previous studies, the modulus of acrylic PSA increases as the content of the crosslinking agent increases, so that the wettability of the substrate and the mobility of the polymer chains are decreased by the presence of the crosslinking system [21].

### Lap shear test

Fig. 7 shows the lap shear test results for the acrylic PSA/SIS blend according to the content of the crosslinking agent. From the peel strength and probe tack tests, it was expected that the elastomer would interfere with the crosslinking of the acrylic PSA, but the shear stress and strain showed a clear dependence on the content of the crosslinking agent. With the addition of the crosslinking agent, the maximum stress increased to approximately 420 kPa from 220 kPa and the maximum strain increased to approximately 19,000% from 6300%. The maximum stress and strain began to decrease at a crosslinking agent content between 0.8 and 1.0 phr, likely owing to overcuring of the acrylic PSA film. In



Fig. 7. Lap shear test results for acrylic PSA/SIS blends as a function of crosslinking agent content: (a) stress-strain curve and (b) maximum stress and strain values obtained at the maximum lap shear stress.

our previous study, the shear stress of the acrylic PSA increased as the content of the crosslinking agent increased, but the strain of the acrylic PSA in the lap shear test started to decrease beyond the content of specific crosslinking agent [19]. However, addition of the elastomer suppressed this decrease in deformation, even when the content of the crosslinking agent was increased.

# Stress relaxation test

Fig. 8 shows the strain change observed in the stress relaxation test according to the content of the crosslinking agent. Compared with the results for the acrylic PSA/SIS blends without crosslinking, the PSA recovery of the crosslinked systems sharply increased. As the amount of crosslinking agent increased, the recovery increased, with a recovery of 72% observed at 1 phr crosslinking agent. Further, as the content of the crosslinking agent increased, the elastic recovery (recovery rate of approximately 1 s at elimination of strain) increased slightly, and the residual creep strain decreased considerably. When the content of the crosslinking agent was 1 phr or more, the acrylic PSA/SIS film became harder owing to excessive crosslinking and failed to withstand a specific deformation owing to a decrease in the plastic region.

Fig. 9 depicts the stress change observed in the stress relaxation test according to the amount of crosslinking agent. Unlike the noncrosslinked acrylic PSA/SIS blends, at a constant strain, the initial stress and the final stress increased continuously as the content of the crosslinking agent increased. However, the increase in the initial stress (from approximately 2 to 18 N) was significantly greater than the increase in the final stress (from approximately 0.03 to 3.8 N), so that the relaxation ratio was reduced to approximately 80%. Similar to the results for the change in strain, the crosslinked acrylic PSA/SIS blend with more than 1 phr crosslinking agent could not bear a specific strain. Fig. 10 provides a summary of the adhesion performance and recovery of acrylic PSA/SIS blends according to the content of the crosslinking agent. Addition of the SIS elastomer to the acrylic PSA allowed the adhesive strength to be maintained, with the peel strength and probe tack exhibiting constant values with crosslinking agent contents of 0.8 and 1.0 phr. In addition, the shear stress and the recovery at a specific strain were improved. These results confirm that addition of an elastomer to PSAs is effective for realizing flexible display applications.

## Conclusions

To determine its applicability in flexible displays, the adhesion performance and recovery of an acrylic PSA/SIS elastomer system were investigated. The long polymer chains of SIS inhibited the wettability of the simple acrylic PSA/SIS blends, decreasing the peel strength and probe tack, but owing to the relatively soft nature of the elastomer, the adhesion properties did not decrease sharply. However, entanglement between the high-molecular-weight chains of SIS increased the lap shear stress and strain slightly. Although the addition of SIS allowed a specific deformation to be withstood, the extent of recovery was limited because there were only weak physical bonds between the molecular chains of the acrylic PSA and



Fig. 8. Stress relaxation test results for acrylic PSA/SIS blends as a function of crosslinking agent content: (a) strain change and (b) elastic recovery and residual creep strain.



Fig. 9. Stress relaxation test results for acrylic PSA/SIS blends as a function of crosslinking agent content: (a) stress change and (b) initial stress, final stress, and relaxation ratio.



Fig. 10. Summary of the adhesion performance and recovery of acrylic PSA/SIS blends as a function of crosslinking agent content.

the SIS elastomer. To enhance the recovery, we improved the bonding between the molecular chains of the two materials by crosslinking the acrylic PSA in the presence of 5 wt.% SIS. In the crosslinked acrylic PSA/SIS system, the SIS elastomer was able to maintain the peel strength and the probe tack while increasing the shear stress and shear strain. Furthermore, crosslinking sharply increased the recovery rate. Thus, optimization of SIS elastomer addition and PSA crosslinking improved the adhesion properties and the recovery of the acrylic PSA/SIS system. However, owing to the characteristics of the SIS elastomer, the PSA sheet was opaque, which will limit the use of this system in flexible displays. To address this problem, our next study will examine the use of other types of elastomers.

# **Declarations of interest**

None.

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