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Molecular weight and crosslinking on the adhesion performance and flexibility of acrylic PSAs

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ABSTRACT
Acrylic pressure sensitive adhesives (PSAs) are used in the production of flexible displays. Acrylic PSAs must have high flexibility and recovery to meet the industry demands for flexible displays. For these reasons, acrylic PSAs were designed to determine the effects of molecular weight and cross-linking on acrylic PSAs. Stress relaxation and creep tests were measured with dynamic mechanical analysis to identify the flexibility and recovery of the acrylic PSAs. The molecular weight and glass transition temperature of the PSAs were measured by gel permeation chromatography and differential scanning calorimetry. A texture analyzer was used to measure the adhesion performance of the acrylic PSAs. With increasing molecular weight, the adhesion performance increased, especially from 86 to 108 K molecular weight PSAs. This is due to the entanglement of the polymer chains. The stress and recovery of cross-linked acrylic PSAs was much higher than that of neat acrylic PSAs. This result is attributed to interactions between the polymer chains due to cross-linking.

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KEYWORDS
Acrylic PSAs; molecular weight; cross-linking system; adhesion performance; flexibility

1. Introduction

Pressure sensitive adhesives (PSAs) are nonmetallic materials that are primarily used to bond the surfaces of various materials through adhesion and cohesion.[1] The function of PSAs is to ensure instantaneous adhesion with the application of light pressure.[2] In particular, acrylic PSAs have many advantages, such as excellent aging characteristics, resistance to elevated temperatures, and exceptional optical clarity. Therefore, acrylic PSAs can be applied in many areas, such as in splicing tapes, protective foils, films for the graphics market, and a range of medical products. The common composites used in acrylic PSAs are hard/soft segment monomers, additives and initiators. The role of the hard segment is to control the adhesive properties of PSAs. An example is acrylic acid (AA), which has a glass transition temperature (T_g) of 106 °C. AA possesses carboxyl groups that provide

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cross-linking sites. Therefore, the cohesion and adhesion properties can be controlled by the AA content. Another important component for controlling the tacky property of PSAs is the soft segment, which is a combination of soft and tacky polymers that have a low glass transition temperature (T_g). Commonly reported monomers include alkyl acrylates and methacrylates with 4 ~ 17 carbon atoms, e.g. butyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, isooctyl acrylate, or decyl acrylate. Other common monomers include additives, such as plasticizers, inhibitors, and fillers.[3] Therefore, the final properties of PSAs are controlled by combining optimal amounts of these components.[4]

A polymer's molecular weight influences thermal, mechanical, and rheological properties.[5–8] An increase in the chain length of higher molecular weight polymers increases the glass transition temperature (T_g) and viscosity and affects the wetting, adhesion, and cohesion properties of the adhesive.[5,6,9] Various polymers have been studied, for example, poly(vinyl alcohol),[10,11] epoxidized natural rubber,[12–14] and poly (isobutylene),[15] all of which have been studied with regard to the influence of molecular weight upon adhesion. In the case of poly (isobutylene), a high molecular weight provided the best shear strength at ~700,000 g/mol, whereas the tack strength decreased when the molecular weight increased.[15]

A cohesive failure occurs below the critical molecular weight of an adhesive, signifying that a higher strength and higher molecular weight polymer may enhance bonding. However, adhesive failure occurs above the critical molecular weight due to lower wettability, which prevents the polymer from interacting well with the surface.[13]

A high cohesion is needed to sustain loads and enable a clean removal. However, a sufficiently low viscosity is required to wet the surface of the substrate and create an intimate contact. These conflicting demands are balanced by adjusting T_g and the molecular weight distribution, as well as the degree of cross-linking and branching of the respective copolymers.[16–22] By increasing the cross-link density of the polymer, the overall performance of the final products can be improved.[23] Some additives are highly reactive and increase both the rate and final degree of curing, such as acrylate, methacrylate, or maleimide functional additives. Other additives, such as allyl-containing cyanurates and isocyanurates, only increase the overall degree of curing and not the rate.[24,25]

The objective of this research was to determine the relationship between the adhesion performance, flexibility and recovery properties of acrylic PSAs and molecular weight and cross linking. This study will allow for the confirmation of the validity of methods for measuring creep and stress relaxation with flexible display PSAs. Acrylic PSAs were synthesized from reactive monomers. The properties of the flexible display PSAs were investigated performing peel, tack and lap shear tests; samples were studied with different molecular weights and with/without cross-linking agents. Creep and stress relaxation were measured with dynamic mechanical analysis (DMA) to assess the flexibility of flexible display PSAs.

2. Experimental

2.1. Materials

Acrylic monomers, including 2-ethylhexyl acrylate (2-EHA, 99.0% purity, Samchun Pure Chemical, Republic of Korea), butyl acrylate (BA, 99.0% purity, Samchun Pure Chemical), ethyl acrylate (EA, 99.0% purity, Samchun Pure Chemical), 2-hydroxyethyl methacrylate (2-HEMA, 99.0% purity, Samchun Pure Chemical), and acrylic acid (AA, 99.0% purity, Samchun Pure Chemical) were purchased and used without further purification. Ethyl acetate (EAc, 99.0% purity, Samchun Pure Chemical) was used as a solvent. 2,2′-azobisisobytyronitrile (AIBN, Junsei Chemical, Japan) was used as a thermal initiator. AK-75 (Aekyung Chemical Corp. Ltd., Republic of Korea) was used as a cross-linking agent.

2.2. Synthesis of acrylic PSAs

The acrylic monomers (2-EHA, BA, EA, 2-HEMA and AA) and AIBN solution with EAc were mixed together in a 500-ml four-neck flask equipped with a stirrer, condenser, and thermometer, and the resultant mixture was heated to 80 °C with constant mechanical stirring. The compositions of acrylic PSAs are shown in Table 1. During polymerization, the system was purged with N_2 gas to prevent the introduction of oxygen which retards the polymerization process. As the viscosity of the polymer increased, a certain amount of EAc was added with the rest of the initiator. The total amount of AIBN was added from 0.2 to 0.6 phr to control the molecular weight of the PSAs. Figure 1 shows an ideal schematic diagram of the PSAs, and the synthesized PSAs are random copolymers.

2.3. Thermal cross-linking behavior of acrylic PSAs

2.3.1. Preparation of cross-linked acrylic PSAs

Cross-linked PSA was prepared by blending 100 wt% PSAs and 0.5 phr of AK-75 (NCO content: 12.5~13.5%). The synthesized PSAs and cross-linked PSAs were coated on 75-μm-thick corona-treated polyethylene terephthalate films, then dried at 80 °C for 10 min, and cross-linked at 120 °C for 2 min and at 60 °C for 12 h.

Table 1. Compositions of the acrylic PSAs.

Composition (wt%)		PSAs sample		
		SNU-A02	SNU-A04	SNU-A06
Reactive monomer	2-EHA	66.75	66.75	66.75
	BA	25	25	25
	EA	5	5	5
	2-HEMA	0.25	0.25	0.25
	AA	3	3	3
Thermal initiator	AIBN	0.2	0.4	0.6

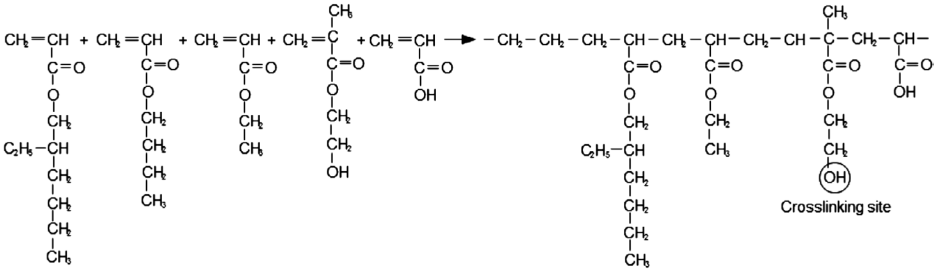


Figure 1. Ideal schematic diagram of the acrylic PSAs and cross-linking site.

2.3.2. Gel content

The gel content depends on the solubility parameter of the solvent; however, the trend of the results is very similar. In this work, toluene was selected as the solvent. The gel fractions of acrylic PSAs blended with AK-75 were determined by soaking 5-g samples in toluene for 1 day at room temperature with shaking. The soluble part was removed by filtration and dried at 80 °C to a constant weight. The gel fraction was calculated by the following equation:

$$\text{Gel fraction}(\%) = (W_1/W_0) \times 100$$

where W_0 and W_1 are the weights before and after filtration, respectively.

2.4. Characterization of the acrylic PSAs

2.4.1. Gel permeation chromatography

The molecular weight and polydispersity were measured using an Agillant 110 equipped with a pump and RI detector. Tetrahydrofuran was used as the eluent, and the flow rate was 1 mL/min. Table 2 lists the molecular weight and polydispersity of synthesized acrylic PSAs.

2.4.2. Differential scanning calorimetry

The glass transition temperatures of the acrylic PSAs were determined by differential scanning calorimetry (DSC) (Q-200 TA Instrument, USA). The samples were scanned form room temperature to 150 °C at a heating rate of 5 °C/min and then to −80 °C. After this process, the samples were scanned to 0 °C at a heating rate of 5 °C /min. In this analysis, the glass transition temperature of the acrylic PSAs was detected from −80 to 0 °C after the samples were scanned at high temperature, excluding the thermal history effects. Table 2 lists the glass transition temperatures of synthesized acrylic PSAs.

2.5. Adhesion performance

2.5.1. Peel strength

The peel strength was measured using a texture analyzer (TA-XT2i, Micro Stable Systems, UK). The specimens were prepared as 25-mm-wide samples. The specimens were pressed onto a stainless steel substrate with two passes of a 2 kg rubber roller and were stored at room temperature for 24 h. The peel strength was determined at an angle of 180° with a crosshead speed of 300 mm/min at 20 °C based on ASTM D3330. The peel strength is the average force on the debonding process. The force was recorded in N units for six different runs, and the average force was reported as N/25 mm.

2.5.2. Probe tack

The probe tack was measured using a texture analyzer (TA-XT2i, Micro Stable Systems, UK) with a 5-mm-diameter stainless steel cylinder probe at 20 °C. The standard probe tack

Table 2. Characterization of the acrylic PSAs.

PSAs sample	M_n (g/mol)*	M_w (g/mol)*	PDI*	T_g (°C)**
SNU-A02	60,503	362,700	6.0	−48
SNU-A04	86,205	524,140	6.1	−49
SNU-A06	107,890	686,870	6.4	−49

*Molecular weight was measured by gel permeation chromatography (GPC).

**T_g was measured by DSC.

test was divided into three stages: approaching the surface of the PSAs; contact with the surface; and detachment from the surface of the PSAs. The speed of the probe was 0.5 mm/s, and the contact time on the PSAs surface was 1 s under a constant force of 100 g/cm^2. The separation rate was 10 mm/s. In the debonding process, the probe tack was obtained as the maximum debonding force.

2.5.3. Lap shear test

The lap shear test was performed using a texture analyzer (TA-XT2i, Micro Stable Systems, UK). The specimens for the lap shear test were cut to a width of 25 mm. After being removed from a silicone release film, each PSA film was attached to another PET film without PSAs. The adhesion cross section area was 25 × 25 mm^2 and a 2 kg rubber roller was passed over it three times. The lap shear test was determined with a crosshead speed of 5 mm/min. Figure 2 is a scheme illustrating the lap shear test and the shear strain was calculated by the following equation:

$$\text{Shear strain rate}(\%) = \Delta L/t \times 100$$

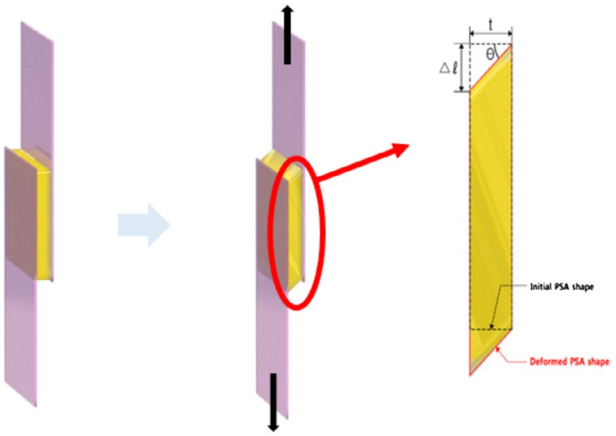
where ΔL is the moving distance and t is thickness of PSA.

PSA-applied flexible displays have diverse shear strain depending on the structure and the curvature radius of the display. Therefore, the rate of shear strain rate is dependent on the thickness of the PSAs, so the thickness data are needed for flexible displays.

2.6. Flexibility of the acrylic PSAs

2.6.1. Stress relaxation test

The stress relaxation test was measured using DMA (Q-800 TA Instruments, USA). For stress relaxation, it is important to determine the correlation between the deformation and



$$\text{Shear strain rate } (\varepsilon) = \tan\theta = \frac{\text{Displacement } (\Delta l)}{\text{Thickness of PSAs } (t)} \times 100$$

Δ l : Displacement (mm)

t : Thickness of PSAs (mm)

Figure 2. Scheme of the lap shear test and shear strain rate.

stress with time for a flexible display. At the initial period of the test, samples were stabilized for 1 min at 0.001 N, and then, the 400% strain was applied over 10 min. The measured specimen was maintained for 5 min at 0.001 N for recovery. Elastic recovery and residual creep strain were obtained from strain of the PSAs as a function of time. The initial stress, final stress, and relaxation ratios were derived from the stress/time graph. Figure 3 shows the detect points for flexible displays in strain and stress from the stress relaxation test.

2.6.2. Creep test

The creep test was measured using DMA (Q-800 TA Instruments, USA). At the initial period of the test, samples were stabilized for 1 min at 0.001 N, and then, the 1 N stress was applied over 10 min. The measured specimen was maintained for 5 min at 0.001 N for recovery. The elastic recovery and residual creep strain were obtained from the strain of the PSAs as a function of time. Figure 4 shows the detect points for flexible display in a strain graph from the creep test.

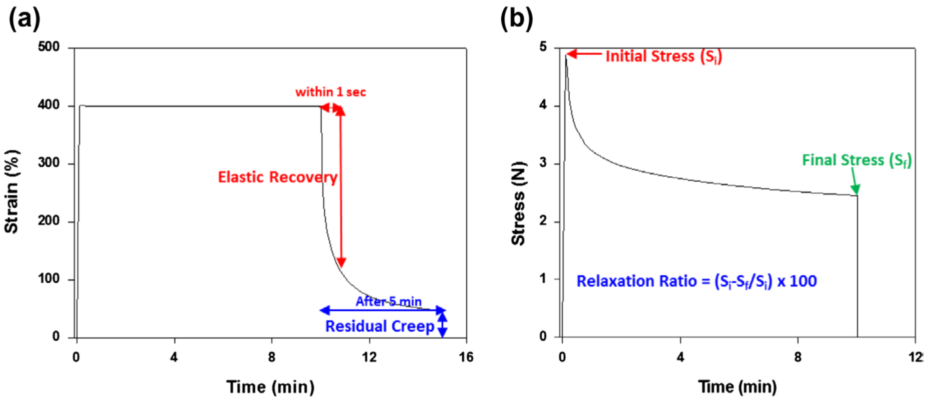


Figure 3. Detect points in the (a) strain and (b) stress change from the stress relaxation test.

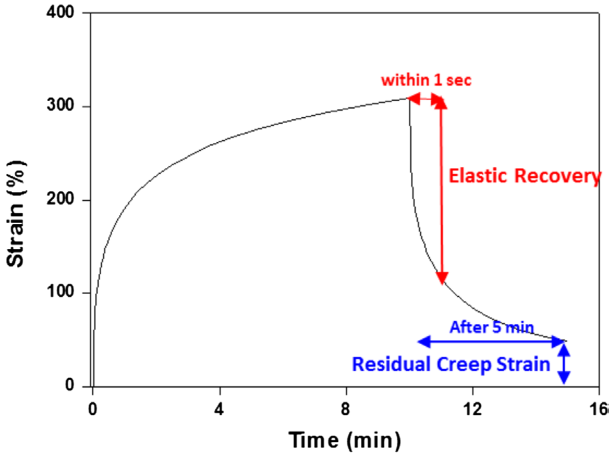


Figure 4. Detect points in the strain graph from the creep test.

3. Results and discussion

3.1. Gel content

The cross-linking density can be measured indirectly by calculating the gel content of each sample by measuring the insoluble fraction in the polymer.[26] The amount of gel phase measured is defined as the amount of cross-linked molecules from both the entanglement of molecular chains and coiled polymer molecules. In the latter case, cross-linking occurs only through entanglement. Moreover, it is not truly a polymeric network and usually consists of interconnected polymeric molecules, wherein the gel phase can vary due to the changes in the polymer molecular weight.[27–29] Figure 5 shows the gel fraction of various molecular weight acrylic PSAs (Neat/Cross-linked). The gel fraction of neat acrylic PSAs was approximately 0%, and the gel fractions of the PSA cross-linked with AK-75 (0.5 phr) were approximately 66%. Acrylic PSAs were well cross-linked by the addition of a cross-linking agent. In the case of cross-linked PSAs, the gel fraction slightly decreased with increasing molecular weight. Due to this, the low molecular weight of PSAs was subjected to fine cross-linking.

3.2. Adhesion performance

Figure 6 shows the dependence of the peel strength of the PSAs on molecular weight and the existence of a cross-linking agent. First, the peel strength of the neat PSAs decreased with increasing molecular weight. This observation is due to the decreasing wettability of the adhesive; a high molecular weight lowers the wettability of an adhesive, probably due to the influence of entanglement, as the entangled polymer chain does not flow effectively.[30] However, the peel strength of cross-linked PSAs showed no change with increasing molecular weight, which is attributed to the decreasing mobility of a cross-linked polymer chain. Figure 7 shows the probe tack results as a function of different molecular weights for neat and cross-linked PSAs. There was no effect on neat PSAs from increasing the molecular weight. However, the tack of cross-linked PSAs slightly increased with increasing molecular weight due to the interaction of the polymer chains from cross-linking and entanglement.

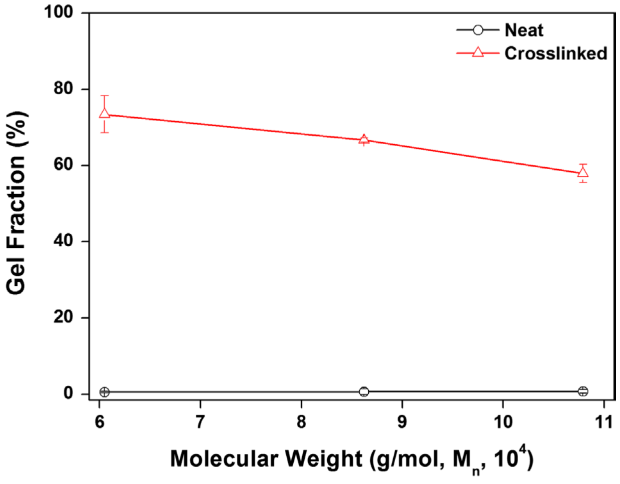


Figure 5. Gel contents of various molecular weight acrylic PSAs (Neat /Cross-linked).

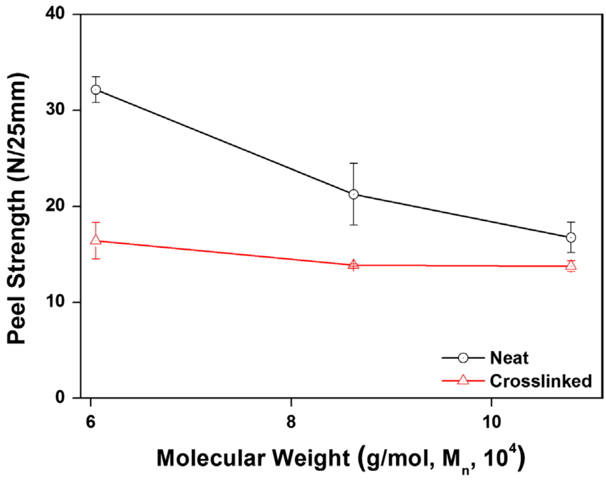


Figure 6. Peel strength of various molecular weight acrylic PSAs (Neat /Cross-linked).

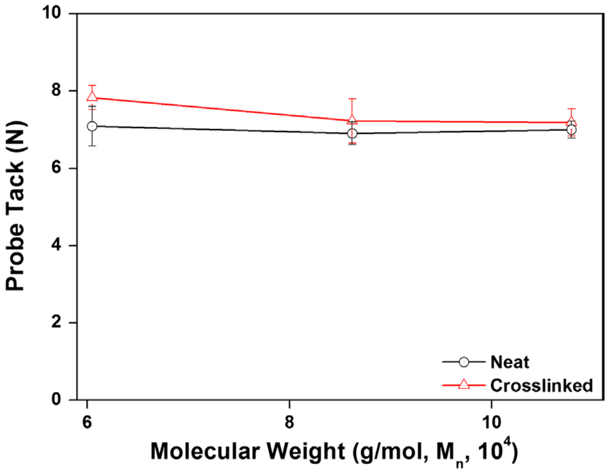


Figure 7. Probe tack of various molecular weight acrylic PSAs (Neat /Cross-linked).

Figure 8 shows the results of the lap shear test as a function of various molecular weights for neat and cross-linked PSAs. These results confirm the maximum stress and the strain at maximum stress. Before cross-linking, the maximum stress and the strain at maximum stress were increased with increasing molecular weight; this was a natural consequence of the increasing length of the chain. In contrast, the strain at maximum stress for the cross-linked PSAs decreased with increasing molecular weight. This is because the angle of the chain that is in a different state confirms the zoom effect on the shear strain of the PSAs, as shown in Figure 9.

3.3. Stress relaxation test

Figure 10 shows the results from a stress relaxation test of neat and cross-linked acrylic PSAs as a function of molecular weight. First, the stress of cross-linked acrylic PSAs increased

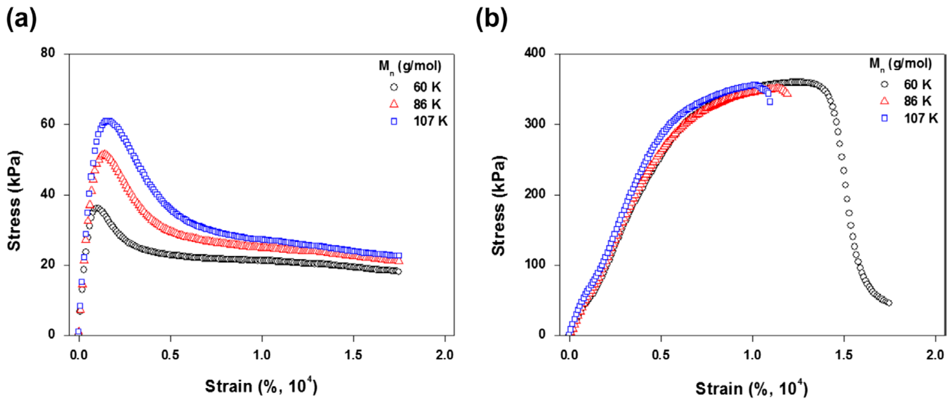


Figure 8. Lap shear of various molecular weight acrylic PSAs: (a) Neat, (b) Cross-linked.

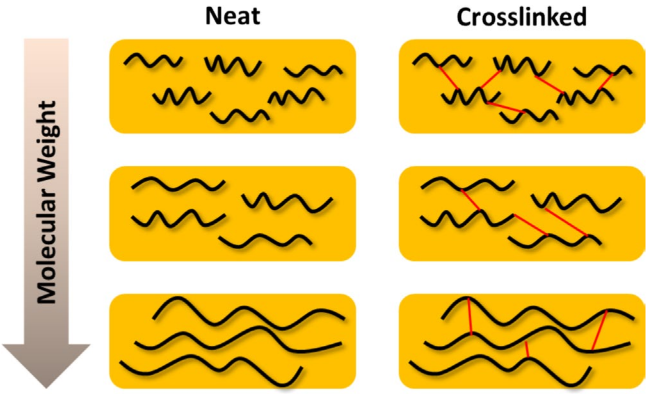


Figure 9. Scheme of various molecular weight acrylic PSAs (Neat/Cross-linked).

considerably with molecular weight. In the case of neat PSAs, the initial stress value was low and the stress was released over a short period of time. On the other hand, regardless of whether there was cross-linking, stress increased with increasing molecular weight. If not cross-linked, PSAs with a molecular weight of approximately 60 and 86 K released stress perfectly. However, the stress of PSAs that had a molecular weight above 108 K did not release stress perfectly due to the entanglement of the polymer chins. This entanglement increased dramatically when molecular weight increased. In the case of cross-linked PSAs, stress relaxation decreased as molecular weight increased, which is due to the entanglement inhibiting the mobility of polymer chains.

Figure 11 illustrates the recovery profile from the stress relaxation test for neat and cross-linked acrylic PSAs as a function of molecular weight. With neat PSAs, recovery only occurs with PSAs that have molecular weights greater than 108 K. The observation is caused from the entanglement of polymer chains because polymer chain entanglement decreases adhesion and increases cohesive properties.[31] In particular, a PSA that had a 60-K molecular weight did not recover even though a structural change was generated within the material; the strain rate was increased after the release of the stress. It can be inferred from these results that there is a critical entanglement point from 86 to 108 K molecular weights.

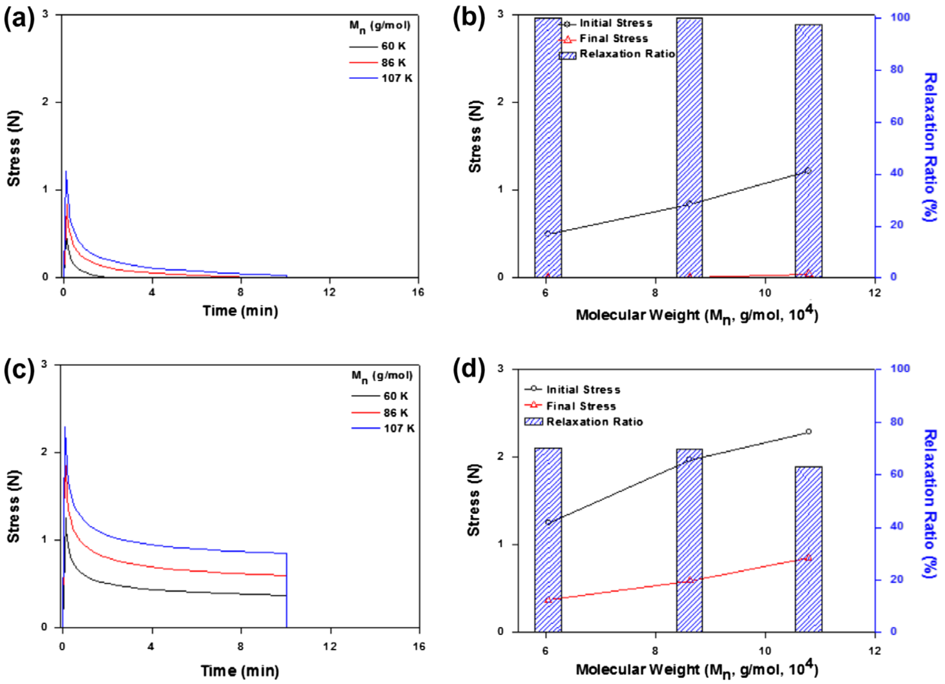


Figure 10. Stress deformation from the stress relaxation test of various molecular weight acrylic PSAs: (a), (b) Neat, (c), (d) Cross-linked.

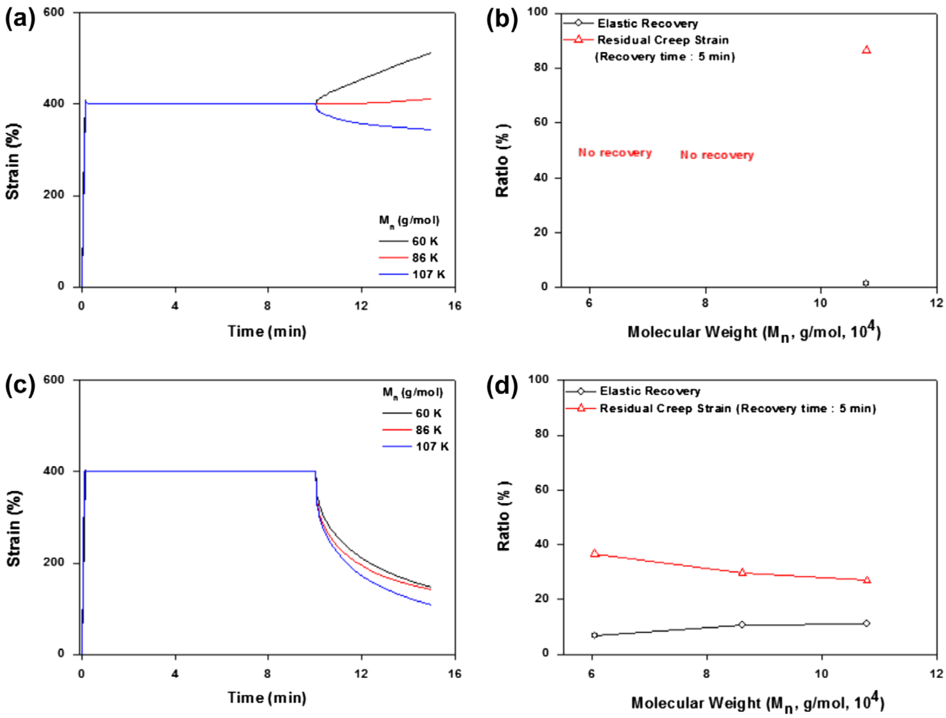


Figure 11. Strain deformation from the stress relaxation test of various molecular weight acrylic PSAs: (a), (b) Neat, (c), (d) Cross-linked.

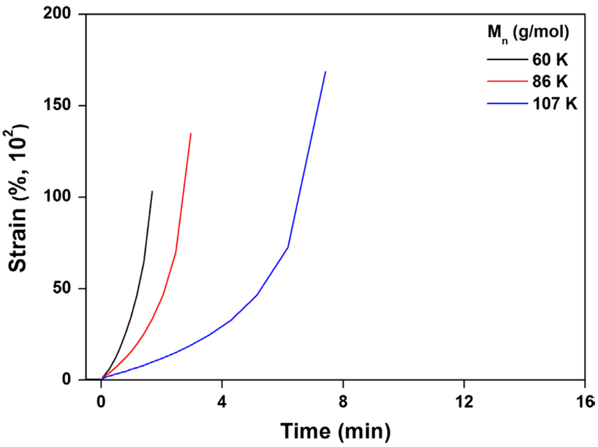


Figure 12. Creep of neat acrylic PSAs with various molecular weights.

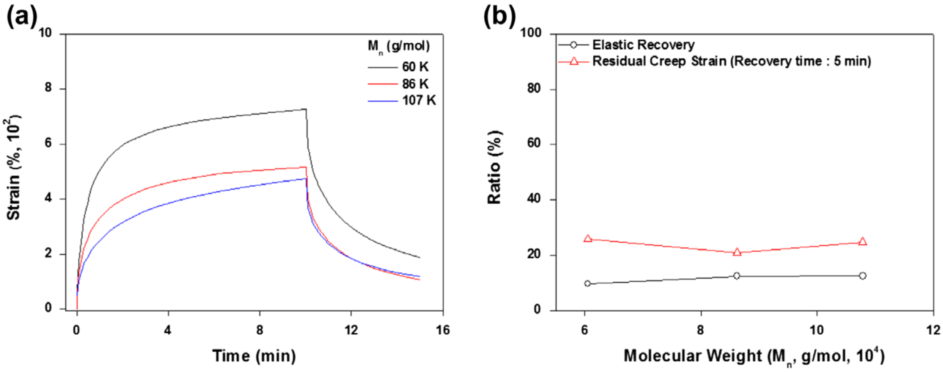


Figure 13. Creep of cross-linked acrylic PSAs with various molecular weights.

3.4. Creep test

Figure 12 illustrates the strain from the creep test of neat acrylic PSAs as a function of molecular weight. In one instance using a neat PSA, strain was dramatically increased and then was destroyed. However, as the molecular weight increased, the time that it took the sample to enter solution lengthened. Figure 13 illustrates the creep test results with cross-linked acrylic PSAs. Strain increased with increasing molecular weight for the same reasons stated above for the stress relaxation test. The recovery of cross-linked acrylic PSAs exhibited no change.

4. Conclusion

Acrylic PSAs of various molecular weights were synthesized and blended with cross-linking agents to prepare cross-linked acrylic PSAs. The PSAs were designed to examine the effects of molecular weight and cross-linking on the flexible display. Adhesion performance, stress relaxation, and creep test were performed to determine the effects of molecular weight and cross-linking on the flexible display. In this research, we investigated the viscoelasticity in the shear sandwich mode by DMA; however, there was no significant change as a function of molecular weight, except in a high temperature range. This was not a suitable method to detect the viscoelasticity of PSAs. Thus, we decided to determine whether DMA is a suitable method for stress relaxation and creep tests, as well as to measure the flexibility and recovery of PSAs. The investigation of the viscoelasticity of PSAs using other modes will be performed in further studies.

From the gel fraction results, it was confirmed that the acrylic PSAs were well cross-linked. The peel strength of the neat acrylic PSAs decreased with increasing molecular weight; the peel strength of the cross-linked acrylic PSAs did not change due to impaired wettability. The probe tack of the cross-linked acrylic PSAs was slightly increased, which suggests that the interaction of the cross-linked acrylic PSAs was stronger than that of the neat acrylic PSAs. The strain at maximum stress of neat PSAs from the lap shear test increased with increasing molecular weight; on the other hand, the maximum stress of cross-linked PSAs did not change with molecular weight. According to the stress relaxation test, the stress and recovery of the cross-linked acrylic PSAs increased due to cross-linking effects. Recovery of the neat acrylic PSAs increased with increasing molecular weight due to the entanglement of polymer chains. Regarding the creep test, the strain of the cross-linked acrylic PSAs decreased with increasing molecular weight, which is attributable to entanglement. These results show that the molecular weight and cross-linking of acrylic PSAs are powerful factors affecting adhesion performance, flexibility, and recovery. In addition, these results confirm that DMA is suitable for measuring the flexibility and recovery of acrylic PSAs as well as performing stress relaxation and creep test.

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