

International Journal of Adhesion & Adhesives 25 (2005) 288-295

International Journal of Adhesion & Adhesives

www.elsevier.com/locate/ijadhadh

# Effect of substrate and tackifier on peel strength of SIS (styrene-isoprene-styrene)-based HMPSAs

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> Accepted 4 October 2004 Available online 22 December 2004

#### Abstract

SIS (styrene–isoprene–styrene)-based hot-melt pressure-sensitive adhesives (HMPSAs) have high tack, high cohesion (at ambient temperature), and good adhesion to various substrates. In this study, we investigated the effects of the properties of the substrates and tackifier on the characteristics of the SIS-based HMPSAs. SUS (stainless steel), PE, PP, PVC, Bakelite, Teflon, and glass were used as the substrates. The substrates used in this study were classified into three groups (high, medium, and low peel). A similar classification was obtained using surface tension as the distinguishing factor. The maximum peel strength of the HMPSAs made with high softening point (SP) tackifier shifts to lower tackifier content than those made with low SP tackifier. This may be due to blends made with high SP tackifiers that have higher G' at use temperature than those made with high SP tackifiers. () 2004 Published by Elsevier Ltd.

Keywords: Hot-melt; Pressure-sensitive; Peel; Viscoelasticity

# 1. Introduction

Styrene–isoprene–styrene (SIS) is widely used in pressure-sensitive tapes. Adhesive systems based on SIS can be formulated to give aggressive tack, tailored peel adhesion and high cohesive strength. These are key attributes for tape applications [1]. SIS-based pressure sensitive adhesives (PSAs) are used in numerous applications and using a variety of different substrates. Therefore, the properties of the substrates and the characteristics of the interface have an effect on the performance of SIS-based PSAs.

The peel strength of a PSA-coated flexible material is the force required to remove this material from a specified test surface under standard conditions. This parameter provides a measure of the adhesive or cohesive strength, depending on the mode of failure [2]. Generally, the failure mode is classified into several different types, including cohesive failure, adhesive (or interfacial) failure (between the adhesive and the substrate), stick slip, and glassy failure (failure between the tape and the adhesive) [3].

Adhesive failure occurs when the adhesive strips cleanly from the adherend, leaving no visually noticeable residue. Some adhesives may fail cohesively, leaving adhesive residue on the test panel. If the adhesive is not firmly anchored to the backing, it may transfer from the backing to the test panel, leaving at least part of the backing bare. In the case of transfer tapes, such behavior is intentional, otherwise it denotes a product failure [4,5].

Since the peel edge can be regarded as the tip of an advancing crack, the adhesive ahead of the advancing peel front is subjected to very large hydrostatic tension, due to the lateral constraint imposed by the substrate and the cover sheet [6].

Peel strength data can yield a considerable amount of information about the adhesive character and its

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<sup>0143-7496/\$ -</sup> see front matter  $\odot$  2004 Published by Elsevier Ltd. doi:10.1016/j.ijadhadh.2004.10.001

expected performance, but such data depend on many extraneous factors (the test conditions, backing properties, interfacial conditions and others).

The properties of the substrates constitute an important factor in PSA. PSA adhesion is determined by two key factors. First, the two surfaces must be brought into contact with each other to such a degree that molecular attraction forces come into action (wetting). The second factor is the energy of deformation of the viscoelastic adhesive, which comes into play until rupture or separation from the surface occurs [7,8].

Hata et al. [9] investigated the influence of the critical surface tension of various adherends on the rolling friction coefficient and peel strength of the PSAs. In the velocity region where interfacial failure occurs in the case of acrylic PSA, the rolling friction coefficient and peel strength have a positive correlation with the critical surface tension of the adherends.

The peel force-peel rate curve can be divided into three segments. The dependence of the peel force on the substrate can also be demonstrated by showing how peel rate affects peel force. Not only is the peel force reduced in the case of a higher energy surface, but also the positions of the transitions from cohesive to adhesion and adhesion to stick slip failure are changed [2,5].

SIS-based HMPSAs have high tack, high cohesion (at ambient temperature), and good adhesion to various substrates. In this study, we investigated the effects of substrates and tackifier on SIS-based HMPSAs.

#### 2. Experimental

### 2.1. Materials

The SIS (styrene–isoprene–styrene) selected for this study comprised one diblock containing SIS and one triblock SIS. The diblock containing SIS was Kraton D 1107 (diblock content 15 wt%, styrene 15 wt% made by Kraton polymer), and the triblock SIS was Vector 4111 (diblock <1%, styrene 18 wt% made by ExxonMobil Chemical Co.). The characterization data of the polymers are given in Table 1.

The tackifiers selected for this study included one type of rosin ester and four types of synthetic hydrocarbon resins. The rosin ester was GA 100 (Nichimen Corp.), and the synthetic hydrocarbon resins were Hikorez A 1100S (C-5, Kolon Chemical Co. Ltd.), Regalite R 125 (Hydrogenated C-9, Eastman Chemical Co.), Quintone U 185 (C5-C9, Nihon Zeon Corp.) and Sukorez SU 100 (Hydrogenated DCPD, Kolon Chemical Co. Ltd.). The properties of the tackifiers are shown in Table 2.

## 2.2. Preparation of HMPSAs

The HMPSAs were blended in an internal mixer at 170-180 °C. An antioxidant, Irganox 1010, was used as a thermal stabilizer. The SIS/tackifier blend ratios were 30/70, 40/60, 50/50, and 60/40 by weight.

The PSA specimens were prepared by melt-coating onto a PET film with an average thickness of  $75 \,\mu$ m, using an automatic film applicator with a hot-plate

Table 1		
Polymer	characterization	data

-								
Trade name	Types	Styrene content (wt%) <sup>a</sup>	Diblock content (wt%) <sup>a</sup>	$M_{ m w}{}^{ m b}$	$M_n^{b}$	$M_{ m w}/M_{ m n}$	<sup>b</sup> $T_{g}^{c}(^{\circ}C)$	Manufacturer
Kraton D1107	Linear SIS	15	15	129,000	106,000	1.21	-61	Kraton Polymer
Vector 4111	Linear SIS	18	< 1	114,000	108,000	1.06	-61	ExxonMobil Chemical Co.
<sup>a</sup> Data supplie <sup>b</sup> Determined 1 <sup>c</sup> Determined 1	d by the manu by GPC. by DSC.	facturer.						
Table 2 Tackifiers prope	rties							
Trade name	Types		Softenin	g point (°C) <sup>a</sup>	$M_{ m w}{}^{ m b}$	$M_n^{b}$ M	$M_{\rm w}/M_{\rm n}^{\rm b}$ $T_{\rm g}^{\rm c}$ (	°C) Manufacturer
<u>a.</u>								

Trade name	Types	Softening point (C)	$M_{\rm W}$	Mn	$M_{\rm W}/M_{\rm n}$	$I_g$ (C)	Manufacturer
GA-100	Rosin ester	95–105	857	532	1.61	47	Nichimen Corp.
Hikorez A-1100S	Aliphatic hydrocarbon	98	946	382	2.48	46	Kolon Chemical Co.
Regalite R-125	Hydrogenated Aromatic hydrocarbon	123	816	369	2.21	68	Eastman Chemical Co
Quintone U-185	Modified C5	86	1580	478	3.30	39	Zeon Corp.
Sukorez SU-100	Hydrogenated dicyclopentadiene	105	200	105	1.91	56	Kolon Chemical Co.

<sup>a</sup>Determined by Ring and Ball methods. Data supplied by the manufacturer.

<sup>b</sup>Determined by GPC.

<sup>c</sup>Determined by DSC.

(Kee-Pae Trading Co.) operated at  $150 \,^{\circ}$ C. A No. 9 bar coater (wetting thickness 20.6  $\mu$ m) was used.

# 2.3. Molecular weight

The weight average molecular weight  $(M_w)$ , the number average molecular weight  $(M_n)$  and the molecular weight distribution  $(M_w/M_n)$  of the HMPSAs (SIS/tackifier:50/50) were determined by gel permeation chromatography (GPC, Waters Co.).

## 2.4. Thermal properties

The glass transition temperatures  $(T_g)$  were measured using a differential scanning calorimeter (DSC, TA Q-1000, in NICEM at Seoul National University). The samples were first cooled to -80 °C, then heated to 150 °C at a heating rate of 5 °C/min (first scan). Then, they were immediately quenched to -80 °C and kept at this temperature for 5 min. The sample were than reheated to 150 °C at a heating rate of 5 °C/min (second scan). The  $T_g$  defined in this study was obtained from the second scan to assure reproducible thermograms free from thermal history effects.

## 2.5. Viscoelastic properties

The viscoelastic properties (storage modulus, loss tangents and complex viscosities) of the blends were determined using ARES (advanced rheometrics expansion system, Rheometric Scientific Inc., in NICEM at Seoul National University) in the 8 mm parallel plate mode. A typical scan covered the range from -40-120 °C.

## 2.6. Peel strength

To examine the effects of the surface tension of the substrates on peel strength, seven different substrates were used. The substrates were SUS (stainless steel), PE (polyethylene), PP (polypropylene), PVC (polyvinylchloride), Bakelite, Teflon and glass. The surface tensions of the substrates are listed in Table 3.

The surface tension measurements were obtained using an image analysis system (SEO 300A, Surface &

Table 3			
Surface	tension	of	substrates

Substrates	SUS	PE	РР	PVC	Bakelite	Teflon	Glass
Surface tension $(\gamma_c)^a$ (mN/m)	b	31	33	37	31	18	73

<sup>a</sup>Determined by contact angle measurement.

<sup>b</sup>Not available.

Electro-Optics Corp.), by calculating the contour of a drop observed in an image captured by means of a video camera. In order to determine the surface free energy, the acid-based theory and the three liquid method were used. The contact angle components of the probe liquids used in this study were diiodomethane, formamide, distilled water, ethylene glycol, and glycerol.

The HMPSA specimen was pressed onto the substrate by two passes of a 2 kg rubber-roller. The  $180^{\circ}$  peel strength of the HMPSA specimens coated onto PET (polyethylene terephthalate) was measured by tension with a cross-head speed of 75, 150, 300 and 600 mm/min at room temperature after keeping the specimen at this temperature for 1 h.

#### 3. Results and discussion

## 3.1. $T_g$ as a function of tackifier content

The properties of the PSAs are known to be strongly dependent on the glass transition temperature of the adhesive. However, exploiting this correlation can be complex, because there is more than one way to measure the glass transition temperature,  $T_g$ , and the value of  $T_g$  can vary considerably depending on the measurement method which is employed [10,11].

A mixture of two compatible polymers with different  $T_g$  values exhibits a single  $T_g$ , which is influenced by the individual  $T_g$  values and weight fractions of the two components. In many cases, the  $T_g$  value of the mixture can be approximately determined by means of the Fox equation [12].

$$1/T_{\rm gm} = w_1/T_{\rm g1} + w_2/T_{\rm g2}$$

where  $T_{\rm gm}$ ,  $T_{\rm g1}$ ,  $T_{\rm g2}$  are the glass transition temperatures of the mixture and of components 1 and 2, and  $w_1$ ,  $w_2$ are the weight fractions of components 1 and 2 [10–13].

The DSC curves of the HMPSAs is shown in Fig. 1.

According to many previous studies [3,14], miscible blends have composition-dependent  $T_g$  values.

For all of the blends, only one well-defined glass transition lying between the glass transition of the pure components was detected for each blend, and it changed gradually depending on the composition of the blend. The existence of one composition-dependent  $T_g$  value is evidence that the SIS materials used in this study (Kraton D 1107 and Vector 4111) were miscible with the tackifiers used in this study (Hikorez A 1100 S, Regalite R 125, Quintone U 185, Sukorez SU 100, and GA-100) below the  $T_g$ .

## 3.2. Viscoelastic properties

The viscoelastic property of the HMPSAs is shown in Fig. 2. As shown in this figure, plateau modulus



Fig. 1.  $T_g$  by DSC of SIS/Hikorez A 1100S blends. (a) Kraton D 1107 blends; (b) Vector 4111 blends.



Fig. 2. Viscoelastic properties of SIS/Hikorez A 1100S blends. (a) Kraton D 1107 blends; (b) Vector 4111 blends. ( $\circ$  60/40,  $\triangle$  50/50,  $\nabla$  40/60,  $\diamond$  30/70).

decreases as the tackifier content increases. Also, the  $T_g$  (temperature at peak of tan  $\delta$ ) increases as the tackifier content increases.

For all of the blends, only one well-defined glass transition lying between the glass transition of the pure components was detected for each blend and it changed gradually depending on the composition of the blend. The existence of one composition-dependent  $T_g$  value is evidence that the SIS materials used in this study (Kraton D 1107 and Vector 4111) were miscible with the tackifiers used in this study (Hikorez A 1100 S, Regalite R 125, Quintone U185, Sukorez SU100, and GA-100) below the  $T_g$ .

Generally, blending the PSA with the tackifier lowers the modulus and increases the  $T_g$  of the blend. The minimum modulus depends on the tackifier/rubber solubility. On the other hand, a high tackifier loading increases the modulus as well. A lowered modulus always promotes bond formation (creep compliance), while a higher  $T_g$  invariably makes bond rupture more difficult (debonding resistance) [1,2,8].

In terms of the diblock content, the tan  $\delta$  of the blends made using triblock SIS was higher than that of blends made using diblock containing SIS. This is due to the fact that the inherent elastic response (G') of the triblock copolymer network is diminished and the corresponding inelastic or loss modulus response (G'') is increased. The polydiene endblock on the SI diblock copolymers imparts an imperfection within the triblock thermoplastic elastomer network, in effect weakening it [1,10,11].

## 3.3. Peel strength

#### 3.3.1. Effects of surface tension of substrates

The  $180^{\circ}$  peel strength of the SIS/Hikorez A 1100S (40/60) blends at various peeling rates are shown in Fig. 3.

As shown in this figure, the substrates used this study were classified into three groups (high, medium and low peel). High peel strength was observed in the case of SUS and glass. Medium peel strength was observed in the case of Bakelite, PVC and PP, and low peel strength was observed in the case of PE and Teflon. A similar classification was obtained using surface tension as the distinguishing factor.



Fig. 3. Peel strength of SIS /Hikorez A 1100S (40/60) blends as a function of the peel rate. (a) Kraton D 1107 blends; (b) Vector 4111 blends. ( $\Box$  SUS,  $\circ$  PE,  $\triangle$  PP,  $\nabla$  PVC,  $\diamond$  Bakelite,  $\triangleleft$  Teflon,  $\triangleright$  Glass).

Although PE exhibits a surface tension which is similar to that of medium peel substrates, it has low peel strength. This is due to differences in the failure mode.

Urahama et al. [15] investigated the relationship between the failure mode and the peel rate. According to their results, the peel failure mode changes from cohesive failure within the adhesive to adhesion failure with increasing peeling rate.

Interfacial failure was observed in the case of PE, while cohesive failure was observed in the case of other medium peel substrates. This may be due to differences in the characteristics of the substrates.

In our previous study [16], we investigated the peel strength of various SIS/tackifier/plasticizer blends using PE and PP substrates. According to this study, in the peel test performed using the PP substrate, cohesive failure occurred at various test temperatures. However, in the peel test performed using the PE substrate at  $25 \,^{\circ}$ C, adhesive failure (interface failure) and slip stick occurred in the case of the Kraton D 1107-based blends and the Vector 4111-based blends, respectively. At an aging condition of 100 °C, cohesive failure occurred, except in the case of the Regalite R 125 based blends. This is due to increasing adhesive wetting on the substrates during heating. The peel strength at an aging condition of 100 °C was higher than that observed at 25 °C.

Itoh et al. [17] investigated the peel strength of different SIS/tackifier blends using various substrates. According to their study, the peel strength on the PE substrate ( $\gamma_c = 28.1 \text{ mN/m}$ ) was lower than that on the PP ( $\gamma_c = 25.8 \text{ mN/m}$ ) substrate. The peel strength on the PE substrate with hydrogenated aliphatic hydrocarbon blends was similar to that on the Teflon ( $\gamma_c = 18.8 \text{ mN/m}$ ).

Their results were similar to those obtained in our study. The main difference was the weaker relationship between the failure mode and the peel rate observed in their studies; however, the reasons for these differences have not been clearly elucidated. In the case of the low surface tension substrates, two types of interfacial failure were observed. One type was stick slip, and the other was cohesive failure. Cavitations and long fibrillations were observed in the case of cohesive failure.

The S–S curve of the Kraton D 1107/Sukorez SU 100 blends with the PE substrate is shown in Fig. 4. In the Kraton D 1107/Sukorez SU 100 (40/60) blends, a stick slip type of S–S curve was observed (Fig. 4-a), while, a cohesive failure type of S–S curve (Fig. 4-b) was observed in the Kraton D 1107/Sukorez SU 100 (50/ 50) blends. The curve in Fig. 4-a corresponds to stick slip, but the adhesive was stripped cleanly from the adherend leaving no visually noticeable residue. Therefore, the surface was clear after the test. This is due to the high cohesion of the SIS-based HMPSAs with a high SIS content.

Zosel [18] investigated the relationship between the peel force and the surface properties. The peel force obtained after a short contact time is heavily dependent upon the critical wetting tension of the substrate. If the wetting tension of the substrate is substantially lower than the surface energy of the adhesive, the result is a lower peel force.

The dependence of the peel force on the substrate has also been reported [9]. Not only is the peel force decreased in the case of a higher energy surface, but also the positions of the transitions from cohesive to adhesion and adhesion to stick slip failure are changed. This indicates that the activation of the stiffening behavior of a PSA depends upon the substrate to which the adhesive is bonded.

## 3.3.2. Effects of tackifiers

The peel strength of the SIS/tackifier blends at various tackifier contents are shown in Fig. 5.

As shown in this figure, those blends made using Sukorez SU 100, Hikorez A 1100S and Quitone U 185 exhibit maximum peel strength at 60 wt% of tackifier



Fig. 4. S-S curve of interfacial failure on low surface tension substrate (PE). (Kraton D 1107/Sukorez SU 100; 40/60). (a) stick slip type; (b) cohesive failure type.



Fig. 5. Peel strength of SIS/tackifier blends on SUS substrates (peeling rate:300 mm/min). (a) Kraton D 1107 blends; (b) Vector 4111 blends. ( $\circ$  Hikorez A 1100S,  $\triangle$  Regalite R 125,  $\nabla$  Quintone U 185,  $\diamond$  Sukorez SU 100,  $\Box$  GA-100).

content, while those blends made using GA-100 and Regalite R 125 exhibit maximum peel strength at 40–50 wt% of tackifier content. Similar results were obtained for the seven different substrates. This is due to the characteristics of the tackifiers used in this study. In our previous study [16], those HMPSAs made with SIS/Regalite R 125 exhibited higher maximum peel strength than those HMPSAs made with other tackifiers. Regalite R 125 has a high softening point and  $T_g$  and those HMPSAs made with this tackifier have brittle properties. Thus, the maximum peel strength of the HMPSAs made with Regalite R 125 shifts to lower tackifier content.

It is a fact that the tack and peel adhesion (especially the peel on nonpolar, untreated surfaces) of PSAs by themselves (independent of their chemical basis, converting technology, and end-uses) do not satisfy most practical requirements. Therefore, it is usually necessary to formulate (compound) the raw adhesives with chemicals providing a better level of tack and peel [2,5].

A lot of publications have shown the effect of tackifiers on peel strength of PSAs.

Fujita et al. [3] investigated the effects of miscibility on the peel strength of natural rubber-based PSAs. According to their study, in the case of miscible PSAs, the peak positions in the pulling rate-peel strength curve shifted to a lower velocity as the tackifier content increased. Immiscible PSAs had lower peel strengths than miscible ones, and did not exhibit any apparent shifts in their peaks.

Hayashi et al. [19] investigated the miscibility and PSA performances of acrylic copolymers and hydrogenated rosin systems. According to their study, in the case of the miscible blend systems, as the molecular weight of the tackifier increases, the viscoelastic properties (such as the storage modulus and the loss modulus) shift toward a higher temperature or a lower frequency.

Takashima and Hata [20] investigated the adhesive properties and viscosity of block copolymers mixed with tackifier resins. According to their study, the addition of a resin which is compatible with the rubber phase increases the peel strength and tackiness of the mixtures, with a resultant maximum peel strength being observed at 75 wt% of the resin and the maximum tackiness at 50 wt%.



Fig. 6. A schematic illustration of the effects of substrate and tackifier on the peel strength of SIS-based HMPSAs. (a) effects of substrates; (b) effects of tackifiers.

#### 3.4. Schematic illustration

A schematic illustration of effects of surface tension of substrate on peel strength of SIS-based HMPSAs is shown in Fig. 6. As shown in the figure, substrates used in this study were classified into three groups (high, medium, and low peel). A similar classification was obtained using surface tension as the distinguishing factor.

The maximum peel strength of the HMPSAs made with high softening point (SP) tackifier shifts to lower tackifier content than those made with low SP tackifier. This is may be due to the fact that blends made with high SP tackifiers have higher G' at use temperature than those made with low SP tackifiers.

## 4. Conclusion

For all of the blends, glass transition temperature changed gradually depending on the composition of the blend. The existence of one compositiondependent  $T_g$  is evidence that the SIS materials used in this study were miscible with the tackifiers used in this study.

The substrates used in this study were classified into three groups (high, medium and low peel). High peel strength was observed in the case of SUS and glass. Medium peel strength was observed in the case of Bakelite, PVC and PP, and low peel strength was observed in the case of PE and Teflon. A similar classification was obtained using surface tension as the distinguishing factor.

In the case of the low surface tension substrates, two types of interfacial failure were observed. One type was stick slip, and the other was cohesive failure. Cavitations and long fibrillations were observed in the case of cohesive failure. Those blends made using Sukorez SU 100, Hikorez A 1100S and Quintone U 185 exhibited maximum peel strength at 60 wt% of tackifier content, while those blends made using GA 100 and Regalite R 125 exhibited maximum peel strength at 40–50 wt% of tackifier content.

Those HMPSAs made with a tackifier which has a high softening point and  $T_g$  have brittle properties due to higher modulus. Thus, the maximum peel strength of these tackifier/SIS blends shifts to a lower tackifier content.

## Acknowledgements

This work was partially supported by the Brain Korea 21 Project and by Vixxol Corporation.

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