EB Application In Pressure Sensitive Adhesives

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SYNOPSIS

Two kinds of pressure sensitive adhesives (PSA's), that were formulations of radiation crosslinkable styrene-isoprene block copolymer (SIS) and complete hydrogenated aliphatic tackifying resin or non-hydrogenated, were prepared and the electron beam (EB) irradiation effect on these PSA performances such as peel strength against some kinds of adherends was studied. The results from measuring of PSA performance exhibit the close correlation between EB irradiation effect of these and the miscibility of the tackifying resin against SIS. Further it was clarified that PSA performance was influenced by the surface tension of adherends.

INTRODUCTION

At RadTech Asia '97, we focused on holding power of PSA based radiation crosslinkable SIS and reported about the improvements of heat and solvent resistance of these through EB irradiation[1]. Results showed that the improvement was influenced by the miscibility of PSA mixtures which derived from the molecular structure of tackifying resin. SIS has the micro-phase structures that consist of polystyrene and polyisoprene phase, because these are intrinsically immiscible. PSA performance and EB irradiation effect on these were determined by which phase the tackifying resin dissolved in.

In this report, we discuss mainly the following points.

- The correlation between EB irradiation effect on PSA performance such as peel strength and PSA elasticity derived from the difference of the miscibility of these formulations.
- The correlation between PSA performance and the surface of adherends.

EXPERIMENTAL

Material and test sample

PSA material used in this study were as follows: radiation crosslinkable SIS; Kraton D1320(Shell Chemical Co.), tackifying resins; complete hydrogenated aliphatic hydrocarbon resin; ArkonP100(Arakawa Kagaku-kogyo Co.) and non-hydrogenated; Quintone1325(Nihon Zeon Co.). The difference of both tackifying resins was only hydrogenated or no. Adherend materials of testing PSA's performances were used as follows: Poly tetrafluoro ethylene(PTFE), Poly propylene(PP), Poly ethylene(PE), Poly vinyl chloride(PVC), and Poly methyl methacrylate (PMMA). The PSA was made by mixturing SIS and the tackifier in toluene at the weight ratio of 40/60, 50/50, 60/40. The test samples for measuring PSA performance were prepared by coating the mixtures on 25 micrometers polyethylene telephthalate films, which resulted in a final dry adhesive volume of 3mg/cm^2. Electron beam irradiation was carried out in a nitrogen atmosphere using a Nisshin Curetron System. The cathode voltage was held at 200keV and the dosage at 100kGy.

Measurements

PSA performance against 5 kinds of adherends was evaluated by 180 degree peel. 180 degree peel was tested at 300 mm/min peel speed according to JIS-Z0237.

Elasticity (shear modulus) and Tan δ were measured by Dynamic mechanical spectrometer (Seiko,Inc.) in the parallel plate mode using small plates of PSA(cross section:1cm^2,thickness: 1mm). These were plotted from the room temperature to 150℃ at 0.05Hz. Glass transition temperature(Tg) of polystyrene phase in SIS was determined by the peak of Tan δ.

The critical surface tension of adherends was estimated by contact angle method. The contact angle of aqueous solutions of dipropylene glycol was measured by Erma goniometer. The critical surface tensions were determined by Zisman's plot.

RESULTS AND DISCUSSION

Table I shows the changes in peel strength after EB irradiation and against different kinds of adherends. The result leads to the following trends; 1) In almost all conditions, peel strength became slightly lower after EB irradiation, creating SP46, SQ55 and SQ46 slip stick phenomena. 2) The difference of peel strengths between SP and SQ series increased with the content of tackifying resin, although peel strengths of SP64 and SQ64 show almost same. Comparing SP46 and SQ46, the latter, formulated with non-hydrogenated aliphatic resin as tackifying resin, performed clearly worse than the

Table I EB irradiation and adherends effect of peel strength

Formulation_dose(kGy)/Adherends	PTFE	PP	PE	PVC	PMMA
SP64_0	0.47	1.19	0.63	1.20	0.95
SP64_100	0.24	0.82	0.35	1.10	1.01
SP55_0	0.78	1.32	0.91	1.43	1.65
SP55_100	0.47	1.11	0.74	1.43	1.33
SP46_0	0.69	2.11	1.28	1.83	2.50
SP46_100	0.42*	1.87	0.27*	1.91	1.99
SQ64_0	0.55	1.18	0.79	1.33	1.43
SQ64_100	0.49	0.95	0.59	1.16	1.01
SQ55_0	0.50	1.25	1.03	1.42	1.51
SQ55_100	0.31*	1.20*	0.91*	1.41	0.65*
SQ46_0	0.22*	1.27	1.09	1.50	1.56
SQ46_100	0.29*	1.00*	0.42*	1.44*	0.76*

UNIT : kg/25mm

SP : formulation of SIS and ArkonP100 SQ : formulation of SIS and Quintone1325

* : slip stick phenomena

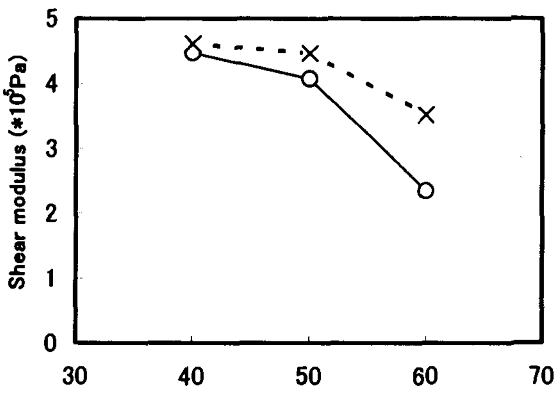
former formulated with hydrogenated. 3) Peel strength against various adherends shows a regular increase in the order PTFE < PE < PP < PVC < PMMA.

Table II The changes in elasticity after EB irradiation

formulation/dose(kGy)	0	100
SP64	4.24	4.78
SQ64	4.59	5.06

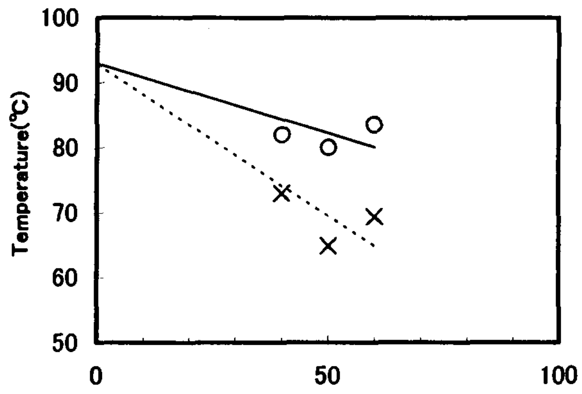
UNIT : $x10^5$Pa

Table II shows the changes in shear modulus of SP64 and SQ64 at room temperature after EB irradiation. Both shear moduli were increased with the dose of EB irradiation. It was considered that the increasing of elasticity had an effect on the decline of peel strength. But the explain could not simply apply in case of slip stick phenomena because of drastic changes.



FigI Shear modulus at RT vs tackifying resin content

—O— SP series - ×- SQ series



FigII Tg of polystyrene phase vs tackifying resin content

O SP series ×SQ series

Fig.I shows the changes in the correlation between the content of tackifying resin and the shear modulus at room temperature. Shear moduli of both formulations decrease with increasing the content of tackifying resin. Therefore it was considered that both peel strengths increase with the content of tackifying resin. Besides it was proved that the changes in shear modulus were dependent on the kind of tackifying resin, observing from the result that the degree of downward trend in formulation of SP was larger than SQ. In consequence, the difference of the changes would be reflected in peel strength.

Fig.II shows the Tg curves derived from polystyrene phase as the content of tackifying resin increases. The effects of an addition of tackifying resins on the Tg exhibit the miscibility to polystyrene phase in SIS. The result demonstrates that the non-hydrogenated tackifying resin was more miscible to the polystyrene phase than the hydrogenated. In previous report[1], it demonstrated that the hydrogenated tackifying resin was good miscibility to the polyisoprene phase. Consequently it was apparent that the close relation between PSA performance derived from the elasticity and the miscibility of PSA mixture.

Besides the miscible effect of the EB irradiation on PSA performance was considered the followings. Increasing the content of non-hydrogenated resin that was more immiscible to polyisoprene-phase than hydrogenated, the separate-phase occurred in polyisoprene-phase. Consequently the changes in PSA performance after EB irradiation showed different between miscible and immiscible condition because the polyisoprene-phase would be mainly crosslinked(by EB irradiation).

Table III Critical surface tension of adherends

	PTEF	PP	PE	PVC	PMMA
γ_c (measurements)	18.8	25.8	28.1	34.6	39.9
γ_c (literature)	18.5	29.0	31.0	39.0	39.0

UNIT : mN/m

The critical surface tensions of adherends obtained from experiments and literatures[2] are shown in Table III. The properties of the surface of adherends are affected by the condition in processing and molding. But the experimental results agree approximately with the values of literatures except PVC. Considering from Table I and III, there is a linear correlation between peel strength and the critical surface tension if classifying PTFE into low critical surface tension material, PP and PE into medium, PVC and PMMA into high.

Conclusion

We presented the results that the miscibility of PSA mixture affected the elasticity itself, therefore the PSA performances were changed. We found the correlation between the miscibility of PSA mixture and the EB irradiation effect on PSA performance. Further it was clarified that PSA performance was influenced by the surface tension of adherends.

References

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