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## Contents Lists and Abstracts from the Journal of the Adhesion Society of Japan

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# Contents Lists and Abstracts from the Journal of the Adhesion Society of Japan

## Journal of The Adhesion Society of Japan Vol. 29 No. 2 1993

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### Preparation of the Polymer Containing Malononitrile Structure and Its Application to Thermally Latent Cationic Catalyst

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### Abstract

Thermally latent cationic catalysts have become more important in the curing of epoxy resin and so on. We already reported that benzylsulfonium and benzylammonium salts served as thermally latent cationic catalysts. In this paper, we wish to report a new thermally latent cationic polymer catalyst, which is a complex of  $\text{SnCl}_4$  with polystyrene containing the malononitrile structure.

The requisite polymer was prepared by the polymer reaction of styrene-chloromethylstyrene copolymer with malononitrile sodium salt formed from malononitrile and sodium hydride. The polymer obtained was then allowed to react with  $\text{SnCl}_4$  to afford a polymer catalyst, which was stable in the air. Next, the cationic polymerization of styrene oxide was carried out by using the polymer catalyst. Although the polymerization did not proceed at room temperature, the polymerization was initiated by the polymer catalyst at higher than  $80^\circ\text{C}$ . In this way, we could demonstrate that the complex of  $\text{SnCl}_4$  with the polymer containing malononitrile structure served as a new type of thermally latent cationic polymer catalyst.

(Received: May 18, 1992)

### The Reaction of Bismaleimide and Propargylalcohol in the Presence of Epoxy Resin

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### Abstract

By reacting N, N'-4, 4'-diphenylmethane-bismaleimide (BMI) and propargylalcohol (PA) in the presence of bisphenol A type epoxy resin (Ep828) at  $140^\circ\text{C}$  for 2 hours, a homogeneous liquid was obtained (nearly 100% of BMI reacted, and about 24% of PA reacted). This liquid provided good storage-stability.

For the investigation of this reaction, N-phenylmaleimide (PMI), phenyl glycidyl ether (PGE) and PA were selected as model compounds. Two main reactions were recognized. One was the anionic polymerization  $\{\text{PA}-(\text{PMI})_n\}$  caused by the Michael addition reaction between PMI and PA and the other was the radical polymerization  $\{-(\text{PMI})_n\}$  initiated by the charge-transfer complex formed between PMI and PA. PGE reacted only a very little, under 1%, but it acted as a chain transfer agent.

The reactions of BMI and PA were the main reactions. The products of these reactions coexisted with unreacted Ep828 and PA in a state of good compatibility. Steel plates were pasted together with the mixture of BMI, Ep828, PA/2, 4-bis-para-amino benzyl aniline. In the case of exposing them at  $200^\circ\text{C}$  for five months, the change of thermal property was not recognized.

(Received: June 2, 1992)

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**Structure and Properties of Thermosetting Epoxy Adhesives Modified with Acrylic Particles**

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**Abstract**

Structure and properties of thermosetting epoxy adhesives modified with acrylic particles were investigated Poly(methyl methacrylate) (PMMA) and methyl methacrylate-glycidyl methacrylate copolymers {P(MMA-GMA)} were used to modify epoxy resin cured with dicyandiamide (DICY). The adhesive formed a thermoplastic gel by heating and it cured at the activating temperature of DICY. The P(MMA-GMA) blend system revealed higher adhesion strengths than the PMMA blend system. TEM micrographs and the dynamic mechanical data showed that swelled acrylic particles, by imbibing epoxy resin, were cured with the epoxy matrix in the P(MMA-GMA) blend system. On the other hand, micro phase separation was observed in the PMMA blend system. Accordingly, it was concluded that the differences of adhesion strengths between the P(MMA-GMA) blend system and the PMMA blend system are based on the phase structure of cured resin which is affected by the reactivity of GMA with the epoxy matrix and the degree of crosslinking in acrylic particles.

(Received: May 15, 1992)

**Effects of Curing Conditions in Adhesion on the Bonding Strength of Oily Steel Plates**

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(Accepted for publication: September 5, 1992)

**Abstract**

The properties of an adhesive for use on oily surfaces were evaluated by changing the curing conditions. A T-peel test was carried out, and the fractured surface was observed. The observed bonding strength was largely influenced by the curing temperature. However, this dependence was not due to the real adhesion strength but due to bulk properties of the adhesive, because fractures occurred within the adhesive sheet, *i.e.*, cohesive failure. This fact was also proven by using tension testing on the sheet prepared from the adhesive only. Namely, the strength of the sheet varied along with the adhesion one which was observed in this study. Interfacial failure was partly observed when the oil thickness on the steel plate was increased, leading to a decrease of adhesive strength.

(Received: July 13, 1992)

**Journal of The Adhesion Society of Japan**  
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**Studies on Compoundings for Adhesion of Natural Rubber and Steel Cord**

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(Accepted for publication: October 5, 1992)

### Abstract

The adhesion between rubber and steel cord is one of the most important factors controlling the quality of steel cord reinforced rubber composites. The influence of the compounding ingredients on the adhesion strength was investigated by changing the amount of cobalt, accelerator, and sulfur under various conditions. The pull-out strength was assumed to be a measure of adhesion, which was measured before and after being subjected to various aging conditions and dynamic fatigue. The dynamic fatigue was measured using a flexometer and a heat build up tester for rubber goods. Excellent adhesion strength of compounds after water or salt agings was obtained by increasing cobalt up to 2 phr. Compounds with more than 2 phr produced negative effects. It was also found that a low accelerator and high sulfur system produced high and stable adhesion strength. The change of adhesion strength after the dynamic fatigue was explained by the effect of initial tensile properties rather than the effect of the compounding ingredients.

(Received: September 2, 1992)

### A Simplified Design Process of Adhesive-bonded Joints Based on Static and Fatigue Strength

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(Accepted for publication: October 14, 1992)

### Abstract

In the procedure for the design of adhesive-bonded joints, there are, in general, seven main steps, as follows.

- Step 1. Determine the design ultimate load  $P_B/P = M_B/M = T_B/T = 4$  (so-called factor of safety = 4), the design environmental temperature  $\theta_c$ , and the number of cycles of repeated loading  $N$  (where  $N=1$ , being called static design and where  $N=10^7$ , namely fatigue design).
- Step 2. Determine the values of the modulus of transverse elasticity  $G_a$ , the shearing strength  $\tau_B$  and the tensile strength  $\sigma_{aB}$  of the adhesive by Eq7 and Eq8.
- Step 3. Determine the values of the shearing strength  $\tau_{IN}$  and the tensile strength  $\sigma_{BN}$  of the adherend under the number of cycles  $N$  by Eq9.
- Step 4. Determine the most suitable size of adherend by Eq0-2.
- Step 5. Determine the most suitable length of overlap  $l = 1.2l_M$  by using Eq5.
- Step 6. Determine the most suitable thickness of adhesive  $d = 0.9d_m$  by using Eq6.
- Step 7. Draw the lines of the Equations 1, 2, 3 and 4 on the  $\tau_u \sim d$  or  $\sigma_u \sim d$  log-log diagram.

The classification of the type of failures is given by: cohesive failure in the adherend (Eq1); interfacial failure (Eq2); cohesive failure in the adhesive layers (Eq3); and adhesive failure (Eq4). Four criteria of the failures are summarized in four formulae (Eqs. 1, 2, 3 and 4).

(Received: September 9, 1992)

### **An Aqueous Emulsion Adhesive for Dry Lamination of Polyethylene Terephthalate Film and Polyethylene-vinylalcohol Film Part 2**

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(Accepted for publication: October 14, 1992)

### **Abstract**

Water-borne dry laminating adhesives are becoming an increasingly attractive solution for the many environmental, health, and safety problems, however there are still some problems remaining unsolved such as coating performance and adhesion.

A one component acrylic-epoxy emulsion which was dispersed independently in an aq. solution was applied to the dry lamination of high gas barrier film of polyethylene-vinyl alcohol and high mechanically strong film of polyethylene terephthalate.

This high solid emulsion adhesive showed a good coating performance, good drying character, and good transparent clarity, and was also shown to be suitable for biaxial stretching of this laminate film.

Bonding strength was increased by the heat setting in this biaxial stretching stage.

The laminate film has a good heat-sealing character, high boiling ability, and good vacuum-packed ability, and was shown to be suitable for flexible package film.

(Received: August 3, 1992)

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**Report of the Mission of the Adhesion Society of Japan to U.S.A. (I)** ..... [244]**Adhesion between the Polypropylene Film Treated with O<sub>2</sub> Plasma and the Vacuum Deposited Aluminum Film**

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(Accepted for publication: October 26, 1992)

**Abstract**

Polypropylene film (PP) was treated with RF-induced O<sub>2</sub> plasma, followed by the vacuum deposition of aluminum thin film (Al), and the peel strength of the PP/Al laminate was examined. The peel strength of the treated-PP/Al varied widely from 6 to 240gf/15mm, depending upon the conditions of the plasma treatment, whereas the peel strength of the untreated-PP/Al was 8gf/15mm. The peel strength was minimized at the O<sub>2</sub> pressure of ca. 0.1 Torr. A higher peel strength was obtained as the discharge power decreased to 5W. The peel strength rapidly increased at the initial stage of the plasma treatment (~several seconds), decreased at the second stage, and increased again gradually at the third stage. A good relationship was found between the peel strength of PP/Al and the amount of oxygen introduced onto the PP surface only at the initial stage. At the second stage, a large content of carbon was found by XPS on the Al side of the peeled interface of PP/Al, having the decreased peel strength. SEM photographs showed that the PP surface was etched by O<sub>2</sub> plasma at the third stage. These adhesion behaviors of PP/Al were explained by the surface changes caused by O<sub>2</sub> plasma treatment:

- (1) introduction of O-containing polar groups onto the PP surface at the initial stage,
- (2) the formation of Weak Boundary Layers resulting from the scission of the main chain of PP at the second stage,
- (3) plasma etching of PP surface at the third stage.

(Received: October 1, 1992)

**The Role of Solubility Parameter (SP) in Solubility Theories  
Part 1, Method of Estimating Solubility Parameters (SP) by Means of  
Molar Attraction Constants**

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(Accepted for publication: November 25, 1992)



### Abstract

The most simple method of estimating solubility parameter (SP) was a method by means of Small's molar attraction constants ( $\Delta F$ ), but this method was remarkably inaccurate in the case of the solvents and the polymers with hydrogen bonding. The author will suggest more accurate  $\Delta F$  values in this paper, therefore more accurate SP values of 113 solvents and 30 polymers were obtained by means of the author's  $\Delta F$  values. Then the author will report that SP value of Epoxy/Dimer acid polyamide adduct calculated by means of the author's  $\Delta F$  nearly coincided with SP value of the solvent which showed maximum swelling for these polymer adduct films in various solvents.

(Received: September 19, 1992)

### Effects of Resin Construction on the Compression Shear Strength of the Epoxy Resin System with Some Foreign Matter Inserted

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(Accepted for publication: November 30, 1992)

### Abstract

When foreign matter was inserted into the epoxy matrix, its apparent bonding strength depended on both the ordinary adhesion strength of the matrix and the strangling pressure generated with the contraction of matrix. It was tried in this report to evaluate these two factors separately.

The apparent bonding strength  $\sigma_a$  of the inserted system is expressed by the equation:  $\sigma_a = A + k \cdot P_N$ , where  $A$ ,  $P_N$ , and  $k$  are the ordinary bonding strength, the strangling pressure, and the constant, respectively.

Thus, we could estimate the values of  $A$  from the data of the temperature and the curing dependence of  $\sigma_a$  values in the region where the  $\sigma_a$  is proportional to the  $P_N$ .

The values of  $A$  increased remarkably in the initial stage of curing and kept a constant value in the stage after the vitrification point.

(Received: September 25, 1992)

### On Glass-Glass Adhesive of Ultrasonic

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(Accepted for publication: November 26, 1992)

### Abstract

The purpose of this article is to investigate the effect of tensile adhesive shear strength on glass-glass adhesive of ultrasonic under the static pressure from 600 to 1200gr (2.12 to 4.24kgf/cm<sup>2</sup>) and PTFE tapes from 50 to 500  $\mu$ m.

The film adhesives are nitrile-phenolic resin, polyamide and EVA.

The results obtained are as follows:

- 1) The empirical formula for tensile adhesive shear strength of glass-glass bonded joint on nitrile-phenolic film adhesive is presented in the following

$$\sigma = (\sigma_{00} + n_1 t) + (m_0 + n_2 t) \log \delta$$

where  $\sigma$  is the tensile adhesive shear strength,  $\sigma_{00}$  is the tensile adhesive shear strength at tape thickness 50  $\mu\text{m}$ ,  $m_0$  is the amount of strength change per log of  $\delta$ ,  $n_1$  and  $n_2$  are constants,  $t$  is weld time and  $\delta$  is the tape thickness.

The value of  $n_1$  ranges from 1.4 to 2.0, and that of  $n_2$  from  $-0.6 \sim -0.4$ .

- 2) The tensile adhesive shear strength of glass-glass bonded increases with weld time or static pressure from 600 to 1200gr and PTFE tape thickness from 50 to 500  $\mu\text{m}$ . Also ultrasonic adhesion agreed with the qualitative theory for bonding with the frictional heat bond.

(Received; September 24, 1992)