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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. 31 No. 7 1995

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#### Improvement of Adhesion Properties of Polyolefins by Sand Blasting

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(Accepted for publication: December 8, 1994)

#### Abstract

This paper is concerned with the adhesion of coating film on polyolefin surfaces by applying sand blasting. A main component of sand blasting materials is  $Al_2O_3$ . Various

kinds of  $Al_2O_3$  having different particle sizes were used. It was proved that the sand blasting was very effective for the adhesion. Furthermore, the optimum surface roughness was confirmed for the adhesion. The surface of polyolefins after sand blasting was analysed by ESCA (Electron spectroscopy for chemical analysis). However, no polar groups were found on the surface. We can conclude that the adhesion of coating film on polyolefins can be attained only by anchoring effect.

(Received: November 22, 1994)

#### A Two-Dimensional Finite Element Thermal Stress Analysis of Butt Adhesive Joints of Dissimilar Adherends

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(Accepted for publication: April 17, 1995)

#### Abstract

Thermal stress distributions in a butt adhesive joint of dissimilar adherends are analyzed by using a two-dimensional finite element method (FEM). Both end surfaces of the joint are kept at a constant temperature and the other surfaces at a different temperature. The effects of the thermal expansion coefficient ratios, Young's modulus and the thickness of the adherend to those of the adhesive on the thermal stress distributions are studied. Especially, the thermal stress distributions near the edges of the interfaces are examined. It was found that the thermal stress at the interface of adherend with higher Young's modulus is greater than that at the interface of the adherend with smaller Young's modulus. For verification, the photoelastic experiment was carried out. Good agreement was seen between the numerical and the experimental results.

(Received: January 19, 1995)

#### Surface Modication of Aramid Fibers by Excimer Laser Irradiation

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(Accepted for publication: February 22, 1995)

#### Abstract

It was attempted to improve the fiber/rubber adhesion by surface modification of fibers by irradiating aramid fibers (PPODPTA, PPTA) with excimer laser. Both fibers were first treated with glycidyl ether compound containing acrylic acid which is sensitive to ultraviolet rays, and cured with a KrF laser, then treated with an ordinary RFL (resorcinol formalin rubber latex), and cured in heat. As a result, PPODPTA fibers were improved in the adhesion performance, while PPTA fibers were not. A reason was estimated as follows. In PPODPTA fibers, co-polymer components compose crystalline domains independently of each other, and are oriented sufficiently, leaving almost no noncrystalline state in which molecules remain relaxed, whereas PPTA fibers comprise highly oriented skin layers and core layers stacking up fibril groups in blocks, and there is a noncrystalline phase containing relatively many end groups of molecules between blocks, which is likely to be come fibril. Such difference in orientation of noncrystalline phase seems to result in difference in susceptibility to ablation, and difference in affinity of an acrylate compound and fibers, which is considered to be finally expressed as a difference in adhesion performance.

(Received: January 25, 1995)

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#### Dispersion and Polar Components of Surface Free Energy of Transition Metal Films Measured by Contact Angle Method

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(Accepted for Publication: April 24, 1995)

#### Abstract

Dispersion and polar components of surface free energy of various transition metal films (Ti, Ni, Cu, Mo, Pd, Ag, W, Pt and Au) are determined by the contact angle measurement in air. The dispersion components changes cyclically as the atomic number increases. This result indicates that the 3d, 4d and 5d orbitals are filled out continuously with electrons, respectively. The polar components are separated into two groups, (1) Cu, Pd, Ag and Au, (2) Ti, Ni, Mo, W and Pt, in terms of its magnitude. This result corresponds to the d-orbital structure, closed shell and nonclosed one, respectively. These results indicate that the contribution of d-shell structure to cohesive property of metal has close relationship with the components of surface free energy. By using the contact angle method in air, the property of metal can be detected.

(Received: December 9, 1994)

#### A Two-Dimensional Finite Element Thermal Stress Analysis of Butt Adhesive Joints Containing Some Hole Defects

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(Accepted for publication: May 25 1995)

#### Abstract

Thermal stress distributions in a butt adhesive joint containing some hole defects under uniform temperature changes were analyzed by using a two-dimensional finite element method (FEM). Thermal stresses in the cases where 3-hole, 8-hole, and 16hole defects were contained in an adhesive layer and where no hole was contained were studied. Especially, the stress concentrations around the hole defects and near the edge of the interface between adherends and an adhesive were examined. From the results, the thermal stresses around the hole defects located near the center portion of the adhesive layer were larger than those of the hole defects located near the free surface of the adhesive layer. The thermal stress distributions near the edge of the interface decreased with an increase of the all hole areas and with a decrease of the Young's modulus of the adhesive layer. For verification, a photoelastic experiment was carried out. Fairly good agreement was seen between the numerical and the experimental results.

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#### Fracture of Oriental Lacquer Film on the Ware with Water Absorption or Desorption

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(Accepted for publication: June 16, 1995)

#### Abstract

Water absorption and desorption processes were examined experimentally in detail for lacquer wares. It was found that the rate of these processes was governed by thickness of the lacquer film. Further, the stresses on the interfacial strength between oriental lacquer film and wood were analyzed, concering water absorption and desorption processes by finite element analysis (FEM), which is based on the complete elasticity. It was clarified that peeling stress was generated between them under dry conditions, and that tensile stress along with the interface was generated under humid conditions, when the curvature of lacquer wares was convex. These calculated stresses were so strong that fracture was expected to occur at the interface or the lacquer film. However, no fracture was recognized experimentally except for a special case. It is considered that the stresses are relaxed during water absorption and desorption processes.

(Received: April 19, 1995)

#### Detection of Organic Contamination on Aluminum Surface by Water Condensation Method and Adhesion of Photoresist Micro Pattern

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(Accepted for publication: April 3, 1995)

#### Abstract

By the surface condensation method (SCM), the organic contamination of aluminum surface can be detected. Correlation between the surface contamination and adhesion of photoresist micro pattern is studied. The as-sputtered aluminum surface has already the organic contamination, mainly, the outer area of wafer. The most of contamination can be easily eliminated by the oxygen plasma treatment. These results can be explained with the XPS and contact angle analysis data. The adhesion of photoresist micro pattern is improved by eliminating the organic contamination from the aluminum surface. The surface condensation method is available to study the surface contamination of solid surface.

(Received: January 30, 1995)

#### Adhesion of Micro Window Pattern Formed by Photolithography

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(Accepted for publication: June 19, 1995)

#### Abstract

Adhesion behavior of photoresist thin film in HF aqueous solution is studied. The photoresist films are fabricated as various micro window shapes by means of photoli-thography. The envelope shape of silicon-oxide substrate after the HF etching can be controlled by changing the window sizes.

(Received: May 26, 1995)

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#### Miscibility Phenomenon and Pressure Sensitive Adhesive Properties of Polybutadiene (PBD) and Pinene Blends Adhesives

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(Accepted for publication: Auguest 9, 1995)

#### Abstract

Miscibility and PSA properties of blends of various 1, 2-unit contents PBD and different type of pinenes were studied by DSC and laser transmission light intensity. The blends of PBD/ $\beta$ -pinene resin were immiscible, while PBD/ $\alpha$ -pinene resin blends were found to be miscible at various 1, 2-unit content of PBD. At the same time, PBD/ $\alpha$ pinene series had the LCST phase diagrams. Therefore, the chemical structures of  $\alpha$ -, and  $\beta$ -pinenes were also investigated by using <sup>13</sup>C NMR. In the miscible blends, miscibility window was also indicated at critical blend compositions.

And PSA properties of miscible  $\alpha$ -pinene blends were estimated by DMA and PSA tests (ball tack, probe tack, 180° peel srength, holding power). As the results, good

adhesive properties were found in PBD  $(30.1wt\%)/\alpha$ -pinene system with 50/50 blend ratio, and the reason was interpreted in terms of the dynamic mechanical behavior of the blend adhesive.

(Received: June 21, 1995)

#### Effect of Phase Structure on the Adhesion Properties of Modied Silicone/Epoxy Resin System

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(Accepted for publication: July 24, 1995)

#### Abstract

In order to understand the effect of phase structure on the adhesion properties of modified silicon/epoxy resin system, cured resins were prepared at four different amount of epoxy resins per one hundred parts of modified silicone, i.e, 25, 50, 75 and 100 parts of epoxy resins were mixed with 100 parts of modified silicone and cured at two different pre-curing temperatures, 25°C and 80°C. Totally, eight types of cured resins having different phase structure were prepared.

Adhesion properties, dynamic mechanical properties, fracture energies and morphologies of the cured resins were investigated. The fracture energies and T-peel bonding strengths show peak values at 50 parts of epoxy resin at  $25^{\circ}$ C pre-curing, however, at  $80^{\circ}$ C pre-curing, these two properties were enhanced on each part of epoxy resin and the peak positions were moved from at 50 parts to at 75 parts.

These results were explained by two phenomena. One is decrease in the diameter of dispersed epoxy phase, i.e, an increase in the interfacial area between matrix and dispersed phase. The other is an increasing in the deforming ability of cured resin under stress with increase in the Poisson's ratio by being uniform distribution of epoxy particles diameter.

(Received: June 29, 1995)

#### Invence of Miscibility between the Components in Acrylic Pressure-Sensitive Adhesives upon the Temperature and Rate-dependence of Their Peel Strength

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(Accepted for publication: July 17, 1995)

#### Abstract

Influence of miscibility between the components in acrylic pressure-sensitive adhesives upon their peel strength as a function of temperature and rate has been studied.

In case of miscible blend systems, incorporation of tackifier resins into the pressuresensitive adhesives resulted in the systematic modification of the peel strength. The master curves of peel strength for a series of the blends were obtained. Each master curve had a peak at some rate. Cohesive failure occurred in lower rate side of the peak, and stick-slip failure occurred in higher rate side of the peak. As the concentration of the tackifier became higher, the peak of the master curves shifted toward lower rate.

In case of immiscible blends where two phases existed in the system, however, incorporation of tackifier resins into the pressure-sensitive ahesives did not result in the significant modification of the peel strength. As the concentration of the tackifier became higher, the master curves of peel strength did not vary greatly along the rate axis. It is evident that peel strength mostly depends upon the viscoelastic properties of the matrix phase, and that a dispersed phase acts as a kind of filler.

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#### Morphology of Thermoplastic Polyimide-Epoxy Blend Matrix in CFRP

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(Accepted for publication: June 19, 1995)

#### Abstract

Thermoplastic polyimide (PI)-Epoxy (EP) blends system were employed as a matrix resin for carbon fiber (CF) reinforced plastics (CFRP). Micro-structure and dynamic mechanical properties were investigated for bulk and CFRP consisting of EP and 20 wt% PI blends. In CFRP matrix, low CF contents (20 wt%) and high temperature cure (200°C) yielded micro-structure similar to that in bulk resin. High CF contents (60 wt%) and/or low temperature cure (130°C) yielded unique structure, namely EP rich continuous phase localized arround CF. Irrespective of the curing conditions and CF contents. CF were in contact with EP rich phase in all the investigated fractured surface in CFRP. Molecular weight of added PI also affected for morphology of the bulk resin and CFRP matrix. The structure in CFRP is assumed to be controlled by competition of phase separation, wetting process in CF surface, and crosslink reaction.

(Received: May 31, 1995)

#### Physical Properties of Elastic Gel/Epoxy Resin Composites

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(Accepted for publication: August 8, 1995)

#### Abstract

The epoxy resin was blended with crosslinked elastic particles for the improvement of the mechanical properties of epoxy-composites. Elastic gels were synthesized by the emulsion copolymerization of *n*-butylacrylate, divinylbenzene and methacrylic acid in various molar ratio. The epoxy-composites were prepared by the method of coprecipitation from the emulsion mixtures of epoxy resin and elastic gels with various crosslinking degrees and concentrations of carboxyl groups. And epoxy-composites were cured with a tertiary amine compound as a hardener.

A little influence of the blended gels on the bending properties of cured epoxy-composites was observed. On the other hand, the adhesive properties of Fe/Fe joints were improved by blending with gels to a epoxy resin and were dependent on the crosslinking degrees and the concentration of carboxyl groups. The results of dynamic mechanical analysis and photographs of scanning electron microscope of fracture surfaces showed that the cured epoxy-composites had the microphase separated structure and the reaction phase of elastic gels with epoxy resin.

(Received: June 5, 1995)

#### Efiect of Silane Coupling Agent on the Adhesion of Aluminium Film and PET Film

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(Accepted for publication: July 17, 1995)

#### Abstract

Polyethyleneterephthalate (PET) film was treated with N-(2-Aminoethyl)- $\gamma$ -aminopropyltrimethoxysilane (ATS) and  $\gamma$ -Glycidyloxypropyltrimethoxysilane (ETS), and aluminium (Al) was deposited onto the treated PET.

A glass-like coating film was formed by treating with ATS and ETS on the PET film. Hydrophile of the treated PET film was enhanced by the orientation of unreacted functional groups of ATS and ETS. The peel strength of the adhesive interface at the Al thin film between the treated PET film was enhanced to 3.5 times compared with the case of the untreated PET film. The peel was caused at the interface of the coating film between the PET film and the adhesion of the interface at the Al thin film between the coating film was forced sufficiently. It was found that the peel strength was dependent on compatibility of the PET film with the coating film, that was anchor effect of ATS and ETS onto the PET film. This fact was confirmed by SEM photographs.

(Received: May 29, 1995)

#### Effect of Silicon Introduction into Aromatic Polyimide

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(Accepted for publication: June 7, 1995)

#### Abstract

Recently, many kinds of polyimides have been developing and applying in electronic industries. In this work, various silicon containing polyimides derived from 3,3', 4,4'-benzophenonetetracarboxylic dianhydride (BTDA), 4,4'-diaminidiphenyl ether (DADE) and 6 kinds of silicon compounds were synthesized and characterized. It was proved that the introduction of disiloxane derivatives with aromatic end group was remarkably effective for thermal stability. Among them, the copolyimide derived from disiloxane containing dianhydride exhibited lower glass transition temperature. The adherence of copolyimides on silicon wafer was maintained even after three days in high humidity condition. In addition, incorporation of silicon component into aromatic polyimide led to lower moisture up take.

(Received: May 24, 1995)

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