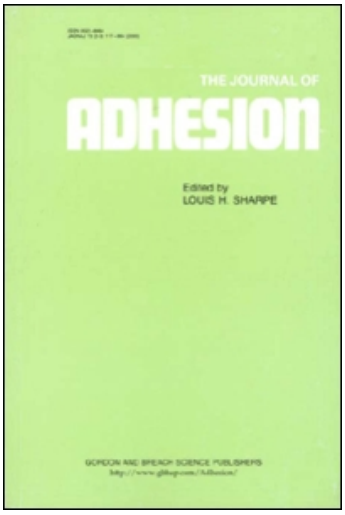

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Analysis of Adhesion Property of Photoresist Micro Pattern by Scanning Method of Micro Tip with Atomic Force Microscope

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(Accepted for publication : March 16, 1999)

By using an atomic force microscope (AFM), we discuss validity of analysis method of adhesion and cohesion property of micro photoresist pattern. Dot shaped photoresist patterns, 0.60 μm in square and 1.01 μm in height were formed onto a flat substrate by photolithography. Load for collapse of photoresist pattern and its deflection are measured by applying load with an AFM micro tip. By combining with stress and deformation analysis by finite element method, internal stress for collapse and Young's modulus of the photoresist pattern can be obtained. These value increase with heating temperature of photoresist pattern. By the thermal analysis of weight loss, hardness and refractive index, we confirmed the validity of this method. This method can be applied to adhesion and cohesion analysis of micro condensed matter adhered on a flat substrate.

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Crack Growth Evaluation on Cleavage Adhesive Joints Using Surface Wave

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The crack growth behavior on the cleavage adhesive joints with various adhesive thicknesses was investigated by the echo characteristic of surface wave emitted toward the adhesive layer under the tensile load. The cross section of adhesive layer was $25 \times 25\,mm^2$. The echo height reflected from the edge of the adhesive layer and the round-trip propagation time from the surface wave probe to the adhesive layer were measured for evaluating the crack growth behavior. The crack was generated along the adhesive interface when the ratio of the round-trip propagation time suddenly increased. After that, the crack rapidly grew. In the case where the distance between the surface wave probe and the adhesive layer was 10 mm, the crack of the adhesive layer was detected when the crack length was more than 0.2 mm or the ratio of the echo height reflected from the edge of adhesive layer became about 60%. Moreover, the crack growth behavior was evaluated for measuring the ratio of the round-trip propagation time or the ratio of the echo height. The load of crack generation detected by the change ratio of the round-trip propagation time agreed well with that of the echo height. The load of crack generation and breaking load on the adhesive layer decreased as the adhesive layer thickness was thicker.

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Interdiffusion in Poly(isobutylene)/ Poly(butylacrylate) Interfaces

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(Accepted for publication : May 11, 1999)

This study investigates the interdiffusion of a poly(isobutylene) into a poly(butylacrylate) layer. The presence of interdiffusion at the adhesive/polymer substrate interfaces is essential for the development of pressure sensitive adhesive tapes. Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (FTIR - ATR) studies have been used to characterize the interdiffusion behavior. The interdiffusion and reptation behavior was studied at various time intervals by measuring the changes in absorbance bands of selected characteristic peaks in each polymer. The interdiffusion time of poly (isobutylene)/poly(butylacrylate) depended on the molecular weight of poly(isobutylene) ($t \sim M^{1.2 \sim 1.5}$). Still more, different behavior of interdiffusion between above and below the critical molecular weight (Mc) was observed.

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Collapse Behavior of Organic Dot-Pattern Analyzed by the Tip Indentation Method

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By applying a certain load directly with a microcantilever tip, a resist micropattern adhering to a substrate can be collapsed accompanying a residue formation. By combining with the finite element method for the internal stress analysis, it is clear that the crack formed in the resist micropattern affects the stress distribution and the residue shape. By the tip indentation method, the collapse and destruction mechanism of the resist micropattern can be analyzed.

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Mode II Interlaminar Fracture Toughness of Ionomer Interleaved Carbon Fiber/Epoxy Laminates

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The effect of ionomer thickness on mode II interlaminar fracture toughness was investigated for ionomer-interleaved CF/epoxy laminates. Laminates were fabricated with Toho UT500/111 prepregs. Ethylene based ionomer, which forms the toughened interphase at the ionomer/base lamina interfaces, was used as an interleaf material. Thickness of ionomer film was selected as 25, 100 and 200 μm. Mode II fracture toughness tests were carried out using end notched flexure specimens. Fracture toughness values were much improved by interleaving the ionomer films. The fracture toughness values increased linearly with increasing the ionomer thickness. This thickness effect of the ionomer interleaf differs from that under mode I loading where the presence of ionomer is more effective than the increase in its thickness. Microscopic observation revealed that the main crack path was inside the toughened interphase and at its interfaces. Plastic deformation of the ionomer expanded through the whole ionomer region in the thickness direction. Difference in the thickness effect on fracture toughness between under mode I and II loadings were discussed from the view point of the size of the plastically deformed region.

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Effect of Amino Silane–Treatment on the Mechanical Properties of Glass Beads-Filled Poly(Vinyl Chloride)

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(Accepted for publication : May 4, 1999)

The effect of a surface treatment of glass beads with a silane coupling agent on a yield stress was investigated in the beads-filled poly(vinyl chloride) as typical ductile polymer matrix. γ-Aminopropyl methyldiethoxysilane was used as silane. The amount of silane necessary for creating monolayer coverage was calculated. The beads were treated with the silane in the range from 0.5 to 10 times of the amount necessary for monolayer coverage in water by some different conditions. The treated beads were washed with methanol to remove the unfixed silanes on the surfaces. The fixing ratio of the silane was determined by a carbon analysis of the treated beads both for as treated and methanol washed. A comparison between the yield stresses of the resins filled with both as treated and methanol washed beads was made. As a result, the yield stress increased with increasing the amount of silane treated in the range below that for monolayer coverage. However, a further improvement on the yield stress was not obtained in the range above the amount. The unfixed silanes on the bead surface depressed the improvement on the yield stress.

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Toughening of Silica Filled Acrylic Composite

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In order to improve the toughness of silica filled acrylic composite, two typical impact modifiers, multi-layer core/shell particle and polymerisable elastomer, were examined. The IZOD impact strength of the composite was not affected by the core/shell particle, while significant improvement was observed in the elastomer modification. The difference was detected at the fracture surface by scanning electron microscope (SEM) observation and elemental analysis which was given by energy-dispersed X-ray micro-analyzer equipped with the SEM (SEM – EDX). The result suggested that the curable elastomer transferred the destruction phase from silica matrix interface to inside of the matrix. The major contribution of the elastomer for this transfer was thought to be adhesion promotion at silica/PMMA interface, while the core/shell particle toughened the matrix only. Consequently, the elastomer modified acrylic composite was toughened farther by core/shell particle very efficiently, because the destruction occurred in the toughened matrix phase. The concept for toughening in silica filled acrylic composite was provided through this work, and the effective toughening method was proposed.

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Thermal Stress of Scarf Adhesive Joint Under Uniform Temperature Changes

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This study dealt with thermal stresses and delamination growth in scarf joints under uniform temperature change by photoelastic measurement and a two-dimensional finite element analysis. The adherends were aluminum plates and an adhesive layer was modeled and manufactured from an epoxide resin plate. Adherends and epoxide resin plate were bonded using a heat setting and one component type adhesive. The adhesive was cured at high temperature and cooled to room temperature. The thermal stress was then generated in the scarf joint during temperature change and measured by the photoelastic experiments. After that the scarf joints were cooled stepwise, delamination growth from the edge of the interface was measured. It was confirmed that the delamination initiated from the edge of the interface was not the obtuse angle side, but the acute angle side. When the scarf angle was 90 degree, *i.e.*, in adhesive but joints, the resistance against the delamination was minimum. The thermal stresses in the scarf joints with thin adhesive layer were also analyzed. It was expected that the thermal strength increased with a decrease of an adhesive layer thickness. The stress singularity near the edge of the interface was calculated from the stress distributions in the joints with different scarf angles. As a result, the stress singularity in the scarf joints under thermal loads was quite different with that under mechanical tensile loads.

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Mechanical Properties of Polyimides Microparticles/Epoxy Resin Blends

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(Accepted for publication : August 31, 1999)

In order to improve the fracture toughness of epoxy resin (Ep), three kinds of polyimides microparticles (PI) were incorporated with Ep.

Material properties of the blends were estimated by flexural and fracture tests and WAXD (*etc.*) and the toughening mechanism was studied by SEM micrographs taken after the toughness tests.

The mechanical properties of PI-A ("AURUM")/Ep depended on the mixture ratio of PI-A.

And there were characteristic differences in the flexural properties and fracture toughness of three kinds of PI/Ep. Namely, the blend which is composed of PI with the lower crystallinity showed the higher value of flexural strength. And the blends which are composed of PI with the higher crystallinity showed the higher values of fracture toughness. These results indicated that the flexural properties and fracture toughness of PI/Ep had relations to the crystallinity. Also the fracture toughness of the PI/Ep depended on the feature of PI.

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Synthesis and Bonding Properties of Epoxy Resin Containing Mesogenic Group

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Liquid crystalline epoxy resin with mesogenic group was synthesized and its adhesive bonding properties were compared with that of bisphenol-A type epoxy resin. Bonding strength of the former resin system showed higher value than that of the latter resin system. It was suggested that the high bonding strength of the liquid crystalline epoxy resin system was due to the large creep deformation of this resin system along the stress direction. Bonding strength of the cured systems has a maximum peak with the progress of curing, because of the increase in the internal stress occurred with the curing shrinkage of epoxy resins.

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Control of Catalytic Activity in Hydrosilylation by Using Acetylene Derivatives

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The catalytic activity of H_2PtCl_6 in the hydrosilylation was evaluated in the presence of acetylene derivatives to clarify the relationship between the acetylene structures and their controllability of the hydrosilylation process. From the study on the hydrosilylation reaction of trimethylvinylsilane with triethylsilane at various temperatures, it was found that the catalytic activity was dependent upon the substituents (electronic and steric factor) on the added acetylene derivatives. In cases of diacetylenes, the catalytic activity was suppressed remarkably compared to the cases of monoacetylenes. The curing reaction of silicon resin was also carried out in the presence of acetylene derivatives to observe the same tendency to the model reaction.

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Infrared Microspectroscopic Mapping of Residual NCO Group in Wood-Based Materials

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Infrared microspectroscopic spectra of wood-based materials including an isocyanate adhesive were recorded by diffuse reflectance method. We investigated the relationship between the NCO concentration and the absorbance of asymmetric stretching vibrational band (2270 cm^{-1}) of the functional group in the MDF boards. Automation and refinement of the two-dimensional infrared measurements were carried out, and the distributions of residual NCO group were obtained in several kinds of MDF boards and plywood. Furthermore, with a view to visualizing the distributions, the maps of NCO group were displayed by the color corresponding to its concentration.

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