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### **Durability Tests of Various Adhesives for 30 Years in Interior Condition**

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### Durability Tests of Various Adhesives for 30 Years in Interior Condition

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(Accepted for publication: September 27, 1994)

### Abstract

This experiment aimed to determine the durability of various adhesives for 30 years. Wood test pieces bonded with several kinds of adhesives were exposed to indoor environment at Urawa Laboratory. This test carried out since 1964 and still gives an experiment. The test term was set up 7 day, 1 year, 5 years, 10 years, 20 years and 30 years. For this experiment, 13 kinds of adhesives (6 kinds of PVAc emulsions, 3 kinds of urea resins, phenolic resin, epoxy resin, resorcinol resin and animal glue) were investigated. The summary of the

result is: from 20 years, the compression shear strengths by 3 kinds of urea resins lowered rapidly, but the other 10 kinds of adhesives were not recognized the lowering of strengths.

(Received: July 20, 1994)

### Wetting Failure of Photoresist on Organic Spin-On-Glass Film

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(Accepted for publication: August 24, 1994)

### Abstract

The wetting failure of photoresist is a major problem for the multilayer-resist lithography in large scale integrated circuit (LSI) fabrication. The wetting failure occurs when a micro particle exists on the spin-on-glass (SOG) surface. The concentration of (CH<sub>3</sub>—) group decreases as the baking temperature increases. No wetting failure occurs under the baking temperature of 175°C. This phenomenon is discussed by using surface free energy of each surface.

(Received: July 14, 1994)

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### **Curing Behavior of Molding Compound from Liquefied Wood and Properties of Cured Liquefied Wood**

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(Accepted for publication: September 30, 1994)

#### **Abstract**

Liquefaction of wood in the presence of phenol by using sulfuric acid as catalyst was carried out. Molding compounds for injection molding were prepared by hot roll-kneading from the liquefied wood, curing agent, lubricant, curing coagent, and filler. The influences of lubricants, curing coagent, and filler on flowability and curing behavior of molding compound were examined by Disk Cure Test, Curelastometer, and Brabender Plastograph. Properties of test pieces prepared by transfer molding from the molding compounds were also investigated. The results were as follows: (1) Content of acetone extraction from cured liquefied wood by Soxhlet's extractor decreased with an increase of the content of hexamethylenetetramine (hexamine) as curing agent and became almost same when the content of hexamine was above 15 phr. (2) Flowability of the molding compound was best, when the content of zinc stearate as lubricant and calcium hydroxide as curing coagent were 4 phr and 1 phr, respectively. (3) Minimum torque at kneading of those was almost same as that of phenolic resin for commercial injection grade, when the content of zinc stearate, calcium hydroxide, and wood flour were 4 phr, 1 phr, and 40 phr respectively. (4) Flexural strength of test pieces was more than 600 kgf/cm<sup>2</sup> which was standard value according to JIS K 6915 for general molding grade of phenolic resin.

(Received: September 7, 1994)

### **Adhesive Properties of Resin Materials for Forming Printed Circuits by Electroless Plating**

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(Accepted for publication: October 19, 1994)

#### **Abstract**

Resin materials are investigated to achieve strong adhesion between an insulated iron board and circuits photochemically formed by an additive process. Nitrile rubber mixed with phenolic resin has excellent properties with regard to peeling strength, heat and chemical resistances. It has a high peeling strength of more than 3 kg/cm, when circuits are formed under optimum conditions. As for component materials, nitrile rubber with high nitrile content is suitable because of its high peeling strength, and phenolic resin of novolak type made from alkyl phenol source is preferred because of its high miscibility with nitrile rubber.

The resin surface is chemically roughened with chromic etchant and the relationship between the surface conditions and the adhesive strength is studied. The adhesion of resin materials containing resol type phenolic resins arises from the mechanical anchoring effect which is brought about when solid masses of phenolic resin are wrapped with a copper foil plated on the roughened adhesive surface.

(Received: August 5, 1994)

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**Relation between Phase Structure and Interface Structure of Segmented Polyurethanes**

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(Accepted for publication: October 5, 1994)

**Abstract**

Polyester segmented polyurethanes that had various hard-segment (HS) content were synthesized and relation between phase structure and interface structure of them was investigated. Soft-segment (SS) units mainly existed in interface for various HS content. However, interface structure varied uncontinuously with formation of phase separation structure in the bulk, and it was clear that phase separation structure in the bulk affected interface structure. Hard domains (HD) formed by condensation of HS units behaved like hydrophobic groups in spite of existence of many urethane groups. Adhesion properties of SPU were also studied.

(Received: July 18, 1994)

**Adhesion of Photoresist Macro Pattern Depended on Roughness of Aluminum Surface**

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(Accepted for publication: October 13, 1994)

**Abstract**

In Large Scale Integrated Circuit (LSI) production, the adhesion control of a photoresist micro pattern to a substrate has become important. Dependency of resist adhesion on surface roughness of aluminum is studied.

Adhesion strength indicates a maximum value at average roughness  $R_a = 5.5$  nm among three kinds of surface roughness. This result can be explained by two factors: (i) stress concentration at the interface region, (ii) increase of the contact area between resist and substrate surface, as the surface roughness increases.

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**Relation between Phase Structure and Interface Structure of Segmented Polyurethanes Having Functional Groups**

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(Accepted for publication: September 12, 1994)

**Abstract**

Segmented polyurethanes having functional groups in hard-segment (HS) or soft-segment (SS) were synthesized and the effect of the number and position of functional groups on phase and interface structure was investigated.

Phase separation structure of SPU having functional groups in HS (H-SPU) disappeared with increasing of functional groups. While, in the case of SPU having functional groups in SS (S-SPU), phase separation structure was preserved in spite of increasing of functional groups. It was caused by the side chains of functional groups that obstructed condensation of HS.

Orientation of functional groups in interface of S-SPU was higher than that of H-SPU. It was caused by the difference of mobility between HS and SS. So, S-SPU had better adhesion to polar surface than H-SPU.

(Received: August 4, 1994)

**Photosensitizing Resin Materials for Forming Printed Circuits**

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(Accepted for publication: October 19, 1994)

**Abstract**

Resin materials which have a photosensitive effect are investigated to form printed circuits by photoadditive process. Three component resins composed of nitrile rubber, phenolic resin, and epoxy resin mixed with zinc oxide sensitize the photolysis of disilver glutamate by roughening their surface with liquid honing. It results in the sufficient deposition of silver catalyst and the excellent plating even in the through-holes on which only a small amount of UV light are exposed.

Addition of zinc oxide to the resin of optimum composition achieves a strong adhesion over 4.0 kgf/cm between the substrate and plated circuits, excellent heat resistance and durability. The adhesion and heat resistance are markedly improved by heating after the electroless plating. This is caused by migration of silver catalyst into the resin which exists in the interface between the substrate and plated metals. It is also supported by the result that the thinner photosensitive material gives the higher peeling strength and heat resistance. Concerning the roughening condition, the finer abrasives and higher roughening pressure give the higher peeling strength.

(Received: September 13, 1994)

**Curing Behavior of Epoxy Compound Having Hydroxymethyl Group and Acid Anhydride**

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(Accepted for publication: October 24, 1994)

**Abstract**

Curing behavior of epoxy compound having hydroxymethyl group cured with an acid anhydride was studied. An epoxy compound having hydroxymethyl group (EOCDA) was mixed with commercial epoxy resin (DGEBA) in various ratios. The mixed epoxy resins were cured with methyl tetrahydrophthalic anhydride (Me-THPA) as a hardener and 2,4,6-tris(dimethylaminomethyl) phenol (DMP-30) as a catalyst. Kinetic parameters of cure reaction were obtained by using differential scanning calorimetry (DSC). It was found that the higher the concentration of EOCDA, the higher the rate constant ( $k$ ) and the smaller the activation energy ( $E_a$ ) and the log of the frequency factor ( $\ln A$ ) were. Reaction mechanism of epoxy resin and acid anhydride with tertiary amine at 80°C was studied by using  $^1\text{H-NMR}$  and FT-IR. The concentration of epoxide group, acid anhydride and hydroxymethyl group decreased with the reaction time. Glass transition temperature ( $T_g$ ) of cured epoxy resins was measured by using dynamic mechanical analyzer (DMA).  $T_g$  of cured resin was highest when the epoxy resin was mixed with excess of acid anhydride. From these results it was considered that the following reactions occurred in this system: (1) hydroxymethyl group accelerates the reaction of epoxide group and acid anhydride, where hydroxy methyl group acts as a catalyst. (2) hydroxymethyl group reacts with epoxide group and acid anhydride.

(Received: September 9, 1994)

**Spreading of Water Drop on Geometrical Rough Surface Formed by Photolithography**

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(Accepted for publication: January 18, 1995)

**Abstract**

Dependency of spreading velocity of water drop on roughness is studied. The roughness of surface is controlled by changing the aspect ratio of photoresist micro patterns formed by photolithography. The spreading speed of water drop decreases as the aspect ratio increases. Results obtained in this experiment appear to confirm the Newman's spreading model.

(Received: December 16, 1994)