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## **The Journal of Adhesion**

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713453635>

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

**To cite this Article** (1999) 'Contents Lists and Abstracts from the Journal of the Adhesion Society of Japan', *The Journal of Adhesion*, 70: 3, 321 – 332

**To link to this Article:** DOI: 10.1080/00218469908009562

**URL:** <http://dx.doi.org/10.1080/00218469908009562>

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

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# An ATR-FTIR Spectroscopic Study on the Chemical Interaction of a New Monomer Bearing Carboxylic Moiety with Dentin Apatite and Collagen

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*(Received 4 February 1998; In final form 10 March 1998)*

To clarify the bonding mechanism of dental adhesive to ground dentin, the chemical interaction of 4-acryloxyethyltrimellite acid (4-AET) with dentin apatite and collagen was investigated, using fourier transform infrared spectroscopy with attenuated total reflectance (ATR-FTIR) and scanning electron microscopy (SEM). An experimental 4-AET/HEMA (40/60. wt%) primer was prepared. A block of bovine dentin was treated with NaOCl (Apatite group) or 6N HCl (Collagen group), and also treated with the primer. ATR-FTIR measurements of the primer and the surface of the dentin (Apatite and Collagen groups) treated with or without the primer were carried out. The SEM micrographs revealed that apatite crystal was exposed (Apatite group) or the apatite was selectively removed (Collagen group) from the surface of dentin. The results of ATR and its difference spectra of Apatite group suggested that absorbance of phosphoric ion ( $1020\text{ cm}^{-1}$ ,  $-\text{PO}_4^{3-}$ ) of hydroxyapatite was significantly reduced and the Ca-carboxylate ( $1557$  and  $1413\text{ cm}^{-1}$ ) was formed. The results of ATR and the difference spectra of Collagen group indicated that amide I band was shifted and a new peak ( $1670\text{ cm}^{-1}$ ) was grown. It was suggested that an ionized divalence carboxylic group in 4 AET molecule can combine with  $\text{Ca}^{2+}$  cation of hydroxyapatite,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , to form chelate bond (or Ca salt) or an inter hydrogen bond between carboxyl group of 4-AET and amide bond of collagen was formed at the interface.

## Effect of Strain Rates on Interfacial Adhesive Shear Stress of GF/PP Injection Molding Composite

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*(Received 23 February 1998; In final form 25 March 1998)*

To analyze strength and deformation of structures made of composite material, which subjected to high speed loading, the effect of strain rate on the mechanical properties of the material should be clarified. In the present study, the interfacial adhesive shear stresses in the composite material was evaluated from a modified viscoelastic model and stress relaxation tests which conducted under several strain rates. From these studies the elastic modulus of the composite was found to be increase with strain rate. Especially increase in the elastic modulus is bigger than the value which could obtain it from the rule of mixture. This increase was considered to be caused by interfacial adhesive shear stress increases. The increase of elastic modulus of resin and the decrease of Poisson's ratio of composite were considered to be responsible for the increase of interfacial adhesive shear stress of composite.

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## Vibrational Spectroscopic Study of Polyurethane Films Prepared from Crude MDI and Polyethylene Glycol

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*(Received 20 February 1998; In final form 20 April 1998)*

Polyurethane (PU) films with the isocyanate index range from 40 to 220 were prepared from crude diphenylmethanediisocyanate (MDI) and polyethylene glycol (PEG) under air. Chemical behavior of unreacted isocyanate group in the PU films was studied by infrared (ATR method) and Raman spectroscopy. The spectral data indicated that the PU mainly contains the urethane bonds some of which receive intermolecular hydrogen bond. It was, however, revealed that urea bond arising from reaction of isocyanate group and water impurity is also included in the product. Moreover, the solid PU underwent thermally induced self-condensation of unreacted isocyanates to cause formation of carbodiimide at 100°C, whereas the isocyanates reacted with urethane or urea bonds to form allophanate or biuret bonds below room temperature.

# Influence of Miscibility Between the Base Polymer and the Tackifier in Acrylic Pressure-Sensitive Adhesives Upon the Rate-Dependence of their Rolling Friction Coefficient

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*(Received 19 March 1998; In final form 20 April 1998)*

In this study, the influence of miscibility between the components in acrylic pressure-sensitive adhesives upon the rate-dependence of their rolling friction coefficient has been clarified.

In case of miscible blend systems, incorporation of a tackifier resins into the pressure-sensitive adhesives resulted in the systematic modification of the rolling friction coefficient. Each rolling friction coefficient *vs.* velocity (*f vs. log v*) curve had two peaks or shoulder at some rates. Cohesive failure occurred in lower rate side of the lower peak, and interfacial failure or stick-slip occurred in higher rate side of the higher peak, and complicated mixed failure occurred between two peaks. As the concentration of the tackifier became higher, the peaks of the curves shifted toward lower rate.

In case of immiscible blends where two phases existed in the system, as tackifier content became higher, the curves did not vary greatly along the rate axis, but the absolute value of rolling friction coefficient decreased. It is evident that rolling friction coefficient mostly depends upon the viscoelastic properties of the matrix phase, and that a dispersed phase acts as a kind of filler.



## Adhesion of 6-Nylon Resin and Phosphor Bronze Plates Treated with Triazine Thiols by Injection-Molding\*

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*(Received 25 February 1998; In final form 1 April 1998)*

6-Nylon resin (PA6) adhered tightly to phosphor bronze (PB) plates treated with an aqueous solution of 1,3,5-triazine-2,4-dithiol-6-sodium mercaptide (TTN), during injection-molding. The adherends between PA6 and PB plates treated with TTN had highshear strength of 5 MPa or more. The mechanism of adhesion was explained to be due to interfacial bonds formed by the nucleophilic substitution of amino end group in PA6 with 1,3,5-triazine-2,4,6-tri-copper [Cu (I)] mercaptide [TT-3Cu (I)] formed on the PB plates. The optimum TTN concentration and treating time for adhesion were in the range of 0.5–1 mmol/l and 10 to 60 sec. at 50–70°C. TT-3Cu (I) film on the surface changed to the structure of high density during TTN-treatment when PB plates were immersed in TTN aqueous solution for a period longer than 60 sec. Also in higher TTN concentration than 1 mmol/l, TT-3Cu (I) film of high density was formed on the surface of PB plates. TT-3Cu (I) film of high density on the plates inhibited nucleophilic substitution with amino end group in PA6 because of steric hindrance. As a result, PA6 did not adhere to these treated plates.

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\* Studies on Direct Adhesion between Plastics and Metal Using Triazine Thiols II.

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## Three-Dimensional Finite Element Analysis of Stress Response in Adhesive Butt Joints Subjected to Impact Bending Moments

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The stress wave propagation and the stress distribution in adhesive butt joints of similar T-shape adherends subjected to impact bending moments are analyzed using three-dimensional finite-element method (FEM). An impact load is applied to a joint by dropping a weight. The FEM code employed is DYNA3D. The effects of Young's modulus of the adherends, the adhesive thickness and geometry of T-shape adherends on the stress wave propagation at the interfaces are examined. It is found that the maximum of the maximum principal stress  $\sigma_1$  is caused at the interfaces. It is also seen that the maximum value of the maximum principal stress  $\sigma_1$  at the interfaces increases as Young's modulus of the adherends increases. In the special case where web length of T-shape adherends equals to the flange length, it is seen that the maximum value of the maximum principal stress  $\sigma_1$  at the interfaces increases as Young's modulus of the adherends decreases. The maximum value of  $\sigma_1$  at the interfaces increases as the adherend thickness decreases. In addition, experiments were carried out to measure the strain response of adhesive T-shape butt joints using strain gauges. Fairly good agreement is seen between the analytical and the experimental results.

# A Three-Dimensional Thermal Stress FEM Analysis of Thin Wall Box-Shape Bolted Flanged Joints with Silicone Sealant

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*(Received 13 May 1998; In final form 22 September 1998)*

Thin-wall bolted flanged joints with gaskets or sealants have been used in mechanical structures such as combustion engine and transmission in automobile vehicles from the standpoints in order to lighten the weight and reduce the energy loss. The thin-wall bolted flanged joints with gaskets or sealants in oil pan structures are sometimes subjected to heat conduction as well as internal pressure. Thus, it is necessary to examine the thermal stress of thin-wall bolted flanged joints with sealant. This paper deals with the thermal stress analysis of the joints with silicone sealants by using finite-element method. In addition, the effects of the ratio of the stiffness and thermal linear expansion coefficient of the flanges to those of the sealant and the flange thickness are examined on the interface stress distributions in the joints. Experiments were carried out to measure the thermal strain occurred near the interface of the flanges using strain gauges. Leakage tests were also carried out at room temperature. Fairly good agreements are seen between the numerical and the experimental results concerning the thermal strain and the internal pressure when leakage occurred. It was found that the sealing performance was more improved as the stiffness of the flanges and the thermal linear expansion coefficient of the sealant were increased. Thus, it can be concluded that thermal linear expansion coefficient of sealant in joints must be increased in order to prevent the failure of sealant under elevated temperature.

# Elastoplastic FEM Stress Analysis and Strength Evaluation of Adhesive Tapered Lap Joints of Hollow Shafts Subjected to Tensile Loads

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(Received 18 May 1998; In final form 9 July 1998)

Tensile strength of adhesively bonded tapered lap joints of hollow shafts is investigated in this study. In tapered lap joint, bonded area increases and assembly and adjusting of the shafts are easy because of its self-aligning effect. In the experiments, at first, the elastoplastic stress-strain behavior of the adhesive was measured by uni-axial tensile test of the bulk specimen and then the tensile strengths of the joints with various taper angle were tested. In the analysis, the stress distributions at the interface between the shafts and the adhesive are calculated by the finite element method taking the elastoplastic behaviors of the adhesive and the shafts into consideration. A tensile strength prediction method is proposed based on the numerically obtained stress distributions and its features are discussed by comparing the measured strengths with the predicted ones.