

HUMIDITY EFFECTS ON MECHANICAL PROPERTIES OF KAPTON® E

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Abstract

The effect of water on the mechanical properties related to energy dissipation abilities of Kapton® E are studied. Various moisture contents induced using several humidity chambers ranging from 8% to 98% relative humidity. Water uptake values of 0.2 wt% to 1.4 wt. % were observed. The magnitude of the α transition and the low-temperature component of the β transition increased with increased humidity. Room temperature tensile modulus values were not affected by variations in humidity.

RELEASABLE IONICALLY SELF-ASSEMBLED MONOLAYERS

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Abstract

Ionic self-assembled monolayers, ISAMs, are nanostructured, layered films created by the cyclic dipping of a charged substrate into oppositely charged polyelectrolyte solutions. ISAMs have remarkable properties including control of the film deposition in two dimensions and the formation of uniform, pinhole free film structures using simple laboratory procedures. The release of an ISAM from a substrate was approached by theorizing two chemical mechanisms: reversible and irreversible. A reversible release mechanism based on the processes used in Immobilized Metal Affinity Chromatography was attempted. ISAMs built on chelating agents were incubated with an EDTA and an EDTA/NaCl solution. Although release of the ISAM from the microtiter plate was not observed, ISAMs were created on substrates functionalized with metal chelating surfaces. The produced ISAMs, created using both PAH/PS-119 and poly-L-histidine/PS-119, demonstrated reproducible results and constant bilayer thickness. These results, to the best of our knowledge, have not been previously discussed in the literature. Also shown is that by varying the ionic strength of the base polyelectrolytes, the bilayer thickness of ISAMs created on a metal chelator may be changed. Other release mechanisms must be investigated to obtain the release of an ISAM from a substrate.

SYNTHESIS AND CHARACTERIZATION OF NORBORNENE AND MALEIC
ANHYDRIDE ALTERNATING COPOLYMERS

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Abstract

A great deal of research is currently underway to develop new photoresist materials for 193 nm lithography using ArF-eximer laser technology, which would allow 0.18 and 0.13 micron etching.¹ Current lithographic techniques allow sub-0.5 micron features using 248 nm lithography and KrF eximer lasers, but current photoresist materials absorb strongly at 193 nm.² An excellent alternative is copolymers of cyclic olefins, which are both transparent and have a high C/H ratio that gives high etch resistance.³ This paper describes work performed to develop a better understanding of one of the most promising photoresists, poly(norbornene-alt-maleic anhydride).

ADHESIVE BONDING AND DURABILITY OF PLASMA SPRAYED ALUMINUM ADHERENDS

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Abstract

The durability of adhesively bonded plasma sprayed adherends has been examined by studying directly bonded plasma sprayed adherends. Aluminum alloy was plasma sprayed with epoxy. The aluminum plasma sprayed adherends were bonded in a wedge test configuration (fig. 1) and the durability was determined by measuring the crack growth. The plasma sprayed adherends were bonded either with a commercial room temperature curing epoxy, by coating the plasma sprayed epoxy with an amine-curing agent and heating above T_g , or by direct bonding of the plasma sprayed adherends. The strain energy release rate (SERR) was evaluated from the crack length data. The adherends were tested in three different environments including: 1) air at room temperature 2) water immersion at room temperature 3) boiling water immersion. The durability tests were carried out for 48 hrs to ensure that that crack had reached an arrest length. P2 etched aluminum adherends were bonded with the commercial epoxy at room temperature and were subjected to the same environmental tests as described above. This testing was done as a standard for comparison. Surface analysis using x-ray photoelectron spectroscopy (XPS) was used to determine the mode of failure for each tested adherend. The results of the durability test indicated cohesive failure for the P2 etched aluminum for both tests at room temperature in air and water immersion. The same adherend in boiling water showed adhesive failure. The adherends that were plasma sprayed and bonded with the commercial epoxy failed cohesively within the plasma sprayed epoxy for testing in all three environments. In testing of the plasma sprayed epoxy with the curing agent, the bond failed cohesively for measurements in both room temperature air and in water. When immersed in boiling water, the initial growth occurred via cohesive failure, then changed to adhesive failure before reaching the arrest crack length. The directly bonded plasma sprayed samples showed cohesive failure in the room temperature test in air. In both of the water tests, the directly bonded plasma sprayed specimens started with cohesive failure and changed to adhesive failure during prolonged exposure in the testing environment.

INFLUENCE OF STRUCTURAL CONSTRAINTS ON THE CRYSTALLIZATION AND
MELTING BEHAVIOR OF POLY(ARYLENE ETHER ETHER KETONE)

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Abstract

The secondary crystallization behavior and the glass transition temperature of PEEK are studied as a function of crystallization temperature and time by differential scanning calorimetry. Investigations of the low endotherm transition temperature and latent heat of fusion as a function of crystallization time and temperature lead to the following conclusions. First, the melting temperature of secondary crystals increases linearly with the logarithm of secondary crystallization time at a rate, $B(T)$, increasing linearly with decreasing temperature. Second, the Avrami exponent, which characterizes the initial stage of secondary crystallization, is independent of crystallization temperature below 310C and equal to 1/2. Studies of the evolution of the glass transition after secondary crystallization indicate the calorimetric glass transition temperature increases linearly with the logarithm of time at a rate, $b(T)$, increasing with decreasing temperature. The observation that $B(T)$ is proportional to $b(T)$ allows us to suggest that the evolution of the glass transition and low endotherm melting temperatures with crystallization time arises from the decrease in molar conformational entropy of the remaining amorphous fraction as the result of secondary crystallization.

FIBER REINFORCED POLYMER (FRP) BRIDGE DESIGN: A CONNECTION SOLUTION FOR PREFABRICATED CELLULAR BRIDGE DECKS AND STRINGERS

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Abstract

In this study, a connection system between a fiber-reinforced polymer (FRP) cellular bridge deck and winged-box stringer was designed and tested. The deck beams used were pultruded Strongwell Extren™ Series 500/525 glass/polyester square tubes. The stringer used was a pultruded glass/polyester winged-box stringer, also provided by Strongwell Corporation. The connection system consisted of steel "shear" plates bolted to angle iron sections, which were in turn bolted through the web of the stringer. These shear plates were positioned to inhibit slip between the stringer and the deck during bending. Through-bolts were run through the shear plates and deck tubes to post-tension the entire deck. The entire structure was tested in four-point bending. The structure connection spacing was varied between 1-ft., 2-ft., 3-ft., and 4-ft. The testing revealed that structure stiffness varied inversely to connection spacing. Strain distribution results demonstrated a structure with behavior corresponding to a significant degree of composite action. At a deflection of 1.0 in., the stiffness of the structure with 1-ft. spaced connections was equivalent to 93.6% of the stiffness of full composite action. With 4-ft. spaced connections, the stiffness was equivalent to 86.0% of full composite action stiffness. This represents a significant increase over the minimum theoretical stiffness (no composite action), 66.7% of full composite action stiffness.

ELECTRICAL PROPERTIES OF METAL-FILLED COMPOSITES

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Abstract

The electrical properties of a new conductive adhesive were studied. In particular, direct current (DC) electrical measurements at various temperatures were made to study the conduction behavior using a four point probe. The samples were then subjected to three different tests using a four point probe. A percolation curve was made using these measurements in order to determine the percolation threshold of the conductive adhesive paste. Measurements were also made at various temperatures in order to determine the current/voltage behavior of the samples. An anomalous behavior was observed when the sample was subjected to a temperature cycle.

EFFECT OF PRESTRAIN ON THE CRYSTALLINE MORPHOLOGY OF A
SEMICRYSTALLINE COPOLYESTER

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Abstract

This study investigated the influence of uniaxial prestrain of amorphous precursors on the later thermally induced crystalline morphology. A 10/90 lactide/glycolide copolyester by weight was utilized for this investigation. The polymer was chosen on the basis of its hydrolytic sensitivity so that the fine crystalline morphology could be seen following chemical etching with an alkaline solution by Scanning Electron Microscopy (SEM). Other techniques were employed to confirm earlier reports of increasing nucleation density with increasing prestrain, as well as a transformation from spherulitic to row-like morphology at a critical prestrain level for a given set of prestrain conditions. The prestraining procedures involved uniaxially deforming the sample to a prescribed level prior to crystallization, after which it was allowed to rapidly retract and then quenched below T_g . These same precursors were later crystallized isothermally at 150°C. In selected cases, the prestrain level was maintained at the time of quenching as well as during the later crystallization step. Both methods showed similar results, with the transition to row-like morphology occurring near 200% prestrain. Following etching, the morphology revealed by SEM and Atomic Force Microscopy (AFM) indicated a fibrous, row-like structure with lamellar growth and polymer chains oriented in the direction of the strain. Morphological models are proposed to account for how prestrain causes local ordering which promotes higher nucleation.

COMPETITIVE ADSORPTION BETWEEN A CATIONIC POLYELECTROLYTE AND A CATIONIC SURFACTANT ONTO NEGATIVELY CHARGED SILICA

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Abstract

The competitive adsorption between a cationic polyelectrolyte, polydiallyldimethyl-ammonium chloride (PDADMAC), and a cationic surfactant, hexadecyltrimethylammonium chloride (CTACl), from aqueous solution onto silica substrates was examined using atomic force microscopy. Surface images and force measurements revealed the adsorption behavior for single-component and mixed systems. CTACl adsorbed as spherical micelles onto silica in a reversible process, and pre-adsorbed CTACl prevented PDADMAC adsorption. PDADMAC adsorbed as a featureless layer, independent of polymer concentration and molecular weight, in an irreversible process. CTACl adsorbed onto pre-adsorbed PDADMAC in a structure that was consistent with either spheres or hemispheres. Co-adsorption from polymer-surfactant mixtures showed the formation of a CTACl layer on top of adsorbed PDADMAC.

MECHANICAL MILLING AND ALLOYING OF HIGH PERFORMANCE POLYMERS

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Abstract

Mechanical milling (MM) (one component) and mechanical alloying (MA) (two components) were originally developed for processing metals. These new solid-state techniques are now used for processing and blending polymers. Two high performance polymers (PEEK and LaRC-TPI) were selected for a study of their morphology after this processing. The samples were milled at both cryogenic and ambient temperatures. A decrease in the crystallinity of the LaRC powders was found. However, strain induced crystallization was found in the PEEK powders upon milling. Milling and alloying produced a refined size and a more flake like texture of the powder particles. Cryogenic alloying produced a high level of mixing at the sub-micron scale in a plate-like lamellar structure. Processing produced some lowering of the degradation temperature. Based upon these experiments, MM and MA are viable processes for the processing and blending of polymers to achieve new and useful properties.

CHARACTERIZATION OF LIQUID CRYSTALLINE POLYESTER MORPHOLOGY

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Abstract

Novel thermotropic liquid crystalline polyesters were characterized using differential scanning calorimetry (DSC), polarized light microscopy (PLM), and melt rheology (MR). The control liquid crystal polyester, poly(hexamethylene 4,4'-biphenylate) (B6) was then modified with four different monomers: sulfonated dimethyl isophthalate (SDMI), dimethyl isophthalate (DMI), cyclohexane dimethanol (CHDM), and trans and cis isosorbide (ISB and ISM respectively). Each modifier was selectively chosen to induce certain characteristics in the liquid crystal. SDMI was chosen to introduce ionic intermolecular attractions and thus maintain liquid crystalline structure, although it also introduces a kinked effect because of its meta-substitution on the phenyl ring. The DMI was chosen to investigate this kink effect and serves as a control for the SDMI. The SDMI modified B6 was found to have a maximum effect of intermolecular attractions somewhere between 5 and 20 mole percent SDMI, and a comparison between 10% SDMI modified B6 and 10% DMI modified B6 showed a much greater degree of liquid crystallinity in the 10% SDMI modified B6. SDMI modified copolymers also generally formed a smectic A (Sa) phase as observed in PLM. CHDM modified B6 was found to raise the isotropic temperature (Ti) because it contains a more rigid spacer than the hexamethylene diol of B6. Addition of the ISB and ISM produced very interesting optical textures, either a chiral smectic C (Sc*) or a smectic E (Se) phase, although the region between the melting point (Tm) and the isotropic point was narrowed with increasing concentration of the isosorbide. Preliminary melt rheology studies of the liquid crystalline polyesters demonstrate transitions at temperatures indicated by corresponding DSC experiments.

SYNTHESIS OF PERFLUOROARYLENE-CYCLOPENTADIENE ALTERNATING COPOLYMERS

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Abstract

We investigated nucleophilic aromatic substitution polycondensation reactions of cyclopentadiene and decafluorobiphenyl. Characterization was hampered by dimerization of residual cyclopentadiene end groups and by poor solubility. The use of tetramethylcyclopentadiene in place of cyclopentadiene also afforded only low oligomeric species ($n=2$). Plans for an alternative route to perfluoroarylene-cyclopentadiene alternating copolymers are presented.

SYNTHESIS OF SULFONATED POLY(ARYLENE ETHER PHOSPHINE OXIDE)S FOR
PROTON EXCHANGE MEMBRANE/FUEL CELL APPLICATIONS

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Abstract

Sulfonated poly(arylene ether phosphine oxide)s are high performance materials that have the potential to be utilized as a proton exchange membrane in a fuel cell. Sulfonated bis(4-fluorophenyl)phenyl phosphine oxide, a new monomer, was prepared in 90% yield by the sulfonation of bis(4-fluorophenyl)phenyl phosphine oxide with fuming sulfuric acid. Sulfonated poly(arylene ether phosphine oxide)s were prepared by the aromatic nucleophilic substitution of bis(4-fluorophenyl)phenyl phosphine oxide and sodium sulfonated bis(4-fluorophenyl)phenyl phosphine oxide with 4,4'-biphenol in the presence of potassium carbonate in N-methyl-2-pyrrolidinone. The condensation polymerization was performed at 170°C for 16 hours and very high molecular weight polymers were obtained. The polymers had good thermal stability displaying 10% weight loss at temperatures above 460°C in nitrogen, which allowed the conclusion that the thermal stability of the polymers was not significantly affected by the substitution with sodium sulfonate groups. These polymers also had high glass transition temperatures, which increased with the concentration of the sodium sulfonate groups in the polymers. The polymers were quite soluble in dipolar aprotic solvents, the sodium sulfonate groups did not strongly influence the polymer solubility, and strong transparent cast films could be formed.

MEASUREMENT OF RESIDUAL STRESSES AND THERMAL EXPANSION IN ADHESIVE BONDS

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Abstract

Residual stresses are known to play a significant role in the mechanical failure, such as delamination, in adhesive layers and coatings. Curvature measurements of bilayer systems have been employed for several centuries to obtain information related to relative shrinkage and expansion of the layers. Timoshenko and Corcoran have laid the groundwork for beam and plate bending models, respectively, associated with this phenomenon. Recent work has sought to determine optimal geometries, which improve the sensitivity of the techniques for measuring the coefficients of thermal expansion for the coating. Common, commercially available DMA (dynamic mechanical analysis) and TMA (thermal mechanical analysis) equipment provides an especially attractive approach for quantifying the displacements under carefully controlled temperature exposure. As such, measurements of coefficient of thermal expansion and stress-free temperature for adhesives, coatings, and other laminated material can be conveniently made. This simple technique may prove useful in modifying processing steps to minimize residual stresses or increase dimensional stability of coated or bonded systems.

MECHANICAL PROPERTY ANALYSIS AND PREDICTION OF CORD-RUBBER COMPOSITES

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Abstract

A model to predict tire failure and damage modes would save the industry time and money. A few of the mechanical properties that affect these damage and failure modes are studied. Rubber compounds used in the matrix of the cord-rubber composite that comprise the tire were subjected to stress and creep testing. The test results are reported and the effects of slight changes in chemical composition of the compounds are analyzed. The test results were then used to predict mechanical properties of the composite material. This was done through using the mechanics of materials, improved mechanics of materials and the concentric cylinder models. Theoretical analysis of the elastic shear modulus is discussed.

CURE SCHEDULE EFFECTS ON THE GLASS TRANSITION OF CARBON FIBER
REINFORCED PET-ULTEM COMPOSITES

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Abstract

The use of phenylethynyl-terminated Ultem in carbon fiber reinforced composites was studied in order to achieve the optimal composite cure schedule. On the basis of previous research, T_g reduction due to matrix degradation was expected for long cure times and high cure temperatures. Cure advancement and possible matrix degradation were monitored via the glass transition temperature. Using a modified central composite design of experiment, nine composite panels were formed at various cure times and temperatures and then analyzed for T_g by DSC and DMTA. Quadratic surface response fits were applied and contour plots were produced. It was found that the composite specimens showed no T_g reduction for the tested conditions of 330 to 360°C and 40 to 120 minutes; longer cure times and higher cure temperatures produced higher T_g composites. This procedure was repeated for DSC cured neat resin samples and T_g reduction was observed.

APPLICATION OF PHOTOPOLYMERIZED RESINS IN ARTIFICIAL HIP REPLACEMENTS

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Abstract

The substitution of polymethyl methacrylate bone cements with photopolymerized resins for artificial hip replacements was explored. An annular cavity was created using transparent blue PVC. This cavity would be used to simulate the type of fixation necessary in the cavity between the hollowed out femur and prosthesis in a total hip replacement. This cavity was filled with photopolymerizing resin that was cured using a light source consisting of a linear array of 29 blue Panasonic light emitting diodes at 470 nm. The samples were tested between two parallel plates in a compression frame at a loading rate of 1mm/min. The compressive load applied created a shear stress that broke the bond between the two pipes. It was discovered that within this testing scheme, filler was necessary to create a consistent bond between the two surfaces. Without filler, composite cylinder shear strengths were measured on the order of 120 MPa. Once filler was added, shear strengths increased to over 400 MPa. These values were encouraging when compared to the bulk compressive strength of PMMA bone cements of 100 MPa.

AN EFFICIENT NEW CRYPTAND HOST FOR SUPRAMOLECULAR CHEMISTRY

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Abstract

A new bicyclic crown ether containing two 1,3,5-phenylene units linked by two tetra(ethyleneoxy) units and one pyridine-2,6-dicarboxylic acid unit was synthesized. This new cryptand forms a complex with N,N'-bis(2-hydroxyethyl)-4,4'-bipyridinium bis(hexafluorophosphate) with an association constant, $K = 4 \times 10^6 \text{ M}^{-1}$, 10,000-fold greater than that of an analogous simple crown ether macrocycle.

INVESTIGATION OF THERMAL RATCHETTING EFFECT IN CONDUCTIVE ADHESIVE JOINTS

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Abstract

The effect of thermal cycling on the state of residual stress in a viscoelastic conductive adhesive was investigated using the finite element method and Moir® interferometry technique. Characterization of the thermo-mechanical properties of an electrically conductive adhesive was performed experimentally through TGA and DMA. The actual size model system consisting of a chip capacitor bonded to an aluminum substrate with the adhesive was modelled using ABAQUS, and the ratchetting effect due to cyclic thermal load was reviewed around the corners of the adhesive where stress concentrations occur. The results indicate that thermally induced residual stresses in the adhesive joint may increase incrementally over time. The effect of varying glass transition temperature of the adhesive model on the stress profiles was also investigated using FEM. Moir® interferometry was used to confirm the change in the stress build-up after the model system experienced significant thermal loading.

A LATENT INITIATOR FOR PHENOLIC/EPOXY COMPOSITES

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Abstract

The high viscosity of phenolic/epoxy matrix resins makes them difficult to melt process. It is desirable to have an initiator that does not react at the ~140°C processing temperature, yet rapidly reacts at the ~200°C cure temperature. Thus a latent initiator comprised of triphenylphosphine sequestered in a polyimide thermoplastic was prepared and studied. The effects of this latent initiator on the stability and reactivity rates of phenolic/epoxy matrix resins was studied using differential scanning calorimetry and rheology. The rate of the cure reaction below the glass transition temperature of the sequestered initiator was relatively slow compared to the rate at a temperature above the glass transition temperature. The slower cure rate at the processing temperature allows for a longer processing time because the viscosity of the phenolic/epoxy matrix resin remains low for a longer amount of time than when neat triphenylphosphine is used. It was found that by using the latent initiator the stability of the matrix resin at the processing temperature could be increased and cure times as low as 16 minutes could be obtained.