An Investigation of Adhesion Between Poly(Arylene Ether Sulfone) Membranes and Platinum-Carbon Catalyst

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ABSTRACT:

The adhesion between the proton exchange membrane (PEM)-a proton conducting polymer- and the catalyst is the focal point for improved performance of a fuel cell. Plasmas of different gases, acetylene and acetonitrile, have been used to modify the surface of polysulfone polymers to enhance adhesion of platinum-porous carbon catalyst to sulfonated polysulfone films which are used as PEMs. The surface of the films was characterized by X-ray photoelectron spectroscopy (XPS). Quiet good surface coverage could be obtained for treatments with acetylene and acetonitrile plasmas. The topography of the films and surface modified samples was obtained using atomic force microscopy, AFM. Finally, to investigate the adhesion between the polymer film and the catalyst, peel tests were performed. The peel test results indicated good adhesion between the modified proton exchange membrane and the platinum-porous carbon catalyst.

Proton Conductivity of Sulfonated Poly(arylene ether sulfone) Copolymer Proton Exchange Membranes at Elevated Temperatures (60-150°C)

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Proton conductivities of sulfonated poly(arylene ether sulfone) copolymers with systematically varied degrees of sulfonation were measured between 60-150 °C under fully humidified conditions. Results demonstrated that the proton conductivities of these proton exchange membranes (PEM's) increased steadily with temperature, even beyond 100°C. The conductivities passed through a maximum before falling dramatically at higher temperatures. This maximum, referred to as the upper temperature limit, decreased with increasing degree of sulfonation. Thermal analyses of the membranes under hydrated conditions were utilized to probe the effects of hydration on Tg's. Glass transition temperatures of the fully hydrated copolymers and their upper limit temperatures were within 40 °C for all of the copolymer membranes, including the control perfluorinated copolymer, Nafion™.

Layer-by-Layer Self-Assembly of Nanostructured Polymeric Second Order Nonlinear Optic Ultrathin Films

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Abstract

Ionically self-assembled monolayer (ISAM) films may permit the fabrication of a new generation of second order nonlinear optic (NLO) devices. Hybrid deposition, a variant of the ISAM technique, is used to create films that consist of alternating layers of Procion Red (PR) and polyallylamine hydrochloride (PAH). This self-assembly process results in orientation of the PR, thus creating materials with very favorable second order NLO properties.

This research explored the time required for PR monolayer formation and the effects of the PR ionic strength on deposition. It was determined that PR monolayers are fully formed within five minutes. The introduction of NaCl into the PR solution causes a significant increase in PR deposition.

Controlled Size Macromolecular-Magnetite Complexes

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Abstract

Magnetite (Fe_3O_4) was stabilized in dichloromethane using a carboxylic acid containing poly(caprolactone) (PCL)-poly(vinylmethylsiloxane) (PVMS)-PCL triblock copolymer as an organic stabilizer. The block copolymer was synthesized using dihydroxybutyl-terminated poly(vinylmethylsiloxane) as the initiator, ε-caprolactone as the monomer, and stannous octanoate as the catalyst.[1] The block copolymers were synthesized with the molecular weights of the PCL blocks ranging from 2000g/mol to 8000g/mol, and the molecular weight of the PVMS block ranging from 1300g/mol to 4000g/mol. DSC analysis of the block copolymers displayed two glass transition temperatures (T_g), indicating microphase separation. Addition of carboxylic acid functional groups across the vinyl groups in the central block was ranged from 2.6 to 5 carboxylic acid groups per chain. Controlled nano-sized (~10nm) magnetite synthesized by chemical coprecipitation was sterically stabilized with the organic stabilizer. Magnetic microspheres (~20-80 μm) were prepared from the stabilized magnetite complexes using oil in water/solvent evaporation technique. Larger nano-sized magnetite (~50-500nm) was prepared by reduction of goethite in glycol/water solutions followed by crystallization.[2] The magnetite complexes were characterized using vibrating sample magnetometry (VSM) and scanning electron microscopy (SEM).

Contrasting In-situ and Bulk Constitutive Properties of Adhesives

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Abstract:

Finite element modeling for product design has become standard industrial practice. The modeling can be fairly simple for some material systems. However, when trying to characterize an adhesive bond that is polymeric, modeling complexity increases significantly. Proper modeling of adhesive joints is extremely important. Unfortunately, much of the input data for models come from a combination of bulk and in-situ tests that are assumed identical. This research project was an attempt to produce some fundamental understanding of differences between these in-situ and bulk properties. Several tests were designed to characterize an acrylic adhesive, supplied by Dow Automotive, and the strains at failure for three different geometries; normal bulk geometries, shear bulk and in-situ joint geometries. The results of these tests indicated that there are substantial differences between in-situ and bulk properties. Some preliminary insights were gained on potential factors contributing to the differences in the properties. Specifically, the strains to failure for as-received dogbone specimens were found to be much lower than the strains to failure for similar specimens that were milled to reduce surface irregularities. Similar results were also found in bulk torsion specimens. Specimen ductility was significantly enhanced through this grinding process. In addition, specimens similar to cracked lap shear geometries were prepared, but without the initial flaw. Grids were etched onto the surface of the specimen, and specimens were loaded in tension until debonding occurred. Photographs taken during the loading process were recorded in order to quantify the in-situ failure process. In-situ strains to failure were several times larger than those observed in bulk specimens.

Synthesis and Characterization of Directly Copolymerized Sulfonated Poly(arylene ether sulfone)s for Fuel Cell Applications

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Sulfonated poly(arylene ether sulfone)s were prepared by copolymerizing bisphenols with 4,4'-difluorodiphenylsulfone (DFDPS) and 3,3'-disulfonated-4,4'-difluorodiphenylsulfone (S-DFDPS). The copolymers were characterized by intrinsic viscosity, ^{1}H NMR, FTIR, equilibrium water absorption, proton conductivity, thermogravimetric analysis, dynamic mechanical analysis, atomic force microscopy, aging studies, and X-ray photoelectron spectroscopy. Copolymer films were prepared from the materials in their sulfonate salt forms, then the films were acidified. Materials were compared which had been acidified with sulfuric acid at room temperature versus in boiling sulfuric acid. Both the equilibrium water absorption and fluorine enrichment at the surface of the films were considerably higher for the films acidified at the elevated temperature. Further studies are needed to determine how the acidification procedure affects conductivity and morphology. Thermogravimetric analysis showed that the films were stable up to at least 200 °C, but the degradation temperatures decreased as the degree of disulfonation increased. Glass transition temperatures increased with increased degree of disulfonation. Aging studies demonstrated that crosslinking did not occur up to 220 °C (as determined by intrinsic viscosity) and no spectral changes were observed by FTIR up to 260 °C. The high proton conductivities and excellent mechanical stabilities of these films suggested that they may be effective candidates for proton exchange membranes in fuel cells operating at the desired temperatures of 100-140 °C.

POSS-Polymer Blends at the Air/Water Interface I: Blends of Amphiphilic PDMS and Amphiphilic Trisilanolisobutyl-POSS

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ABSTRACT: With rigid inorganic cores and flexible organic coronae of nanometer dimensions, polyhedral oligomeric silsesquioxane (POSS) molecules have attracted considerable attention for hybrid organic-inorganic nanocomposites, and synthetic templates for nanostructured materials. This study examines the compatibility of the two amphiphiles, trisilanolisobutyl-POSS and poly(dimethylsiloxane) (PDMS), as blended monolayers at the air/water interface (A/W) by the Wilhelmy plate technique and Brewster angle microscopy (BAM). A/W serves as an attractive surface for both components. Recent studies show trisilanolisobutyl-POSS has a stronger affinity for A/W than PDMS and forms a more condensed phase around a surface pressure of $\Pi \approx 18$ $\text{mN}\cdot\text{m}^{-1}$. Likewise, PDMS monolayers are known to undergo a series of transitions corresponding to multilayer formation at A/W. At surface pressures below any structural transitions for either PDMS or trisilanolisobutyl-POSS ($\Pi < 8$ $\text{mN}\cdot\text{m}^{-1}$), the mixtures are ideal within experimental error. The formation of a PDMS-bilayer that normally occurs around $\Pi \approx 8$ $\text{mN}\cdot\text{m}^{-1}$ shifts to higher surface pressures with increasing trisilanolisobutyl-POSS content indicating strong compatibility between the two materials. Furthermore, the amount of PDMS in the blend also alters the aggregation state of solid-like trisilanolisobutyl-POSS domains that form around ≈ 17 $\text{mN}\cdot\text{m}^{-1}$. These results have potential importance for the mechanical and optical properties of silicone based adhesives and sealants that need to operate in aqueous environments.

CARBON-PHENOLIC AND GLASS/VINYL-ESTER COMPOSITES UNDER COMBINED THERMAL AND MECHANICAL LOADING

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Abstract:

The deformation response of fiber-reinforced carbon-phenolic (FRCP) and glass/vinyl-ester (GVE) composites under combined thermal and mechanical loading is difficult to assess, in part due to the temperature-dependence of the mechanical (modulus, strength, lifetime, etc.) and thermal (specific heat, conductivity, etc.) properties. Analytical procedures incorporating the variable nature of these properties exist in literature but lack experimental verification. This yields a need for accurate quantitative values of these thermomechanical properties as functions of temperature to produce stronger and more rigid composites. The present study experimentally characterizes some of these properties for FRCP and GVE composites as functions of temperature. The study also measures the temperature and deformation response of a GVE composite exposed to compressive and thermal stresses, assuming one-dimensional heat transfer over a small area. An analytical finite-difference method is employed to analytically verify the experimental findings above and back out the incident heat flux. The effect of temperature on these thermal and mechanical properties can then be input into a finite-element package, ANSYS®, to model the local deformations that occur in a larger geometry.

Mechanical Properties of Glass/Feather Fiber Composites

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Abstract

Feather fiber obtained from waste chicken feathers has demonstrated many useful properties for composites as well as for other applications. The fibers are strong and durable with strength and modulus comparable to nylon. Due to recent advances in processing technology, the fibers can be easily extracted from the raw feathers bringing the price of the raw material down. Using wetlay processing, the feather fiber can be combined with a polymer matrix material to form a uniform, homogeneous, air-permeable mat that can be compression molded into desired shapes. In this study, the feather fiber was combined with chopped fiberglass and polypropylene to create a hybrid composite. A designed experiment was carried out to test the mechanical properties at various combinations of fiber loading. Based on the test results and observations made during processing, subjects for future projects are recommended. The experiment revealed that the feather fiber decreased the strength of the composite and had no effect on the modulus. Observations regarding the ability of these materials to absorb energy and sound are discussed.

Mechanism of Self-healing in Ethylene Ionomers

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(Abstract)

A series of poly(ethylene-co-methacrylic acid) (EMAA)-based ionomers have exhibited puncture reversal properties. Previous studies have shown that when healing occurs, the materials are heated by penetration of the projectile. To determine the minimum temperature at which the material significantly "welds" to itself, a simple peel test was performed. In addition, the effect of temperature on the mechanism of self-healing was examined using projectile penetrations on films at different temperatures. Differential scanning calorimetry (DSC) of the punctured samples was conducted to determine the change in the sample thermal properties after penetration. DSC results showed that the projectile erased the thermal history of the samples.

Results of these tests show that the ionomers do not knit until their melt temperature is approached. In addition, no healing was observed for films between 60°C and 90°C. Visual evidence indicates substantial viscous flow around the punctured hole unlike the elastic response that takes place at room temperature.

Modeling and Imaging of Transport Phenomena in the Catalyst Layer of Proton Exchange Membrane Fuel Cells

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A membrane electrode assembly (MEA) from a proton exchange membrane fuel cell (PEMFC) was examined using a Scanning Electron Microscope (SEM) and a Transmission Electron Microscope (TEM) to understand the transport phenomena related to charge and species transfer within the active catalyst/electrode layer of the MEA. These visual insights were combined with an analytical model based on Bultel; Ozil; and Durand (1998) that characterizes electro-catalyst utilization in the catalyst/electrode layers (cathode and anode) of the MEA. Results from the analytic model are presented as are catalyst characterization data from the SEM and TEM analyses.

Material Properties of GFRP Reinforcing Bars for Bridge Decks

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Abstract

Corrosion of steel reinforcing bars in concrete has lead researchers to look for new materials. Glass fiber reinforced polymer (GFRP) bars have become a valid option. Only a few long term studies have been done on the durability of GFRP bars in concrete. This currently ongoing research deals with strength retention over time in an accelerated aging tank. The tank contains water saturated with calcium hydroxide at 30°C, 45°C and 57°C. The bars are submerged in water for specific lengths of times so strength tests can be performed. Tensile tests could not be performed on the bars so flexural tests were used instead. Preliminary findings show a strength reduction as the temperature increases. The GFRP bars also underwent moisture absorption experiments. These tests will determine how much water the bars absorb after various lengths of time. The maximum moisture content has not been reached in the bars yet, so no conclusions can be drawn. The ultimate goal of this experiment is to find an equation that models strength retention over time at a given moisture content and temperature.

Synthesis of Crown Ethers

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Abstract

A new method for selectively monotosylating diols released on the web in June by *Organic Letters* was used in the first step in the synthesis of crown ethers. The starting diol is tosylated in the presence of silver (I) oxide and a catalytic amount of potassium iodide using this method. The products were characterized using NMR and the yields and selectivity were much lower than previously reported. More work needs to be done before this is an efficient and effective method to make crown ethers.

The Influence of Concrete Pore Water on the Epoxy Coating of Reinforcing Steel

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Abstract

In this study, the influence of concrete pore water on the epoxy coatings on reinforcing steel was investigated. The epoxy coatings on epoxy coated reinforcing steel (ECR), exposed to different conditions, were evaluated for direct comparison of coating properties. The samples consisted of controlled ECR samples, ECR from a bridge deck, and ECR from concrete structures in outdoor conditions with and without chloride exposure. Concrete cores extracted measured 75mm in diameter with cover depths of 25 to 75mm. The concrete cores were evaluated for chloride content at the ECR depth by chloride titration. The thickness of the epoxy coatings was determined. The scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) evaluated the topography and chemical analysis of the epoxy coatings. The differential scanning calorimeter (DSC) determined the thermal glass transition temperature (Tg) and thermal gravimetric analysis (TGA) recorded quantitative changes in the mass of the epoxy coatings.

Results from the ECR testing confirmed that the epoxy coatings on reinforcing steel absorb moisture over a period of eight years. Therefore, pore water ions may diffuse into the coating. There exists a correlation between the increased moisture contents and higher Tg temperatures. There is apparent cracking in the coating from the field sample and there are not visible cracks in the laboratory samples up to eight years. The cracking in the coating corresponds with discoloration of the epoxy coating in field samples.

Synthesis and Characterization of Self-Complementary Multiple Hydrogen-Bonded (SCMHB) Polymers

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Abstract

Reversible polymer properties have enhanced the interest in self-complimentary multiple hydrogen bond (SCMHB) polymers. In this study, the effects of hydrogen bonding on polymer characteristics and the preservation of this bonding through rigorous reactions involving pendant groups of the polymer are observed. Polymers containing SCMHB methacrylate were copolymerized free radically with monomers having a relatively high glass transition temperature. The t-butyl group on poly (t-BMA-co-SCMHB methacrylate) was then cleaved by acid catalyst. The monomer, SCMHB methacrylate, was synthesized through a coupling reaction of 2-isocyanatoethyl methacrylate (ICEMA) and methyl isocytosine (MIS) in dimethylsulfoxide (DMSO). SCMHB methacrylate was incorporated with tert-butyl methacrylate (t-BMA) and with methyl methacrylate (MMA). The characteristics of glass transition temperature (T_g), polydispersity (PDI), degradation temperature, molecular number, and molecular weight were then examined as a function of SCMHB and initiator concentration. Using thermo gravimetric analysis (TGA), the t-BMA copolymers displayed a marked weight loss at 240^{o}C and at 415^{o}C for up to 10 mol% SCMHB methacrylate content. Based on size exclusion chromatography (SEC), as initiator, 2,2'-Azobisisobutyronitrile (AIBN) concentration increased in the synthesis of t-BMA and MMA copolymers, the average molecular number decreased. Using proton nuclear magnetic resonance (^{1}H NMR) a spectrum of hydrolyzed poly (t-BMA-co-SCMHB methacrylate) confirmed that the t-butyl group had been removed through acid catalyst; however, hydrogen bonding persistence could not be determined.

Langmuir-Blodgett Film Formation of POSS-Derivatives

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Abstract

Polyhedral oligomeric silsesquioxane (POSS) molecules have attracted considerable attention for hybrid organic-inorganic nanocomposites, and synthetic templates for nanostructured materials by virtue of their rigid inorganic cores and flexible organic coronae of nanometer dimensions. The formation of silicon dioxide upon the thermal and oxidative degradation of POSS/polymer composites or blends, and POSS-polymers offers the possibility of creating thin self-healing heat-resistant coatings. This study examines the surface morphology of mixed monolayers at the air/water interface comprised of poly(t-butyl acrylate) (PtBA) blended with trisilanolphenyl-POSS (TPP), or PtBA blended with POSS-polymer, a statistical copolymer (CpBMA) of n-butylmethacrylate and methacryl-POSS, by Brewster angle microscopy (BAM) and the Wilhelmy plate technique. BAM shows that CpBMA monolayers become heterogeneous as POSS content increases and that PtBA serves as a dispersant of CpBMA when added in the form of a co-spread monolayer. In contrast, PtBA/TPP blends are nearly homogeneous for all compositions in the monolayer state. Both blended systems are capable of quantitatively forming Langmuir-Blodgett (LB) films. Atomic force microscopy is used to determine the thickness, roughness, and long range order within these LB-films. This study provides key advances in sample preparation methods for future studies of ultrathin space-survivable and heat-resistant coatings.

Thermo-Mechanical Properties of Sulfonated Polysulfone Copolymers

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Thermo-mechanical properties of sulfonated polysulfone copolymers (BPSH) were analyzed by thermogravimetric analysis, dynamic mechanical analysis and differential scanning calorimetry. TGA results indicated that the sulfonate salt forms of these materials had higher degradation temperatures (~500 ºC) than the acidified forms of the copolymers (~330 ºC). The temperatures defining the onset of degradation of the copolymers decreased slightly with increasing degrees of disulfonation. Dynamic mechanical analyses indicated that selected compositions of these copolymers had two Tg's which may correspond to the Tgs of the hydrophobic domain (the lower Tg) and hydrophilic domain (upper Tg). The acidified copolymers had higher hydrophobic domain Tg's and lower hydrophilic domain Tg's than the salt forms. BPSH copolymer films which were acidified in sulfuric acid at 100 ºC (vs. acidified at room temperature) had higher Tg's corresponding to the hydrophobic domains and more prominent transitions for the hydrophilic domains. This suggests that the higher acidification temperature resulted in phase mixing of the hydrophobic and hydrophlic regions. DSC demonstrated that Tg's of the BPSH copolymers were depressed by water absorption and these effects could be approximated by Fox-Flory analyses. For example, the Tg of BPSH with 40% of the units sulfonated decreased from 267 ºC to 118 ºC as the water content was increased from 0 to 56%. These results suggested that the hygro-thermal acidification conditions may cause morphological changes in these materials.

Synthesis and Characterization of Sulfonated Poly(arylene ether) Copolymers Containing Aromatic Nitriles

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High molecular weight nitrile-functional, (hexafluoroisopropylidene)diphenol based aromatic poly(arylene ether)s with pendent sulfonic acid groups have been prepared by nucleophilic aromatic substitution. 4,4'-(Hexafluoroisopropylidiene)diphenol, 2,6-dichlorobenzonitrile, and 3,3'-disulfonate-4,4'-dichlorodiphenylsulfone were copolymerized under basic conditions in N-methyl-2-pyrrolidinone at 202 °C. Polymers containing as much as 55 mole % sulfonation formed tough ductile films. The films were converted from the salt to the acid form in dilute aqueous sulfuric acid. Thermogravimetric analysis demonstrated that the acidified copolymers had excellent thermal stability up to 300 °C in air. A series of these materials with systematically varied concentrations of the sulfonic acid moieties had increased glass transition temperatures, proton conductivities, and hydrophilicities as the degree of sulfonation was increased. Films containing greater than or equal to 20 mole % of the sulfonated monomer had Tg's of 250 °C and higher. The copolymer comprised of 35 mole % of the sulfonated monomer had a proton conductivity greater than 0.10 S/cm at 110 °C. Proton conductivities for these materials were also measured under controlled relative humidities. As the level of sulfonation was increased up to 45 mole %, the nitrile containing copolymers became more sensitive to changes in % relative humidity. Atomic force microscopy (AFM) demonstrated that the acidified copolymer with 35 mole % of the repeat units sulfonated was phase separated into a co-continuous morphology comprised of hydrophobic and hydrophilic domains.

MEASUREMENT OF THE INTERLAMINAR STRENGTH OF THE 36″ DOUBLE WEB BEAM FLANGE

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Abstract

Fiber – reinforced polymeric (FRP) composites are being used in bridges, buildings, and other civil structures as alternatives to steel and concrete because of their light weight and durability. Testing conducted on a 36 inch double web beam (DWB) for use in bridge construction has shown failure by delamination due to high interlaminar (out-of-plane) stresses. With a goal of developing a strength prediction to assist in designing bridges using the DWB, a stress based approach using the Quadratic Strength Criterion is utilized. The strength values in the criterion were determined through short-beam shear (SBS) testing and tensile testing where the peak loads were measured. The stresses at failure were obtained by finite element analysis (FEA), analytical models, and Weibull statistics were then utilized to determine the mean strength values. FEA and analytical models are being explored to predict the critical interlaminar stresses to complete the strength prediction