The Time and Temperature-Dependent Behavior of PLASTILOCK® 731SI

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Abstract

Bulk material properties of a two-part epoxy called PLASTILOCK® 731SI were analyzed using an Instron and dynamic mechanical and thermal analysis (DMTA). Elastic modulus, Poisson's ratio, ultimate tensile stress, and percent elongation measurements were taken by the Instron using a test matrix of three temperatures and four strain rates. For the most part, mechanical properties followed expected trends for temperature and strain rate changes. Methods for decreasing voids in casts evolved over time. Also, the material was found to be more sensitive to curing methods than expected. Three different curing methods were tested. One involved heating the samples to 127 ˚C for two to five minutes. Other samples were heating to 127 ˚C for several hours. The final curing method involved three separate bakes of 127 ˚C, 171 ˚C, and 140 ˚C sequentially. This curing method emulated automotive assembly line conditions and proved to be highest in modulus and toughness out of the tested curing methods. The glass transition temperature of the Plastilock material heated using this approach was determined by DMTA to be about 122 ˚C, and the corresponding activation energy of this relaxation was approximated to be about 231 kJ/mol.

SYNTHESIS, CHARACTERIZATION AND PRELIMINARY BIOCOMPATIBILITY STUDIES OF SULFONATED (POLY ARYLENE ETHER SULFONE) COPOLYMERS – ACID AND SALT FORM

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Abstract

The synthesis, characterization and preliminary evaluation of the potential for biocompatibility of sulfonated poly(arylene ether sulfone) copolymers is described. A series of copolymers made from 4,4'-biphenol and a stoichiometric mixture of 3,3'-disulfonated and non-sulfonated dichlorodiphenyl sulfone has been considered with the degree of sulfonation varying from 10-60%. The polymers were characterized by intrinsic viscosity, FTIR, DSC, TGA and molecular weight by GPC. Preliminary evaluation of the potential of these copolymers for biocompatibility and biomedical applications was conducted via water uptake and contact angle measurements. For this, the copolymers were cast into films from 10 wt.% solutions in DMAc. Films were converted from the salt form to the acid form through treatment with concentrated H_2SO_4, and from the acid form to sodium salt form by treatment with base. The surface of the films were characterized by XPS and the characterization showed that films were fully converted to the intended form. The water uptake measurements showed that the acid form absorbed a maximum of 800 wt%, and the salt forms absorbed a maximum of 70 to 99 wt%. The contact angle between a water droplet and the surface of the copolymer films in the potassium salt form was measured using the Sessile drop method, and decreased as a function of the degree of sulfonation, in the range of 80-40°.

PROPERTIES OF SULFONATED POLYMER BLENDS: INFLUENCE OF COMPOSITION OF PARTIALLY FLUORINATED SULFONATED COPOLYMERS ON SURFACE AND MEMBRANE BEHAVIOR. I.

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Abstract

The apparent miscibility and membrane properties of biphenol-based sulfonated poly(arylene ether sulfone) copolymer (BPS-35) with hexafluoroisopropylidene biphenol based poly(arylene ether sulfone) (6F-00) and hexafluoroisopropylidene biphenol based sulfonated poly(arylene ether sulfone) copolymer (6FS-60) copolymer as a function of weight fraction were investigated in this research. Polymer blends were prepared in the sodium salt form from dimethylacetamide (DMAc) and membranes of the blended poly(arylene ether sulfone)s were solution cast onto glass substrates. The miscibility of the blended polymers decreased as the weight percent of the 6F-00 or 6FS-60 to the BPS-35 increased. The thermal stability, mechanical and surface properties of the blended films were characterized using water uptake, specific conductivity, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and contact angle.

Electrospun Poly(L-Lactic Acid) Interfaces for Bone Tissue Engineering

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Abstract

Advancement of the field of bone tissue engineering requires the development of biomaterial scaffolds that can regulate cell adhesion, proliferation, and bone tissue development. Poly-L-lactic acid (PLLA) is a promising scaffold material that has good mechanical strength, and can be processed readily into a variety of porous structures. Conventional processing techniques produce structures with feature sizes greater than or equal to the size of cells ($\geq$ 20 μm), but evidence indicates that cell attachment, spreading, and orientation are especially sensitive to feature sizes of 0.5-2 μm. To test the effects of such small features, substrates were prepared by electrospinning PLLA from a solution in hexafluoroisopropanol. This produces a microtextured surface comprised of randomly oriented fibers with diameters (0.5 to 5 μm) that depend on deposition conditions. Several different electrospinning parameters were varied in order to produce a range of fiber diameters. MC3T3-E1 mouse osteoprogenitor cells were then seeded on these substrates and evaluated for cell density, morphology, and F-actin content. Significant differences between the electrospun surfaces and the control (smooth) surfaces were observed, but the differences between substrates produced from different electrospinning conditions were not observed. Overall, cells on electrospun surfaces exhibited a 70% lower projected cell area, and 40% lower cell density after 1 day of attachment. These initial studies demonstrate that these microtextured substrates have a significant impact on cell behavior, and future work will examine their effect on phenotypic markers of osteoblastic differentiation.

CHARACTERIZATION OF NOVEL POLY(ETHYLENE-BUTYLENE)-SILOXANE SEALING MATERIALS

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Abstract

Synthesis and characterization of allyl terminated- poly(ethylene-butylene) as well as the chain extended allyl-terminated poly(ethylene-butylene) is described. This was used in the formation of poly(ethylene-butylene)-siloxane networks. The low viscosity of the precursor materials and minimal siloxane content of networks make them viable candidates for sealing a proton exchange membrane fuel cell stack. The thermal properties and swelling behavior of aged and unaged networks possessing different degrees of chain extension and filler concentrations were investigated.

OPTICAL PROPERTIES OF GOLD NANOPARTICLE AND POLY(ALLYAMINE HYDROCHLORIDE) SYSTEMS

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Abstract

It has long been known that colloidal gold nanoparticles absorb light because of surface plasmon resonance. The absorbance characteristics of gold nanoparticles are affected by a number of factors including particle shape, particle size, host medium, and surface molecules attached to the particles. In this study, the effect of adding poly(allylamine hydrochloride) to various colloids of gold nanoparticles is investigated. It was found that the positively charged polymer attached to the negatively charged surfaces of the gold nanoparticles. This led to the formation of clusters consisting of gold particles and polymer. It was found that for the 5 and 10 nm particles the addition of PAH to the colloid caused the absorbance peak to red shift, broaden, and increase in intensity. These changes in the absorbance behavior were attributed to interactions between the particles and the PAH, as well as interactions between nearby particles. The clusters formed from the 5 and 10 nm particles were seen to have layers of polymer separating the individual nanoparticles, preventing free electrons from tunneling from one particle to another. When PAH was added to colloids of 20 and 30 nm particles, a second absorbance peak emerged at higher wavelengths than the original peak. This broad peak increased in intensity as more polymer was added to the colloids. We observed that the particles making up these clusters were in contact with one another. It is believed that free electrons are delocalized across the 20 and 30 nm particle clusters, leading to the emergence of the second absorbance peak. Based on the location of the high wavelength peak, it was estimated that the clusters formed from the 20 and 30 nm particles are made up of an average of four to six nanoparticles.

DETERMINING THE MODE I ADHESIVE FRACTURE ENERGY OF DOW AUTOMOTIVE LOW ENERGY SURFACE ADHESIVE

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Abstract

A study to determine the Mode I fracture energy (G_{Ic}) of Dow Automotive low energy surface adhesive (LESA) using double cantilever beam (DCB) specimens was performed. DCB specimens were prepared using aluminum substrates etched with a P2 solution to provide adequate adhesion of LESA. An investigation into the effects of bond thickness and post-cure temperature on G_{Ic} values was carried out. A procedure for testing DCB specimens by Blackman and Kinloch provided the method for collecting and analyzing the necessary data.[1] The average G_{Ic} was 1920 J/m^2 on control specimens with a bond thickness of 0.8 mm and a post-cure temperature of 40 °C. In addition, a preliminary fatigue test at 5 Hz was conducted to determine crack growth properties under cyclic loading.

The Effect of Maleic Anhydride Modified Polypropylene on the Mechanical Properties of Feather Fiber, Kraft Pulp, Polypropylene Composites

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ABSTRACT

Composites made of feather fiber (Ff), kraft pulp fiber (Kf), polypropylene (PP), and increasing amounts of maleic anhydride modified polypropylene (MaPP) were tested in tension and three-point bend tests. The composites were made using a wetlay papermaking process which yields a non-woven, fabric-like material called prepreg. The prepreg was subsequently consolidated into plaques by compression molding multiple layers. Of the plaques containing all four materials, the best mechanical properties were observed with 8 wt% MaPP. The mechanical properties of PP-matrix composites containing either Ff or Kf improved with additions of up to 8 wt% MaPP. The addition of MaPP improved the fiber matrix interface and the effects can be seen on the tensile fracture surfaces using scanning electron microscopy.

Investigation of Macromolecular-Magnetite Dispersions

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Abstract

Two methods were developed for the synthesis of magnetite in the 100-500 nm size ranges. The approximate diameters of the magnetite samples were determined using scanning electron microscopy (SEM). A carboxylic acid-containing polycaprolactone block copolymer stabilizer was used to coat the surface of the 500 nm magnetite particles. The 100 nm magnetite particles were coated with itaconic acid. The coating of the particles was verified using X-ray electron spectroscopy (XPS). The coated magnetite samples were used to form 10 wt% magnetite/vinyl ester resin dispersions. The polycaprolactone-coated 500 nm magnetite samples remained dispersed in the vinyl ester resin for 12-24 hours depending of the molecular weight of the polycaprolactone stabilizer. The itaconic acid-coated 100 nm magnetite samples remained dispersed in the vinyl ester resin for 72 hours.

Synthesis of *trans*-Dibenzo-24-Crown-8 Diester

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Introduction

Crown ethers were first reported in 1967.[1] In the report, it was noted that crown ethers complex with metal cations. The Gibson group has been synthesizing larger crown ethers that complex with organic molecules, such as secondary ammonium salts.[2] The electronegative oxygens donate electron density to cations within the ring cavity, thus creating a thermodynamically stable complex. Hydrogen bonding between acidic ammonium protons and the oxygens in the ring, as well as pi-pi stacking interactions between aromatic rings, stabilize the compound and create a psuedorotaxane. This is a self-assembling, non-covalent interaction, which is reversible. The association between the crown ether (host) and the salt (guest) may be measured experimentally as an association constant (K_a). The association constant provides a quantitative value for the interactions between the host and guest. *cis*-dibenzo-24-crown-8 diester has been successfully synthesized by the Gibson group. *trans*-dibenzo-24-crown-8 diester is desired for complexation and cryptand comparisons.

$$K_a = \frac{[\text{Complex}]}{[\text{Host}][\text{Guest}]} \qquad \textbf{Eq. 1}$$

XPS and Optical Microscopy Analyses of Polystyrene and Trisilanolphenyl-POSS Bilayer Films

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ABSTRACT

Polyhedral Oligomeric Silsesquioxanes (POSS) are hybrid organic-inorganic molecules that have attracted interest as nanofillers for self-healing high-temperature materials for aerospace applications. Here, bilayer films of trisilanolphenyl-POSS (TPP) and polystyrene (PS) have been prepared by spincoating PS onto a hydrophobic silicon wafer substrate and transferring TPP on top of the PS via the Langmuir-Blodgett technique. These bilayer films were then annealed over a range of temperatures (no annealing – 200 °C). The effects of annealing on these bilayer films of TPP and PS were examined by X-ray photoelectron spectroscopy (XPS) and optical microscopy. It has been found that bilayers of spin-coated PS with TPP deposited on top interdiffuse with one another. After the PS and the TPP layers mix, there is still a surface excess of TPP relative to the bulk concentrations of PS and TPP. Furthermore, optical microscopy has shown that the samples undergo phase separation and dewetting as the annealing temperature approaches 200ºC. These results have potential importance as alternatives to solely carbon-based coatings that are currently being used on satellites and other devices that are in space for prolonged periods of time because of POSS' ability to withstand atomic oxygen and large temperature fluctuations.

The Synthesis and Solution Properties of Poly(ethylene oxide)-*b*-Poly(2-ethyl-2-oxazoline)

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Abstract

The synthesis and solution properties of the hydrophilic diblock copolymer Poly(Ethylene Oxide)-*b*-Poly(2-Ethyl-2-Oxazoline)/Poly(Ethylenimine) (PEO-PEOX/PEI) through macro initiation of PEO followed by cationic polymerization with 2-ethyl-2-oxazoline with acid hydrolysis to produce PEO-PEOX/PEI. The molecular weight of the PEO oligomers were established via ^{1}H NMR and GPC. GPC results also indicated a narrow molecular weight distribution of the PEO starting materal. Thissynthesis was repeated with varying molecular weights of PEO and PEOX, then the PEO-PEOX hydrophilic diblock was characterized by dynamic light scattering (DLS) to show that the diblock forms aggregates in aqueous solution. The PEO-PEOX diblock aggregates were determined to have a large hydrodynamic radius of 27 nm.

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Surface Plasmon Resonance Studies Focused on Heparin-Protein Binding

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Abstract

Angiogenesis is the sprouting of new capillaries from pre-existing vessels. In 1971, it was hypothesized that tumors are angiogenesis dependent and later works have lent credence to this theory. Basic fibroblast growth factor (FGF-2) is one of the proteins secreted by tumor cells to promote angiogenesis. FGF-2 must first bind to a heparn sulfate proteoglycans (HSPG) in order to trigger its receptor, which leads to the activity cascade responsible for much of the angiogenic activity in cells. If the FGF-2 can be prevented from interacting with its receptor in tumor cells, angiogenesis would be reduced, preventing further growth. The purpose of this experiment was to develop a procedure using Surface Plasmon Resonance to study the affinity of FGF-2 for a series of heparin fractions so that the heparin with the greatest affinity for FGF-2 could be identified and then used to create an analog that would block angiogenesis. The paper discusses the experiments undertaken to develop an immobilization process for FGF-2 that would allow it to interact with the heparin fractions to produce strong, clear signals that could be used to determine the k_a and k_d of each reaction.

An Investigation of Photo-Crosslinking in the Design of Tailored Macromolecules

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ABSTRACT

The preparation of pressure sensitive adhesives via free radical polymerization allows for tailoring of polymers with specific adhesive properties and applications. The introduction of photoactive groups such as coumarin provides further functionality for photocrosslinking, allowing tunability of adhesive properties. This photoreversible property of coumarin containing polymers was the basis of this study. A series of seven acrylate copolymers with a wide range of glass transition temperatures (T_g) and ester alkyls were synthesized and functionalized with a coumarin derivative. The extent of crosslinking was monitored using UV-Vis spectroscopy and gel fractions. Steric and T_g effects were studied to determine their effects on the extent of photodimerization. At 5% coumarin functionalization, all copolymers were crosslinked after being irradiated at 300 nm. The study demonstrated that T_g and temperature of irradiation influence the extent of photodimerization, while sterics appear to have no effect. However, a steric effect was evident during the introduction of the coumarin derivative to the copolymer precursors. Gel fractions were unaffected by T_g and steric factors.

Surface Composition Of Platinum-Coated Sulfonated Polysulfones For Use As Polymer Electrolyte Membranes In Fuel Cells Using Plasma Deposition

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Abstract

Sulfonated polysulfones are under development as an alternative polymer electrolyte membrane to Nafion. Plasma deposition of platinum complexes was explored as a method of catalyst deposition on the polysulfone surface. Via plasma deposition, platinum adhered to the surface. The atomic percent composition on the platinum-polysulfone surface was determined via x-ray photoelectron spectroscopy (XPS).

Fatigue Performance Variability of a Structural Composite for Calculating Load and Resistance Factor Design (LRFD) Parameters

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Abstract

Load and Resistance Factor Design (LRFD) is emerging as the dominant design philosophy in civil and naval structural engineering that focuses on optimizing a structure for a desired reliability over its lifetime. The introduction of fiber reinforced polymer (FRP) composites to replace steel and concrete in structure designs requires an assessment of how the probability distribution of the material resistance (strength or stiffness) changes with time and environmental effects over long lifetimes. This paper documents a Monte Carlo simulation method under development at Virginia Tech that predicts the remaining strength distribution of a material subjected to tensile fatigue loading. This paper also includes data from fatigue and remaining strength testing of a pultruded composite material for verification of the simulation and the comparison of preliminary results. The simulation currently predicts a wider variation in remaining strength than that of the actual material. Lastly, further testing is underway to develop a more complete set of verification data at a variety of load levels.

The Determination of Moisture Content in Concrete

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August 8, 2003

Abstract

Concrete is a porous, complex material used in a variety of civil engineering applications. Concrete is classified as a composite comprised of aggregate, sand, and lime in a cement paste matrix. The complexity of concrete is derived from the inhomogeneous, porous nature of the material. Pore structure consists of pores in the aggregate, pores between aggregate and paste, capillaries in the paste, micropores, and air voids. Permeability, which consists of flow, diffusion, and adsorption, is directly affected by pore structure, porosity, capillary pressure, gravity, aggregate size, pore geometry, and moisture content. Previous research has found that permeability has the greatest impact on fluid transport in concrete [2].

Fluid transport in concrete has been modeled theoretically by fluid mechanics and diffusion theory. Permeability has been studied using Darcy's Law and Kozeny Theory. Fick's Law is especially useful in determining moisture diffusion as a function of relative humidity. Laplace's Equation models the capillary pressure [8]. Through theoretical modeling, it is possible to develop a greater understanding of fluid flow in concrete.

Theoretical modeling does not take into account the erratic, complex nature of concrete. It also does not model the environment with certainty. Changes in temperature, humidity, conditions during formation, freeze thawing, and deicing agents also affect the moisture content and fluid flow through concrete. As a result, a more reliable method of determining moisture content must be developed to correctly assess fluid transport.

PREPARATION OF THIN FILMS OF FLUORINATED CELLULOSE DERIVATIVES USING THE LANGMUIR-BLODGETT TECHNIQUE

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ABSTRACT

Esters of cellulose and trifluoroethoxy acetic acid (CTFEA) were examined with respect to their surfactant properties and their ability to form Langmuir Blodgett (LB) multilayer films. Esters with low degree of substitution (DS), and with a DS from 0 to 3.0, had hydrophobic character that lessens retention moisture, which is otherwise typical of cellulose esters with low DS. By preparing bilayers of CTFEA on trimethylsilylcellulose (TMSC) or isopentyl cellulose (IPC) on TMSC, we could show through contact angle measurements that CTFEA was more hydrophobic than IPC. This unexpected result was further explored by hydrolyzing the TMSC layer. The resulting CTFEA/cellulose and IPC/cellulose bilayers were again investigated by contact angle measurements. For IPC layers, there is a clear transition from the hydrophilic cellulose surface to a hydrophobic IPC surface with increasing IPC layers thickness. In contrast, the CTFEA covered surface remains hydrophilic over the first 8 layers. These results suggest fluorinated cellulose derivatives are more hydrophilic than expected.

The Determination of the Interlaminar Fracture Toughness of Hybrid, Composite Beams

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Abstract

There are two main approaches for predicting failure: the strength based approach and the fracture mechanics based approach. The fracture mechanics based approach was investigated in this research to assess the moment capacity of the Strongwell 36 inch DWB (double web beam). Test specimens were cut from failed beams far from the damaged area and tested in an Instron-testing machine. Mode I testing was conducted under the guidance of ASTM D 5528-01 using MBT (modified beam theory) and area (trapezoidal rule) methods for data analysis. Longitudinal specimens were successfully tested and apparent mode I fracture toughness values determined, but the transverse specimen values were not valid in this study because the delaminating path could not be controlled. High G_{IC} values (1250-2460 J/m^2) were observed for all specimens, as well as high standard deviations (263-425 J/m^2). All loading-unloading curves were non-linear, and the non-linearity increased with each successive curve. The area method resistance curves were very scattered, indicative of unstable crack propagation. Both the high G_{IC} values and the non-linearity are due primarily to fiber and fiber bundle bridging. Also, unloading curves did not return to zero extension while returning to zero loads giving an indication of plastic deformation. Hysteresis was evident by examining the loading-unloading curves.

It was determined that for the purposes of the 36 inch DWB, the strength based approach is more appropriate for predicting delamination initiation and the fracture mechanics based approach may be useful for predicting delamination propagation in fatigue life predictions.

Surface Active POSS-co-n-butylmethacrylate Copolymers

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ABSTRACT

Polyhedral oligomeric silsesquioxane (POSS) molecules exhibit hybrid organic-inorganic characteristics providing impetus for scientific study and technological applications such as self-healing high temperature nanofillers, low-k dielectric materials, and a platform for nanostructured materials design. Copolymerization of methacrylcyclopentyl-POSS with n-butylmethacrylate yields grafted POSS units on a poly(n-butylmethacrylate) (PnBMA) chain that alters the surface properties and morphology of PnBMA films at the air/water (A/W) interface. While PnBMA forms a two dimensional monolayer, the resulting POSS copolymers, three statistical copolymers (CpBMA) of n-butylmethacrylate and methacrylcyclopentyl-POSS, show decreasing surface activity and increasing rigidity with increasing POSS content. Surface studies of PnBMA and the copolymers were accomplished using □-A isotherms and Brewster's angle microscopy (BAM), while film deposition onto solid substrates was accomplished by the Langmuir-Blodgett (LB) technique. The roughness of the LB-films were characterized using atomic force microscopy in tapping mode.

Mechanical and Electrical Properties of Polymeric Composite Monopolar Plates For Fuel Cells

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Abstract

This investigation studies the electrical and mechanical properties of graphite polymeric monopolar plates for use in fuel cells. The mechanical and electrical properties of unaged plates and plates aged in a fuel cell environment for over 300 hours were measured to determine if the decrease in the fuel cell performance was attributable to changes in the properties in the monopolar plates. Results determined that the electrical conductivity of the plates did not decline over the lifetime of the fuel cell test, even though the performance of the fuel cell decreased. The mechanical strength of the monopolar plates declined approximately 10% after 300 hours of fuel cell operation. While the mechanical properties of the plates does not directly impact fuel cell performance, increasing the duration that the monopolar plates operate in fuel cell may cause significant degradation of the plates and eventual failure. The electrical conductivity of the plates did not after aging, however the performance of the fuel cell declined over the life of the test due to degradation of the MEA. Future work on the electrical and mechanical properties of fuel components should address the factors that involved in the mechanical degradation of the monopolar plates and performance loss of the membrane electrode assembly (MEA).

Network Formation on Magnetic Cobalt Nanoparticles using Poly(styrene-b-4-vinylphenoxyphthalonitrile) Copolymers

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Abstract:

The network formation of poly(styrene-b-4-vinylphenoxyphthalonitrile) anchor blocks on the surfaces of cobalt nanoparticles using nucleophilic initiators was studied. The nucleophilic initiators chosen for this study were bisphenol-6F and phenylhydroquinone. Cure temperatures used were 225˚C, 250˚C, and 275˚C for 12 hours. A model cure study was preformed using only copolymers and nucleophilic initiators with varying molar ratios of 0.0:1, 0.10:1, 0.25:1, 0.50:1, and 0.75:1 (moles of nucleophilic initiator to moles of phthalonitrile). Percent conversion of phthalonitrile anchor blocks were monitored and calculated using Fourier Transform Inferred Spectroscopy (FT-IR). The observed percent conversion of phthalonitrile anchor blocks ranged from 0% to 81%, depending on the nucleophilicity of the initiator and cure temperature. Cobalt ferrofluids were formed via the thermal decomposition of dicobalt octacarbonyl in a solution of toluene and the block copolymer. Ferrofluids were then concentrated to a solid and crosslinked in a quartz-tube furnace under inert conditions. The crosslinked material was then redispersed in toluene via sonication. TEM data presented suggests that the polymer coated cobalt nanoparticles were effectively redispersed.

CREEP RESPONSE OF A GLASS/VINYL ESTER COMPOSITE PRODUCED BY VACUUM-ASSISTED RESIN TRANSFER MOLDING

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Abstract

A summary of viscoelasticity gives context to the current work, which begins with an outline of the fabrication of a particular glass/vinyl ester composite by the vacuum-assisted resin transfer molding (VARTM) method. Experimental details are established for creep testing of the composite and preliminary tests were conducted. Analysis seeks to determine linear and nonlinear Schapery parameters to enable prediction of mechanical behavior of the composite. Although the power law fit the creep curves quite well, creep recovery curve fitting was less successful. Reasons include significant nonlinear behavior at the lowest stress level tested, which hinders the determination of linear parameters, and unaccounted for offset in the creep recovery. Progress made and challenges encountered shed light on future areas of focus.