

CATALYST EFFECTS ON THE KINETICS OF NOVOLAC CURED WITH EPOXY RESIN

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Abstract

The kinetics of curing a phenolic novolac with an epoxy resin were studied using dynamic and isothermal differential scanning calorimetry. Phenolic/epoxy networks have been shown to be flame retardant and tough, ideal characteristics for structural composites. The tertiary phosphine catalyzed cure reaction involves nucleophilic attack by a phenolate on the epoxy ring. Although the reaction will proceed in the absence of a catalyst, relatively high cure temperatures and long times are required. A tertiary phosphine catalyst generates the active phenolate nucleophile and thus allows the reaction to proceed faster and at lower temperatures. Knowledge of the dependence of kinetic parameters on catalyst concentration and temperature will allow the rate of reaction to be controlled for maximum processing efficiency. Following ASTM E698, the activation energy for epoxy ring opening using triphenylphosphine and tris(2,4,6-trimethoxyphenyl)phosphine catalyzed reactions was calculated to be 71 ± 3 kJ/mol. A linear relationship between molar catalyst concentration and calculated first order rate constants for disappearance of epoxy groups was observed. Also, the effect of changing catalyst structure on both activation energies and rate constants was insignificant. This data substantiates that the reaction rate was dependent on the concentration of phenolate nucleophile and epoxy groups, and that the rate of phenolate generation was rapid in relation to the epoxy propagation reaction. Rate constants determined from apparent activation energies (from dynamic runs) agree with rate constants determined from isothermal DSC.

PHASE DIAGRAM OF EPOXY RESIN MODIFIED WITH
POLY(PHOSPHINE OXIDE -CO-SULFONE SULFIDE)

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Abstract

A phase diagram of the poly(phosphine oxide-co- sulfone sulfide) (PPO/PSS) &endash; diglycidyl ether of bisphenol A (DGEBA) system was constructed. Homogeneous blends of PPO/PSS and DGEBA were prepared. A light transmission system was used to find the phase separation point (cloud point temperature) for each sample by measuring the transmitted intensity as a function of temperature. Two types of intensity versus temperature curves were obtained, those exhibiting a one-step mechanism, and those exhibiting a two-step mechanism. For each blend, the onset cloud point temperature was plotted against rate and extrapolated to zero heating rate. A plot of cloud point temperature at zero heating rate against weight fraction was constructed, yielding a phase diagram. A lower critical solution temperature was observed in the PPO/PSS-epoxy system, consistent with the hydrogen bonding present between phosphine oxide in the polymer and the hydroxyl group of the epoxy. An upper critical solution temperature was observed at high concentrations of PPO/PSS and will be further investigated.

The Development of a Dynamic Wedge Test for the Study of Double-Cantilever Beam Specimens: Initial Study of the Failure of Polyimide Adhesives Subjected to Impact Loading

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Abstract

The research presented is a study of test design and initial results for the fracture mechanics of double-cantilever beam, polyimide adhesive joints subjected to impact loading. Testing procedures used were specifically designed for this experiment and had to be both developed and calibrated to understand the involved dynamic effects. Loading is applied to the adherends through the impact of two aligned, wedges passing between the load pins. Data was collected with a high-speed camera and analyzed for crack length and adherend displacement. Simple beam theory modeling was used as an approximation so as to employ the load method for calculating the strain energy release rate. Load values were not measured because the dynamics of the test would produce large distortions in the data.

The Effects of Silane Coupling Agents on the Mechanical Performance of Alumina-silica
Ceramic Fiber Reinforced Vinyl Ester Composites

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Abstract

Ceramic fiber/vinyl ester composites were made from fiber pre-forms coated with various silane coupling agents. The two ceramic fibers pre-forms (B and C) were non-woven and isotropic. Three different silane coupling agents (Dow Z-6030, Dow Z-6032, and Dow Q9-6300) were applied to the ceramic fibers before the fibers were fabricated into composites by means of Resin Film Infusion (RFI). Studies on tensile strength and modulus were conducted to elucidate the effects the silane sizing had on mechanical performance. Composite B sized with 0.25wt% Dow Z-6030 displayed a 35% increase in tensile strength and a 10% increase in modulus when compared to the unsized case. Composite C sized also with 0.25wt% Dow Z-6030 showed a 28% increase in tensile strength and a 40% increase in modulus.

Secondary Crystallization Kinetics of Poly(ether ether ketone)

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Abstract

Kinetics of secondary crystallization of poly(ether ether ketone) has been studied by means of differential scanning calorimetry measurements. A systematic study of melting behavior of the semicrystalline polymer leads to the conclusion that its double melting endotherm is not a consequence of melting-remelting-recrystallization processes.

Applying Controlled/"Living" Radical Polymerization to Surface Modification of Silica Gel

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Abstract

Silica gel can be used as a model for glass in order to develop a technique for modifying the surface via a controlled/"living" radical polymerization. The two major types of controlled/"living" radical polymerizations are Stable Free Radical Polymerization (SFRP) and Atom Transfer Radical Polymerization (ATRP). The controlled/"living" radical polymerization produces polymers whose molecular weights and molecular weight distributions can be controlled as has been demonstrated in the literature for both SFRP and ATRP. In our work the initiator 2,2 ϕ -azobis [2-methyl-N-(2-hydroxyethyl)propionamide] (VA-086) has been functionalized by trimethylchlorosilane to produce trimethylsilane azo-initiator (TMSA), which was tested for SFRP. The TMSA was prepared as a model for an azo-initiator attached to a silica gel surface. TMSA has been shown to be an efficient initiator for the SFRP of styrene. Chain extension and block copolymerization reactions have been performed on an isolated polymer prepared with TMSA and 2,2,6,6-tetramethylpiperidine-N-oxide (TEMPO).

Synthesis of Poly(arylene ether phosphine oxide)s via Melt Polymerization

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Abstract

Melt synthesis of poly(arylene ether phosphine oxide)s via the silylation route (1) was investigated. Previously, these polymers were synthesized in solution (2) which produced impurities requiring additional steps for removal. Melt polymerization produces pure products without extra purification steps. One specific poly(arylene ether phosphine oxide), Bis A PEPO based on Bisphenol A and bis(4,4'-fluorophenyl) phenyl phosphine oxide was studied. The catalyst concentration needed to give complete reaction of the monomers was found to be 0.3 mole percent cesium fluoride based on the amount of silylated bisphenol A. The molecular weight of the polymer was controlled by offsetting the stoichiometry of the monomers and the final molecular weight of the products was predictable using Carother's Equation. The thermal behavior of the melt synthesized polymers compared favorably to the solution standard. Endgroups were incorporated into the polymers of lower molecular weight. Data is still being gathered to determine if the polymer chains are completely endcapped.

POLYURETHANE INTERPHASE MATERIALS FOR CARBON FIBER/ VINYL ESTER COMPOSITES

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Abstract

Polyurethanes were synthesized to place in the interphase region of carbon fiber reinforced vinyl ester composites. The polyurethane composition was altered to decrease interdiffusion between the polyurethane and vinyl ester network to improve adhesion within the composite. Two soft segment materials were investigated, and the amount of the di (3-hydroxypropyl) isobutyl phosphine oxide and N-methyldiethanolamine chain extenders were independently varied to study their effects on polyurethane / vinyl ester interdiffusion. Miscibility between the polyurethane sizings and the vinyl ester matrix was studied using dynamic mechanical analysis and atomic force microscopy. For all investigations, the vinyl ester resin was cured for one hour at 140°C. Varying the soft segment material and the amount of phosphine oxide chain extender affected the interphase region. There was a gradual transition from the vinyl ester to the polyurethane, and the interphase region was 60-100mm in width. However, as the amount of amine chain extender was decreased, the microstructure of the interphase region was changed. An interface could be seen where the continuous phase changed from vinyl ester to polyurethane.

THE EFFECT OF SURFACTANT ON THE MOISTURE UPTAKE PROPERTIES OF GLASS-BEAD FILLED EPOXIES

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Abstract

The consequence of an added surfactant on the moisture uptake properties of waterborne epoxies is of key importance in estimating the operational lifetime of a waterbased circuit board. Continuing research of the epoxy from the diglycidyl ether of bisphenol A (DGEBA) cured with 2-ethyl-4-methylimidazole (EMI-24) in the presence of the nonionic surfactant Triton X-100 has been performed. This epoxy was filled with either untreated or pretreated E-glass beads (30% wt.) to study absorption behavior. The pretreatments consisted of the coupling agents 3-aminopropyltriethoxysilane (APS) and 3-glycidoxypropyltrimethoxysilane (GPS). Initial results indicate an enhancement of water uptake in the epoxy by the surfactant is only achieved when the glass filler is present. The initial diffusion of moisture for the pretreated glass beads was similar for epoxy with and without surfactant. However, slight differences were observed in equilibrium moisture uptake percentages. The viscoelastic properties of these filled epoxies were also monitored before and after moisture exposure through the use of cooperativity plots and showed that no significant changes occurred.

Influence of Adsorbed Polymers on Inter-Particle Forces in Silica Colloids

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Abstract

Atomic force microscopy (AFM) was used to model the forces acting on a colloidal silica particle. Surface forces were measured in aqueous solutions of electrolyte and polymer. The polymers were linear poly(ethyl oxazoline), PEOX, of molecular weight 11,700 g/mol and a fifth-generation poly(ethylene imine) (PEI) dendrimer of molecular weight 8,250 g/mol. Cantilever deflection-piezo position data were interpreted to give force-separation plots between silica wafers and spheres with and without polymers present. A short-range repulsive force was introduced by PEOX, suggesting adsorption of PEOX onto silica surfaces. This adsorption reaches equilibrium quickly. We believe the mechanism of repulsion is steric hindrance between interpenetrating polymer chains. Preliminary data were gathered for the PEI dendrimer; this polymer creates a larger repulsive force but reaches equilibrium more slowly.

INVESTIGATION OF OPHTHALMIC LENS SCRATCH RESISTANCE

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Abstract

Investigation on Ophthalmic lens, made of bisphenol-A polycarbonate (PC) and allyl diglycol carbonate (CR-39), scratch resistance was conducted. Two steps of examination were completed. First, coated PC and CR-39 lens materials were tested with industrial scratch resistance techniques, bayer[1] and steel-wool. Results from these two scratch resistance measurements were analyzed quantitatively. Quantitative analysis that utilized optical microscopy, scanning electron microscopy (SEM), and atomic force microscopy (AFM) determined the shape and amount of damage. Coated PC gives longer, thinner, and shallower as well as less scratches than the coated CR-39 in the quantitative outcome. Thus, it was of interest to study the physical properties, such as hardness and surface tension, of the PC and its coating. The hardness analysis from AFM nano-indentation, Vicker's micro-hardness, and AFM nano-scratching experiments show that the coating is harder than the PC itself, and in fact thickening the coating does not provide more scratch resistance. The surface tension measured using contact angle showed that the uncoated PC had higher surface energy than the coated one; therefore, in this case the PC substrate and its coating had good adhesion.

Moisture Exposure Effects on Carbon Fiber-Reinforced PhenylEthyne-Terminated Ultem™
Composites

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Abstract

Environmental exposure can affect the performance of many materials, including fiber-reinforced composite materials. In high-performance commercial aviation applications, materials must be able to withstand both extreme temperature and humidity and have resistance to solvents and other chemicals. Due to their high-performance thermal and mechanical characteristics, poly(etherimides) should be considered for such applications. Prior to such use, environmental effects on composite performance must be understood. Unidirectional composite panels were manufactured from a phenylethyne-terminated poly(etherimide) and G40-800 carbon fiber, exposed to high humidity, and tested for transverse flexural and apparent interlaminar shear strength. Test conditions and results of moisture exposure have been reported.

CORRELATION OF CRACK DENSITY AND RESIDUAL STRENGTH AND STIFFNESS
OF SATURATED VINYL ESTER E-GLASS COMPOSITE

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Abstract

The effect of the combination of fatigue and saturation on the mechanical behavior of stitched [90/0]3S Vinyl Ester E-glass composite was studied. Cracks were formed in the material by fatiguing specimens at 17% of the ultimate tensile strength. The crack density was measured using radiographs. Specimens were then saturated in a 65° saltwater bath. The rate of diffusion did not vary between specimens of different crack densities. Results showed that there was a significant loss in strength in specimens which were both fatigued and saturated, 20% of the normalized strength, compared to specimens which were only fatigued. However, there was not a significant loss in stiffness between fatigued specimens and specimens which were both fatigued and saturated.

INVESTIGATION AND OPTIMIZATION OF PLASMA PRETREATMENT AND POLYMERIZATION ON Ti-6Al-4V FOR ENHANCED BOND PERFORMANCE

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Abstract

The aerospace industry has recently gained particular interest in titanium-adhesive joints due to titanium's high strength to weight ratio, its high performance applications and its ability to withstand high temperatures. Consequently, titanium is used as a material for the high-speed civil transport, which will fly at Mach 2.4. Presently no durable surface preparation has been developed for the Ti-6Al-4V alloy. In the past, chromic acid anodization was found to give superior strength performance; however, degradation occurred at high temperatures. Recently, plasma polymerization has developed into an attractive alternative for surface pretreatment, due to the ability to obtain it in a solvent free process, and its unusual combination of performance, durability, safety, and cleanliness. Plasma polymerization produces thin films on an adherend of less than 1000°, with a high cross-link density, low solubility, and high thermal stability. A bond that fails cohesively, rather than adhesively, is desired. The plasma polymerization of acetylene was investigated in this study. Plasma treated Ti-6Al-4V was bonded with a polyimide adhesive and tested. Bond failure occurred within the acetylene plasma-formed hydrocarbon layer. However, results of single lap shear strengths indicate that oxygen plasma exposed to Ti-6Al-4V for 20 minutes improves the bond strength. Partial cohesive failure occurred in the adhesive indicating that the treatment was sufficient to prevent interfacial failure.

Electronic Effects of Ancillary Ligand Aryl Substituents on the Ethylene Polymerization
Activity of Group 4 Metallocene Catalysts

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Abstract

Two isomeric metallocene catalysts, 1,1'-diphenylzirconocene dichloride and 1,3-diphenylzirconocene dichloride, were prepared by standard chloride-substitution methods. The 1,1'-diphenylzirconocene dichloride catalyst was found to have an activity of $3.4(7) \times 10^7$ gPE mol⁻¹Zr atm⁻¹C₂H₄ hr⁻¹ for methylalumoxane-cocatalyzed solution polymerization of ethylene at 0 °C. Comparison with the results for the isosteric 1,1'-bis(pentafluorophenyl)zirconocene dichloride $7.1(1) \times 10^7$ gPE mol⁻¹Zr atm⁻¹C₂H₄ hr⁻¹ under identical conditions confirms the activity-retarding influence of the highly electron-withdrawing pentafluorophenyl substituents in the latter catalyst.

POLYSTYRENE SPHERES AS A MODEL OF TWO-DIMENSIONAL NON-EQUILIBRIUM
PHASE TRANSITIONS

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Abstract

An apparatus was designed to perform an experiment in which an oscillating drive was applied to a two-dimensional system of spherical polystyrene particles to observe the affect of the drive on the ordering of the arrangement of the particles. In initial experiments, no change in order has been observed with the oscillating drive.

Influence of Polymer Network Structure on Mechanical Response

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Abstract

Vinyl ester and epoxy network polymers were studied for two tasks. Vinyl ester resins were studied for the influence of the network structure on their viscoelastic, physical and mechanical properties. The crosslink density of the resins was altered by changing the molecular weight of the vinyl ester oligomer and by varying the styrene used during the crosslinking reaction. The glass transition temperatures of the polymers and the breadth of the glass transition regions were found to increase systematically with the increase of crosslink density without additional influence of the composition. Cooperativity studies were also performed in the glass formation temperature region of the networks. The cooperativity of the systems seemed to be influenced by both the crosslink density and the styrene content. A linear correlation was found between the fracture toughness of the networks and the cooperative domain size at the glass transition temperature normalized by the crosslink density. The crosslink density of the epoxy resins was varied with three different molecular weights of the crosslinking agent poly(oxypropylene)diamine. The tensile stress-strain behavior of the polymers due to the change in the crosslink density was studied. Ultimate tensile properties of the systems were also obtained to re-examine the existence of a proposed master failure envelope.

The Effect of Surfactant on Water Sorption and Viscoelastic Behavior of Waterborne Latex Epoxy Resins

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Abstract

The effects of added surfactant on the properties of Epi-rez 3510-w60 latex epoxy resin were studied. Neat resin systems and glass cloth composite laminates including 0% and 10% added surfactant (by weight), were prepared. These samples were subjected to a series of experiments including immersion density, gravimetric moisture analysis, and dynamic mechanical analysis (DMA) which involved both a temperature scan and in-situ isothermal immersion test. Immersion density techniques were used to determine the percent porosity in the samples as it may effect the moisture analysis. Apparent diffusion coefficients were determined from the moisture analysis using Fick's law. DMA was then carried out to find what effect both the added surfactant and moisture exposure had on the glass transition of the material. The Fox equation was then employed. It was determined that the surfactant was sufficiently distributed in the system to cause plasticization of the polymer chains. Because this plasticization effect fit the Fox equation, it can be assumed that there are no large domains of surfactant in the neat resin which would affect water sorption. This result was supported by the diffusion coefficient data which showed no statistically significant difference in water sorption between the cast latex resins and the cast latex resin systems with additional surfactant.

RESPONSE OF ELLIPTICAL COMPOSITE CYLINDERS TO TEMPERATURE CHANGES

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Abstract

Elliptical composite cylinders are planned for future high speed aircraft, and since high speeds cause high temperatures, elliptical composite cylinders must be analyzed to find how they respond to temperature changes. This paper develops an analysis for analyzing the thermal deformation of elliptical composite cylinders. As opposed to using equilibrium, the concept of minimizing total potential energy is utilized. Calculus of variations is used to solve for the thermal displacements, and by doing so, boundary conditions for the physical system emerge. The Rayleigh-Ritz method is then employed to approximate the displacements and create ordinary differential equations that can be easily solved. Thermal deformation for several elliptical cylinders, isotropic and composite, are then analyzed. It is found that circumferential displacement accompanies axial and radial displacements for elliptical cylinders, and also that material orthotropy is an important influence in the thermal displacements.

THERMAL STABILITY, CRYSTALLIZATION, AND MORPHOLOGY OF A NEW SEMICRYSTALLINE POLYIMIDE BASED ON 1,3-BIS (4-AMINOPHENOXY) BENZENE AND 3,3',4,4'-BENZOPHENONETETRACARBOXYLIC DIANHYDRIDE

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Abstract

The effects of melt time and temperature as well as crystallization temperature on spherulitic growth rate and morphology were determined for a new semicrystalline polyimide based on 1,3-bis (4-aminophenoxy) benzene and 3,3',4,4'-benzophenonetetracarboxylic dianhydride. Experiments were run on thin films (ca. 1 mils) and thick films (ca. 2 mils). The data show that in the temperature range of 340 – 380°C nucleation density increases as crystallization temperature decreases. The nucleation density decreases however when melt time and temperature are increased. Differential Scanning Calorimetry (DSC) was utilized to determine the thermal stability and recrystallizability after elevated times and temperatures in the melt. DSC revealed that the material had a glass transition of ca. 230°C and two distinct melting transitions at 350°C and ca. 412°C. It was found that the lower melting point and associated heat of fusion (DHf1) remained relatively stable after elevated melt times and temperatures until the harshest melting conditions (460°C, 30 mins) were realized. The higher melting point and associated heat of fusion (DHf2) were much less stable even at milder melting conditions. This indicated that the development of the higher melting crystalline phase was more sensitive to melt time and temperature than the lower melting phase. Structural analyses including Atomic Force Microscopy (AFM) and Small Angle X-ray Scattering (SAXS, data not presented) suggest the morphology is of a folded lamellar structure. Dynamic Mechanical Analysis (DMA) was conducted on various samples of this material that had been annealed at different temperatures from an amorphous state. The data indicate that milder annealing conditions (300°C, 2 hours) produce less crystallinity than annealing at higher temperatures. Also, the modulus of this material remains relatively high well above the glass transition temperature due to the presence of crystallinity.