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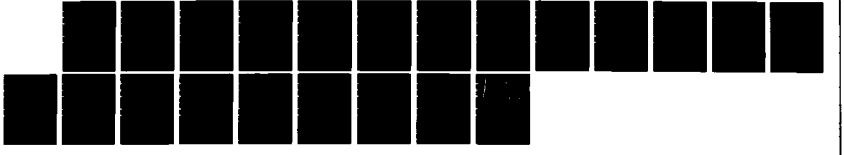

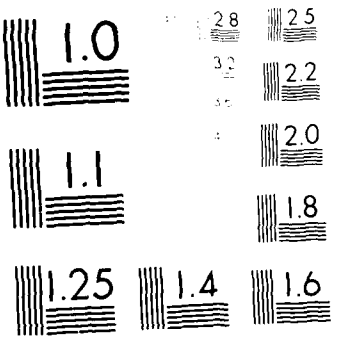
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Composit Structures
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The conference was held as schedules. Topics discussed included composites as orthopedic implants, ceramic particles in colloidal systems, polymers in information systems, multiplayer composites, crystalline polymers, amorphous polymers, crystalline polymers and cellulose fibers.

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PROCEEDINGS

NINTH ASILOMAR CONFERENCE ON POLYMERS

February 9-12, 1986

SPONSORED BY

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NEWARK, NEW JERSEY

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UNIVERSITY OF WASHINGTON
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ABSTRACTS

NINTH ASILOMAR CONFERENCE ON POLYMERS

February 9-12, 1986

Asilomar, California

HIERARCHICAL CLUSTERING OF CERAMIC PARTICLES IN COLLOIDAL SYSTEMS

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Abstract

In ceramic processing, powder consolidation and sintering is commonly used in microdesigning of polycrystalline and polyphase composite systems. In this approach, three basic steps are: (i) processing and dispersion of powders, (ii) consolidation of powders either with the use of liquid suspensions or dry pressing techniques, and (iii) densification of powder compacts with the application of heat and/or pressure. In the first step, colloidal dispersion techniques are needed to eliminate particle clusters that form uncontrollably due to van der Waals attractive interactions. This approach is especially useful when we work with multiphase particle systems in the 10^{-6} to 10^{-9} m range. In the second step, transitions from dispersed to consolidated state start as a nucleation and growth process of first generation particle clusters. These particle clusters form when either the interparticle binding energy, E, or the particle number density, ρ, in the suspension exceeds a critical value. We outline these transitions with a theoretically calculated E vs. ρ colloidal phase diagram. Hierarchically clustered microstructures form due to multiple clustering of particle clusters. Consequently, in such hierarchically clustered microstructures, the classification of the void space follows a similar trend as first, second, and third generation voids. Unlike the particle clusters that we wanted to eliminate in the dispersion stage, the size and the spatial arrangement of the particle clusters that form can be controlled by simply changing the colloidal consolidation path on the phase diagram. In the third step, the evolution of the microstructure follows a path influenced by the hierarchy of particle clustering. First generation particle clusters sinter faster and at significantly lower temperatures than the higher order void regions. This evolutionary path may be characterized by the methods familiar to us in statistical mechanics.

ABSORBABLE COMPOSITES AS ORTHOPAEDIC IMPLANTS

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Combining alpha polyesters, such as polylactic acid polymer (PLA) with carbon fiber, hydroxylapatite ceramic (HA), tri-calcium phosphate ceramic (TCP), various organic acids, and calcium sulphate, we have been able to produce materials of greatly varying mechanical properties that elicit some desirable biological responses. Depending upon the formulation chosen, these materials appear to have applications in both soft and hard tissue repair and replacement. Ligaments and tendons have been reconstructed with a filamentous carbon device coated with an absorbable polymer to prevent premature fragmentation of the carbon fiber. Fracture repair has been effected with a laminated composite bone plate of either carbon or calcium phosphate glass fiber reinforced PLA. HA-TCP-organic acid and HA-calcium sulphate composites have been developed as bone graft substitutes.

Ribbon-like structures of uniaxially aligned carbon fiber coated with PLA have been used to repair defects in tendons and ligaments in a number of animal models and clinically in over 1,500 patients worldwide. The composite acts as a scaffold for the regrowth of host collagenous tissue. As the thin polymer coating on the implant absorbs, fibroblasts from the host tissue invade the carbon fiber scaffold and new collagen is laid down. The new collagenous tissue ultimately provides a considerable percentage of the strength of the repair. Scanning electron microscopy of biopsy material has shown that the polymer has been absorbed and new collagenous tissue has grown in and about the carbon fiber scaffold. Light microscopy showed a proliferation of fibroblasts surrounding each fiber producing a mantle of well-formed collagen.

Reinforcing PLA with carbon or absorbable glass fiber offers the possibility of producing an implant with adequate strength to support the musculoskeletal system - bone plates, screws, IM rods, etc. The advantages of such devices are twofold. First, there would be no need for secondary surgical procedures to remove absorbable devices. Second, with time, the devices degrade mechanically, reducing stress protection and accompanying osteoporosis. By virtue of the fiber reinforcement, the devices have excellent initial properties. Witn time, the PLA matrix degrades and the plate loses rigidity, gradually transferring load to the healing bone. An osteotomy healing study and an osteoporosis study performed with canines have proved this concept. Ongoing in-vivo and in-vitro degradation studies are providing data for an application for the human use of degradable fracture fixation.

Particulate ceramics of hydroxylapatite and tri-calcium phosphate have been used as bone defect-filling materials. These materials have proven quite successful in several oral surgical procedures. However, usefulness as an implant material has been limited partly because of migration of the particles from the implant site. Consequently, we have put these materials in combination with resorbable setting agents. When water is added to these ceramic/binder combinations, they set to form solid composite materials. Setting times, resorbability, and porosity of these materials varied depending on the components used. To test the biocompatibility and bone ingrowth properties, the materials were placed in drill holes in the tibias of white New Zealand rabbits. Histologically, all of the implanted composites exhibited good biocompatibility and bone apposition or tissue ingrowth. They have been found to remain in place until the defect is filled or the implant is secured by new bone.

Ninth Asilomar Conference
on Polymers

HIERARCHICAL ORGANIZATION AND MECHANICAL BEHAVIOR IN COMPOSITE STRUCTURES

by
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Cleveland, Ohio

ABSTRACT

The relationships between hierarchical organization and mechanical behavior will be discussed using two widely different systems. Firstly, thermotropic liquid crystalline polyesters which are a new class of polymeric materials which have unique molecular and solid-state structures, flow characteristics and mechanical properties will be described. Injection molded plaques were found to consist mainly of three, highly anisotropic, flow induced macrolayers: two skins and a core in-between. By using fractographic methods, the internal solid state structure of the macrolayers was elucidated. A hierarchical structure has been proposed describing the observed levels of organization. The skin macrolayer has a distinct structural gradient comprised of three subdivisions from the surface inward: a highly top oriented layer, several oriented sublayers and a less oriented inner zone. The top layer is fibrillar in nature and the sublayers consist of stacks of interconnected microlayers. In the core,no well defined substructure was boserved, yet molecular orientation perpendicular to the injection direction represented the localized flow patterns. Similar structural observations for short fiber reinforced composites have also recently been completed indicating relationships between the anisotropic structure of the fibers and matrix.

Secondly, the structure which results from solid state extrusion using biaxial orientation was analyzed for oriented polypropylene. Structural changes on the spherulitic, lamallar, and macromolecular level during orientation were investigated using optical microscopy (OM), small angle x-ray scattering (SAXS), and wide angle x-ray scattering (WAXS). The results showed that polypropylene spherulites undergo stepwise biaxial affine deformation and deform homogeneously into a disc-like morphology. During this spherulitic flattening process, lamellar rotation into the planar direction occurs prior to lamellar break-up at a biaxial draw ratio of about 1.5. On the macromolecular level, the crystalline c-axis orients in the plane concurrently with the lamellar break-up, while the crystalline b*-axis gradually orients normal to the plane. Amorphous chains are also oriented preferentially in the plane of deformation. A hierarchical model was proposed to illustrate the nature of the orientation in the flattened spherulites. Currently, we are seeking correlations between this hierarchical structure and the unusual mechanical properties of compressively oriented polypropylene.

POLYMERS IN INFORMATION SYSTEMS

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The rapid growth in the generation, processing, transmission and storage of information offers new opportunities for specialty polymers. In particular, the development of optical systems for data processing and storage creates opportunities for polymers which are designed to exhibit specific responses to optical stimuli. The important area of photolithography for circuit construction and fabrication will not be addressed in this paper which will concentrate on two new developments, those of Optical Data Storage and Non-Linear Optical Phenomena.

Optical Data Storage offers important advantages in data density and is dependent on materials which change some physical property under the influence of laser irradiation. There are a number of potential methods of achieving this effect, the most germane for this conference being the dye/polymer system where the input radiation is absorbed and converted to heat, the heat then rapidly and reproducibly causes local, controlled, changes in the polymeric system.

Non-Linear Optical Phenomena rely on the interaction of intense (laser) light with the electronic structure of materials. These interactions lead to specific changes in the transmitted or reflected light; for example, the frequency may be doubled or tripled depending on specific structural parameters. Organic materials are the structures of choice for NLO phenomena due to their attributes of extremely high speed responses. However, organic single crystals are very poor materials from an engineering point of view, thus the drive towards incorporating the NLO active moieties into polymeric structures which would then provide the desired combination of optically non-linear activity together with the durability and ease of fabrication associated with polymeric structures.

MECHANICS OF LOW DENSITY MATERIALS*

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ABSTRACT

Mechanics analyses are used to derive the effective elastic moduli for low density materials. Both open cell and closed cell geometric models are employed in the case of isotropic media. The five independent effective moduli are derived for a low density transversely isotropic medium. Compressive strength, as defined by elastic stability, is also derived for open cell and closed cell isotropic materials.

The work is restricted to materials of very low density (high porosity) specification whereby three dimensional continuum mechanics forms and solutions can be taken to the limit of plane stress conditions in the micro-material members on the scale of individual cells. In this connection, it is intended to use only well developed theoretical mechanics solutions whereby effective properties of the low density materials are fully predicted by information on cell geometry and the inherent properties of the constituent material, with no adjustable parameters or constants of proportionality. The major purpose in the present work is to establish predicted modulus and strength differences between open cell and closed cell materials of very low density, and in the process to infer some characteristics of optimal cell geometry.

The main conclusion to be drawn from this work involves the "trade off" in properties between the open cell type material and the closed cell form. Within the framework of the geometric micro-structure models employed here, the closed cell material has a modulus which is about three times greater than that of the open cell form. On the other hand,under hydrostatic compression the closed cell material has a compressive strength, as limited by buckling, which is less than that of the open cell form by a factor of about "c" which is the volume fraction of material. Since volume fraction "c" could be very small in the low density range, there is a very large difference in performance between the two types of materials, favoring the open cell form.

The theoretical results have been corroborated by comparison with experimental results for a low density, reticulated vitreous carbon material of relatively high uniformity and material distribution. The material has been tested by compression of prismatic specimens. Four specimens had measured moduli of 30.5 MPa. The corresponding theoretical prediction proceeds from Eq. (5) wherein it is necessary to know the material modulus E_m. The appropriate handbook value [12] for E_m of industrial carbon is given by E_m = 6.9 GPa. The volume fraction of the material tested is .031. Using these data in Eq. (5) gives a theoretical prediction of E = 36 MPa, which compares quite well with the experimental value. Therefore, it appears that this material comes close to the "best" performance level predicted by the present theory for an open celled material.

*This work performed under the auspices of the U. S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

"ORGANIZATIONAL STRUCTURE AND MECHANICAL FUNCTION IN SOFT CONNECTIVE TISSUE"

Anne Hiltner

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ABSTRACT

Soft connective tissues are designed to serve unique functions in mammals. Three examples - tendon, intestine and intervertebral disc - will demonstrate that these tissues are constructed from a small number of macromolecular "building blocks". These macromolecules are organized into hierarchical structures that are anisotropic fiber composites. Their widely varying mechanical functions are derived from the specific composite architectures that make each tissue unique.

The structural levels at which the properties are resolved occur at a size scale that can be elucidated using the techniques of modern materials science. The relationships between composite structure and mechanical behavior in both reversible and damage regimes of deformation can be understood by using established modeling methodology. Subsequently, from this type of understanding, it seems reasonable to suggest that new concepts and designs in advanced synthetic composites can evolve from the science of these biocomposites.

STRUCTURE-PROPERTY RELATIONSHIPS OF MULTILAYER COMPOSITES

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ABSTRACT

Physical and mechanical properties of multilayer composites having 49 or more alternating layers of polycarbonate (PC) and styrene acrylonitrile (SAN) were studied. Under dynamic mechanical testing, a unique damping peak with a log decrement intensity of approximately one was observed at a temperature (128-130°C) which is intermediate to the Tg's of the constituent polymers of PC (150°C) and SAN (104°C). Variations in material and experimental parameters had no effect on the occurrence of the intermediate peak--only the disruption of the continuous nature of the layer structure effectively eliminated this novel transition. The origin of this transition will be discussed.

The mechanical properties of PC/SAN composites were studied in terms of the composition ratio and total number of layers. The impact strength was found to increase monotonically with the volume fraction of PC as well as number of layers even if the total thickness of the composite was held constant. Under the tensile test, changes in mode of fracture were noted, again with the variation of composition and number of layers. Suppression of SAN crazing and cold drawing of the entire composite was also possible when the SAN layers were sufficiently thin. Physical reasoning behind these behaviors will be discussed.

HIERARCHICAL STRUCTURE IN CRYSTALLINE POLYMERS

H. D. Keith, AT&T Bell Laboratories

The basic unit in a semicrystalline polymer is a lamellar crystal composed predominantly of folded molecular chains. Depending upon the particular polymer and the manner in which it is crystallized, these lamellar crystals are capable of organization into a wide variety of morphological forms. Properties are influenced significantly by details of this organization ranging from the molecular level to virtually a macroscopic scale. Structures and their influence upon properties will be reviewed with emphasis upon present understanding of how the structures are formed and how their variations are reflected in macroscopic behavior. Much of the thinking in the field is currently being challenged in the light of recent evidence. New trends in interpretation will be discussed with particular emphasis upon molecular mechanisms involved in the growth of lamellar polymer crystals and upon how spherulitic structures develop.

NMR Studies of Molecular Motion in Solid Polymers

J. R. Lyerla
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Recent advances in nuclear magnetic resonance (NMR) spectroscopy afford onportunity to examine details of molecular motion in solid polymers. In this presentation, we report on variable-temperature, magic-angle spinning carbon-13 NMR, deuterium NMR, and proton NMR studies of motions in amorphous and semi-crystalline polymers. Spectral data and spin-lattice relaxation data an polycarbonate, polypropylene, and aromatic polyesters are utilized to discuss mainchain and sidechain motions in these systems.

Ultrasonic Study of Fracture Process in Amorphous Polymers

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Various ultrasonic techniques were developed and utilized to study the mechanical deformation and fracture processes in amorphous polymers.

The three sensors method for AE (acoustic emission) measurement was developted to detect very weak AE signals from polymers under tensile deformation process. It was found for a ductile polymer of polycarbonate that there exists a correlation between the deformation mechanism and the AE characteristics, such as a wave form, duration of AE signals and distribution of AE sources in the tensile specimen. In the case of brittle polymers such as polymethyl methacrylate (PMMA), only few AE events were detected upon the formation of submicrocracks. Furthermore, this AE technique was utilized to confirm the intermittent crack propagation phenomenon observed for a SEN (single-edge notched) PMMA sample during the fracturing process under high pressure.

Simultaneous measurements of AE and high speed shadow optical photographs revealed that the density of the parabolic marking, which appears on the fracture surfaces of SEN-PMMA, has a closer correlation to the amplitude of the stress wave emitted during crack propagation, rather than to the values of crack speed or stress intensity.

Precise measurements of ultrasonic velocity of PMMA under tensile stress condition showed the lowering of the elastic modulus in both the directions parallel as well as perpendicular to the tensile direction. This softening phenomenon in MHz region was further confermed by Brillouin scattering in GHz region, and was proven by small angle X-ray scattering experiments to be attributed to the formation of submicrocracks.

Other techneques are also introduced, such as the ultrasonic spectroscopy, which can measure simultaneously the ultrasonic velocity and absorption in a wide frequency range by the Fourier transformation analysis of a single pulse and be utilized to detect inhomogeneous structure inside polymeric materials, and the ultrasonic fractography, by which the precise and local two-dimensional measurements of crack velocity can be performed.

ON THE RHEOLOGY AND PHASE BEHAVIOR OF POLYMER LIQUID CRYSTALS

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The phase behavior and rheology of polymer liquid crystals have been reviewed with an eye to control of structure-property relationships. Their unique and remarkable rheology is discussed. The control of phase behavior and interreaction is shown to be possible as illustrated with copolyesters.

Features will be illustrated with the phase diagram developed for poly(bisphenol E-isophthalate-co-naphthanate) in a parasubstituted phenyl ester of terephthalic acid, di- p-methylbenzoate terephthalate. This binary system exhibits miscibility in a nematic phase and does not form cocrystals. The crystal to nematic phase transition has been characterized by the temperature and heat of transition data and by the Flory-Huggins theory for the melting point depression of a polymer-diluent system.

The phase behavior was examined at high copolyester content with three different molecular weights of BPE/I/N20. These blends were subsequently transesterified in the solid state at 220°C and the change in phase transitions are reported. The phase behavior and transesterification were characterized by DSC, TGA, polarized light microscopy, x-ray diffraction, and dilute solution viscometry. The thermodynamic criteria for the melting point depression and the kinetics of transesterification are also reported for the utilization of this new processing technique.

MORPHOLOGY AND PROPERTIES OF NATURAL CELLULOSIC FIBERS

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Natural cellulosic fibers have in common the fact that cellulose is the key polymeric component in the structure, although the chemical composition varies widely depending on the specific source of the fibers. Cotton fibers, for example, are nearly all cellulose, while wood fibers may typically be composed of 50% cellulose, 25% other polysaccharides collectively known as hemicellulose, and 25% lignin, an amorphous complex polymeric substance containing aromatic residues. Cellulose is a long-chain linear condensation polymer of β-D-glucose with three free hydroxyl groups on each monomeric unit. These groups provide reactive sites for chemical modification reactions and the opportunity of extensive and strong inter- and intramolecular hydrogen bonds. Because of the hydrogen bond network, and also due to restricted rotation around the polymeric 1,4-β-linkage, cellulose is a rigid and stiff chain with a T_g well above the chemical decomposition temperature.

Despite the high T_g, native cellulose is invariably highly crystalline as a result of the biosynthetic process. In naturally occurring cellulosic fibers, the cellulose crystallites are invariably aggregated into fibrils which constitute the underlying building blocks of the fiber. In cotton, the fibrils are laid down during the development or growth of the fiber in the form of concentric layers. The fibrils are disposed at an angle of 23° with respect to the fiber axis and thus they describe a helical pattern. The sense of the helix reverses frequently along the length of the fiber. This morphology is unique to cotton; other cellulosic fibers such as ramie and jute have similar fibrillar structures, but fibrillar angles in the 5 to 10 degree range with no reversals. Wood fibers, on the other hand, are structually more heterogeneous and may be considered as composites. The individual cells, which also have a fibrillar substructure, are embedded in the lignin, although some lignin is distributed also within the cell wall structure.

The mechanical properties of naturally occurring cellulosic fibers reflect their complex morphologies. Despite the rigidity of the cellulose chain, fibers such as cotton are quite ductile and resilient due to the fibrillar orientation and the helical reversals. Fibers such as ramie and jute are less extensible, reflecting their small fibrillar angles and lack of structural reversals. The mechanical behavior of wood fibers is complex in view of their structural heterogeneity and composite-like morphology.

List of Attendees

Ninth Asilomar Conference

February 9-12, 1986

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