

ORAL CONTRIBUTIONS

OC001

MORPHOLOGY AND PROPERTIES OF SILICONE RUBBER WITH SUPERCRITICAL CARBON DIOXIDE AND ALTERNATING MULTILAYERED CO-EXTRUSION

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Introduction: The properties of silicone rubber foams are depended on the cell structure, such as cell morphology, cell distribution and cell density. Moreover, the cell structure of foams is often determined by the foaming conditions and processing methods. Nevertheless, the most cells in silicone rubber foams are uneven and disordered in the two-dimensional or three-dimensional, meanwhile, it is difficult to obtain microcellular silicone rubber foam with the conventional techniques.

Methods: In this work, processing conditions on the cell morphology of microcellular silicone rubber foams are investigated supercritical CO₂ foaming technology in order to decrease the cell diameter and improve the cell structure. And the silicone rubber foaming sheet is designed as alternating multilayered structure of foaming layers with chemical bubbling method and film layers through the multilayer co-extrusion system.

Results: The silicone rubber foams with the cell diameter of 10 μm and cell density exceeding 10⁸ cells/cm³ were achieved by using supercritical CO₂. The density of foam could be changed by adjusting the pre-crosslink degree and saturation pressure. The foamed silicone rubber and unfoamed silicone rubber were designed as foaming layers and film layers, respectively. The cell structure changed from spheres to rods and saucer shape, with increasing layer number. The compressive response of the foaming silicone rubber showed anisotropic characteristics, and the compressive performance of parallel to layer interface direction was better than that of perpendicular direction.

Discussion: The preparation of silicone rubber foams with microcellular and multilayered structure provide a novel and convenient method for the functional elastomer foams.

Keywords: silicone rubber, foams, multilayered structure, supercritical CO₂.

RHEOLOGICAL CONTROL AND 3D-PRINTING OF SILICON RUBBER-BASED CELLULAR ARCHITECTURES

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Introduction: Creating lightweight materials with desired mechanical properties has long been an engineering pursuit, and the rise of three-dimensional (3D) printing techniques has made the fabrication of lightweight, porous materials with tailored architectures not only possible but also feasible. A variety of cellular materials based on plastics, metals and ceramics with engineered mechanical responses (such as mechanically metamaterials) was created, but 3D printing of cellular rubbers is still a challenge. Here, a 3D direct ink writing technique was adopted to print a viscoelastic silicon rubber/nano silica system into orderly arranged sub-millimeter struts. Unique compressive stress-strain behaviours have been demonstrated in these specially designed cellular architectures.

Methods: The materials were purchased from Chongqing Polycomp International Corporation (China). They were compounded at a given ratio using a Thinky planetary mixer for 15 min at 2000 rpm and centrifugated for 15 min at 2000 rpm for de-aeration. 3D printing was realized using a fluid dispenser (808GX, MUSASHI, Japan), which was programmed using a dispensing robot (shotmaster 350PC-GX, MUSASHI, Japan) with control software. Rheological measurements were performed on an ARES-G2 stress-controlled rheometric system with a parallel-plate geometry. Mechanical properties were measured using a DMA RSA-G2 machine according to ISO 527-4.

Results: By controlling the size, content and interface of nano silica, the rheological behaviour of silicon rubber/nano silica composite could be tailored, and a viscoelastic system with a solid-like rheological behaviour ($G' > G''$) at low oscillation stress and a liquid-like rheological behaviour ($G'' > G'$) at high oscillation stress could be obtained. At a stress higher than the crossover of G' and G'' in the dispensing nozzle, the liquid-like composite could flow and be printed onto the substrate as a strut. On the substrate, the stress in the nozzle is released, and the composite shows a solid-like behaviour without a change of its morphology, so the printed strut could be “solidified” on the substrate. In this way, the system is printable and 3D-printed silicon architectures could be prepared. Typical structures and properties of the architectures are shown in Figure 1. Obviously, the architectures could be greatly varied. By manipulating the architectures of the printed 3D patterns, materials with unique stress-strain behaviour, such as mechanically self-adapting materials with a reversible compressive strain softening behaviour, and quasi-constant modulus materials with a linear compressive stress-strain behaviour, could be obtained.

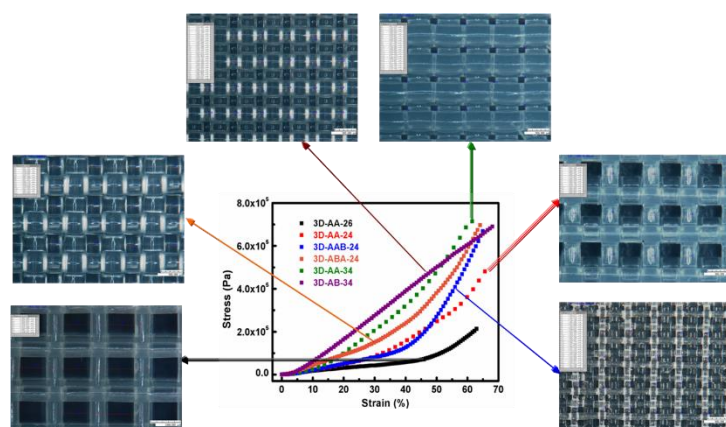


Fig. 1 Typical structures and compressive properties of the printed cellular architectures.

Discussion: By tailoring the rheological behaviour of the liquid silicon rubber/nano silica system via filler network control, cellular rubbers with specially designed architectures could be 3D-printed, and several kinds of materials with unique mechanical responses were achieved. This work not only paves a feasible way to control the porous structure of elastomers, but also suggests the ability to tailor mechanical properties of cellular materials via micro-patterned design, which may find potential applications in emerging fields or replacement of traditional foams.

Keywords: 3D Printing, Elastomer, Cellular Solids, Stress-Strain Behaviour

MECHANICAL PROPERTIES AND MORPHOLOGIES OF RECYCLE-PC / RECYCLE-PET BLENDS FABRICATED BY HIGH-SHEAR ROTATIONAL PROCESSING

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Introduction: Currently, the recycling of plastic materials such as general-purpose resin and engineering plastics has been carried out extensively. However, mechanical properties of plastic materials were gradually decreases by repeating adds heat history on reuse. Therefore, it is often inexpensively provided to low grade products. On the other hand, polymer blends and blends with various polymer materials have been studied to assess their performance, which transcends that of single polymers. Nevertheless, depending on the selected materials and their combinations, they can be immiscible. Sometimes, their physical properties become worse. In this study aims to development of novel polymer blends used recycle PC and Recycle PET fabricated by high-shear rotational processing.

Methods: Recycle-Polycarbonate (R-PC) was used (S3000; Aita shokai inc.) with recycle-poly (ethylene terephthalate) (R-PET) (R-100; Aita shokai inc.) was used. The R-PC and R-PET blending ratio were 9:1, 8:2 and 7:3 (wt). We used a high-shear rotational processing machine (NHS2-28; Niigata Machine Tecno Co., Ltd.) for melt mixing of the R-PC/R-PET blends. We conducted melt mixing at 280 °C. Screw rotation speeds were 0, 500, 1000, and 1500 rpm. The 0 rpm is low shear product by a single screw. Mixing times were 10 s and 30 s. After high-shear processing, dumbbell specimens were molded using hot press machine. Thermal properties were measured using a M-DSC (Q100; TA Instruments). Morphology was observed using a TEM (JEM2100F; JEOL Ltd). Tensile tests were measured Strograph (VGS-E01; Toyo Seiki Seisaku-Sho Ltd.).

Results: The results of M-DSC measurement were shown respectively in Fig .1. TEM images of R-PC/R-PET = 7:3 were portrayed in Fig .2. The stress-strain curves in tensile tests are depicted in Fig .3.

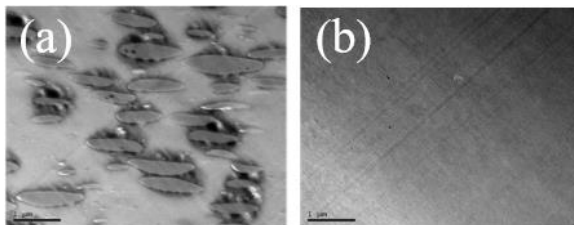


Fig .2 TEM image of R-PC/R-PET = 7:3
(a) 0 rpm, (b) 1000 rpm/10 s.

Discussion: M-DSC measurement results confirmed that T_c of R-PET in R-PC/R-PET was shifted to the high temperature side and T_m was shifted to the low temperature side. The shift amount increases with increased screw rotation speed. These results suggest that the R-PET domains were dispersed similarly to islands in the R-PC matrix. They were finely distributed by high shear processing. TEM images of R-PC/R-PET = 7:3 shows the R-PET domain diameter in R-PC/R-PET was about 1 μ m in the mixed condition of 0 rpm. The R-PET domain under the mixing condition of 1000 rpm/10 s was not observed. As a result, it was found that the R-PC and R-PET were compatibilized. The stress-strain curves in tensile tests are depicted in Fig .3. The R-PC/R-PET = 9:1 and 8:2 fabricated under mixed condition of 1000 rpm/10 s were lower breaking strain than that of neat R-PC. The R-PC/R-PET = 7:3 fabricated under mixed condition of 1000 rpm/10 s was higher breaking strain than that of neat R-PC.

Keywords: Polymer blends, compatibilizing, High-shear rotational processing

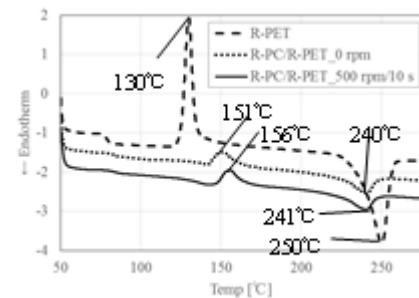


Fig .1 M-DSC measurement results.

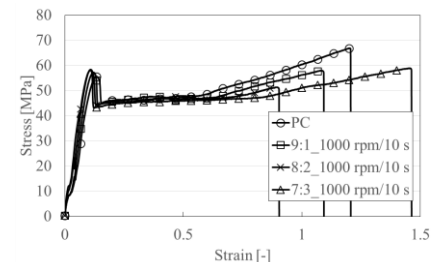


Fig .3 Tensile test results.

CONTROL OF PHASE SEPARATION STRUCTURE IN POLYMER BLENDS FABRICATED BY HIGH-SHEAR PROCESS

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Introduction In general, polymer blends are well known for using as functional materials in the various industrial fields. However, demand for high performance of polymeric materials was dramatically increased which related the research and innovation growth. The polymer alloy is the latest of the state of the art technology that greatly extend the performance capabilities of polymer blends. In the fabrication method, the control of phase separation of the structures is necessary to realize.

The purpose of this research was focused on the control of polymer blend internal structures and also the development of the saving materials weight and low cost materials used. The polyamide6 (PA6) having a high mechanical property was blended with polypropylene (PP) having light weight and inexpensive using the novel high speed rotational or high-shear process. The effect of viscosity ratios on internal structures and mechanical property of the novel polymer alloy were investigated. The internal structure was controlled by rotational speed and melt mixing time.

Methods

1. Materials The material used in this experiment are three PA6 grades are T852 (MFR:5 g/10 min), T802 (MFR:26 g/10 min) and T840 (MFR:80 g/10min) (TOYOBO CO., Ltd) and three PP grades are J105 (MFR:9 g/10 min), J108 (MFR:45 g/10 min) and S119 (MFR:60 g/10 min) (Prime Polymer CO., Ltd) and three compatibilizing agents PP-g-MAH Yumex1010 (Sanyo Chemical Industries, Ltd), Admer QE800 and Admer AT2606 (Mitsubishi Chemical Co., Ltd). PA6/PP ratio 70/30 (w/w %) were used, the PP-g-MAH were added 5.0 w/w % of the blended resins.

2. Experiment High shear processing machine (NHSS2-28, Niigata Machine Techno Co., Ltd.) were used for polymers blending process. The design of the internal feedback screw is shown in Fig.1.

High-speed rotation (Max: 3,000 rpm) is a novel of molding process using the extensional flow at the time of the feedback flow in the hole passage of internal mixing and screw for blending technique. In this study, the blending temperature of 230 °C, rotation speed of 500 and 3,000 rpm, mixing time of 10 and 30 s.

The blended samples were molded using micro-injection molding machine (AU3E-s, Nissei Plastic Industrial Co., Ltd.) and that were followed by fabricating small dumbbell specimen (JIS7162 1BB).

3. Characterization The tensile test (Strograph VGS-E01, Toyo Seiki Seisaku-sho, Ltd.) was carried out by tensile speed of 5 mm/min. The specimen internal structures were observed by using SEM (TM1000, Hitachi High-Technologies Corp.).

Results and Discussion The results of the SEM images and tensile test result of blending materials which obtained by high shear-process were shown Fig.2 and Fig.3. By the results, it can be confirmed PP domain size was decreased from about 20-40 μm to 10-20 μm by decreasing the PA6/PP MFR ratio 80:45 to 80:60 (See Fig. 2 (a, b)). Moreover, adding the PP-g-MAH obtained PP nano-domain less than 2 μm , it was indicated that PP-g-MAH promote the fine dispersion as Figure 2 (c). The nano-dispersion of PP in PA6 improve the elongation and toughness due to the uniform of phase structure lead the deformation difficulty.

Keyword: Polymer alloy, High-shear processing, Morphology

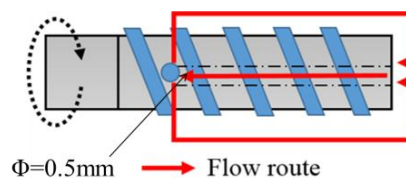


Fig.1 Schematic diagram for screw used in the high shear rotational mixer.

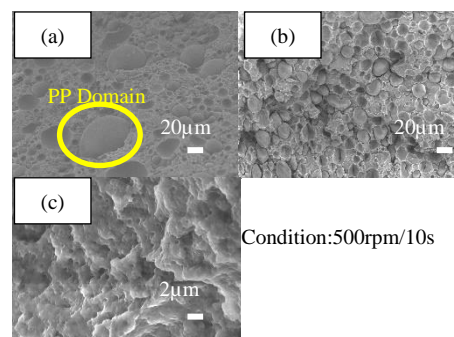


Fig.2 The SEM images of (a) T840/J108 (MFR=80:45), (b) T840/S119 (MFR=80:60) and (c) T840/S119/Yumex 1010

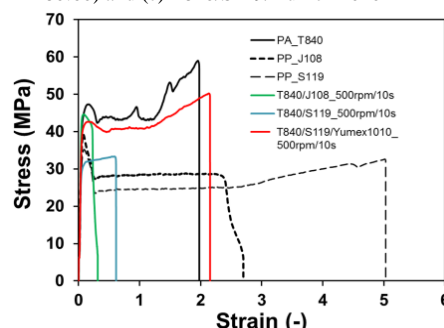


Fig.3. Tensile property of PA6, PP and PA6/PP bending materials.

EPOXY-ANHYDRID BASED THERMOSETS REINFORCED BY INORGANIC NANOPARTICLES: EFFECTS ON SHELF-LIFETIME, THERMAL AND THERMO-MECHANICAL PROPERTIES

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The increasing use of nanoparticles as performance-enhancing fillers in resins for industrial applications has stimulated research for the stable dispersion of nanoparticles in thermosets. The modification of the interface between the particles and surrounding resin matrix is a key strategy to ensure compatibility of the particles with the polymer matrix and to avoid aggregation of nanoparticles. However, surface modification of the nanoparticles can have significant effects on the shelf lifetime of the resin as well as on the thermo-mechanical properties of the final composite.

In this work, inorganic nanoparticles, in particular silica and aluminium oxide nanoparticles with different functionalization of their surface were embedded in an epoxy/anhydride system and the impact on the stability and the shelf life time of the non-cured resin was determined. Furthermore, investigations on the effect of nanoparticles on thermo-mechanical as well as thermal properties and the curing kinetics of the thermoset were performed.

Characterization of the non-cured system was done by rheological investigations and infrared spectroscopy. To characterize the thermo-mechanical properties as well as the curing behavior different analytical methods, such as differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA) and infrared spectroscopy were employed. Moreover, electron microscopy images show the distribution of the modified nanofillers within the polymer matrix.

The different properties of the nanocomposites compared to those of the neat resin can be correlated with the presence of nanoparticles and interactions of the polymer with the dispersed particles. Noteworthy, the stability and shelf lifetime of the nanodispersions are dominated by the interface. Differences in mechanical and thermal properties become more pronounced with the addition of nanoparticles to the polymer matrix; this is due to the altered curing behavior of the epoxy/anhydride resin upon addition of nanoparticles.

This research work was performed within the K-project "PolyComp" at the Polymer Competence Center Leoben GmbH (PCCL, Austria). Funding is provided by the Austrian Government and the State Government of Styria.

MODIFIED SISAL FIBERS CARBON AS CATHODE MATERIALS FOR LITHIUM-ION BATTERY

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Introduction: With the increasing of environment concerns and the development of electronic devices, there is a growing demands for the lithium ion batteries with high power and energy density. However, the graphitic carbon which is widely used for the anode materials only have a theoretical specific capacity of 372 mAhg⁻¹, which can not meet the demands of high specific capacity lithium ion batteries. So it is necessary to develop green and high specific capacity anode materials for lithium ion batteries.

Methods: A kind of microporous sisal fiber carbon (SFC) was prepared by carbonization of sisal fiber under nitrogen protection at high temperature and then modified by hydrothermal method under different conditions. The structure and morphology of the product were characterized by XRD, SEM and Raman spectra. The electrochemical properties of the product were characterized by CV and constant current charge-discharge and so on. The hydrothermal modified conditions such as temperature, acid/alkaline agents and etc. on the electrochemical properties of SFC were discussed.

Results: (1) Modified SFC with hydrothermal temperature at 140°C had a significantly higher capacity than that of other temperature, (2) SFC displayed higher capacity when hydrothermal modified in acid solution than in deionized water and alkaline system, (3) SFC modified in hydrothermal system exhibited a significant change in microstructure.

Discussion: Hydrothermal treatment is an effective method for SFC and other biomass carbons to improve their electrochemical performance.

Keywords: sisal fiber carbon; hydrothermal treatment; anode material; electrochemical performance

ENGINEERING PLASTIC COMPOSITES REINFORCED BY NOVEL HEATPROOF CELLULOSE NANO-FIBER

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Introduction: Cellulose nanofibers (CNFs) have many useful properties, including high strength and low thermal expansion, and are also environmentally friendly, readily renewable, safe, and biodegradable. But the conventional CNF could not stand the heat while polymer processing such as melt compounding and injection molding, especially in engineering plastic matrixes. Recently, novel chemical modified pulp endowed high heat resistance, compatibility with thermoplastics and fibrillation ability while compounding was developed. In this study the CNF reinforced engineering plastics which is impossible to materialize by conventional technology was evaluated.

Methods: Thermogravimetric analysis (TGA) for chemical modified pulps, bending test for composites consisted of engineering plastics and the chemical modified pulps, and observation for cellulose fiber in engineering plastic matrixes were done.

Results and discussion: The reduction temperature of 1wt% of the pulp from TGA was improved more than 50°C, and the improvements was different according to degree of chemical treatment. Fig.1 shows bending modulus and strength of the CNF/polyamide6 composites as a function of degree of hydroxyl group substitution (DS) in cellulose. The maximum bending modulus of composites was 5400MPa which is 2.6times of neat PA6, and the maximum bending strength was 160MPa which is more than 68MPa from neat PA6. Interestingly, the impact strength of composites was same as neat PA6, although the composites exhibited high bending properties. Fig.2 shows SEM photograph of the CNF extracted from PA6 matrix by solvent extraction. The structured CNF in the best composites was quite fine, long, and also network structure was observed. The size was from tens to hundreds of nano-meter. The nano and high aspect ratio fibers was formed while polymer processing owing to inhibit cellulosic hydrogen bond by chemical treatment. And the CNF must contribute the high bending properties with keeping impact strength.

Keywords: Cellulose, Nanofiber, Pulp, Chemical treatment.

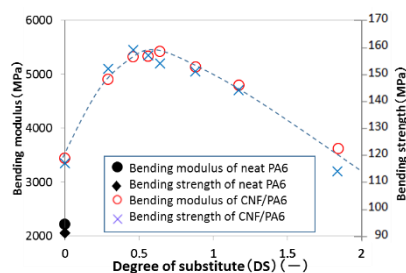


Fig.1 Bending modulus and strength as a function of DS.

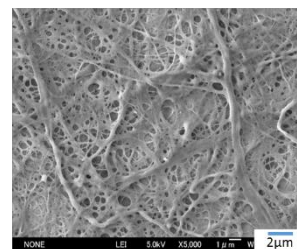


Fig.2 SEM observation results of CNF extracted from composites.

INTERFACIAL PROPERTIES OF LDPE/UHMWPE SINGLE-POLYMER COMPOSITES PRODUCED BY EXTRUSION-CALENDERING PROCESS

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Introduction: Single-polymer composites (SPCs), composed of the matrix and reinforcement with same chemical compositions, get the advantages of light weight, high specific strength, easy recyclability. The methods of preparing SPCs have been reported such as the hot compaction, film stacking, powder impregnation, injection molding, etc. However, the traditional processing methods were not easy to achieve continuous production of industry yet restricted the development of SPCs. In this research, a process of extrusion-calendering was used to realize continuous production of single-polymer composites (SPCs). Low density polyethylene (LDPE) was used as the matrix, and ultra-high molecular weight polyethylene (UHMWPE) fabric was used as the reinforcement. The effect of die temperature on the interlayer bonding strengths was mainly investigated.

Methods: The UHMWPE fabric coated by LDPE melt in a specially designed die, and then they were moved out and passed through the calender, the LDPE/UHMWPE SPCs could be finally prepared. The theoretic temperature range of 145-160 °C was chosen. The interfacial properties of the SPCs were evaluated through T-peel tests. Scanning electron microscopy was also used to determine the fiber-matrix interfacial adhesion and the effects of different die temperatures.

Results and Discussion: Fig. 1 reveals that the interfacial strength increased with temperature raised. The 160 °C achieving an average value of 22.4 N/10mm, but the consecutive T-peel test could not be performed thoroughly in virtue of the sample breaking off in the middle. In Fig. 2, with the temperature raised, there is a surface roughness increasing of the fabric where the fiber bundles shrink and become tight. Fig. 2a shows many fibrils are attached to the fabric surface but fiber bundle maintains the original structure. From Fig. 2b, it is not easy to observably distinguish the single filament since they almost were melted to form a whole, and there is a continuous sheet layer of matrix in the gap of fiber bundles, which directly leads to the further improvement of the interlayer bonding strength. As the surface has been badly damaged (in Fig. 2c) so that the vestiges left after stripping nearly throughout the entirely surface of fabric, such a strong bonding seriously impeded the T-peel testing.

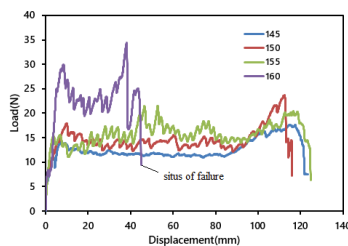


Fig. 1 Peel load as a function of die temperatures

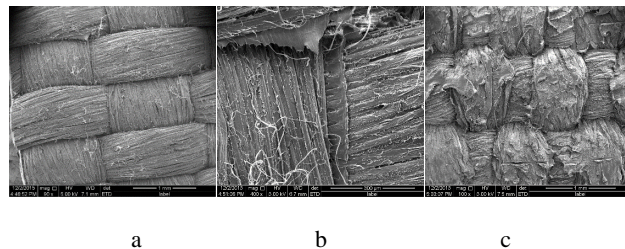


Fig. 2 Peel fracture surfaces of specimens manufactured at (a, b) 150 °C, (c) 160 °C.

Keywords: low density polyethylene (LDPE); ultra-high molecular weight polyethylene (UHMWPE); single-polymer composites (SPCs); extrusion; calendaring

OC009

AN IMPROVEMENT OF GAS TRANSMISSION RATE OF PLA BY STEREOCOMPLEXATION OF PLLA/PDLA-PEG-PDLA

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In this research, we improved the gas transmission rate of PLA by adding modified microporous or hydrophilic materials such as polyethylene glycol block copolymer. PDLA-PEG-PDLA triblock copolymer was prepared *via* ring-opening polymerization in bulk process using tin octoate as a catalyst. Poly(ethylene glycol) with number average molecular weight of 2000 and 6000 g/mol were used as macro-initiator. Two compositions of lactyl unit (L): oxyethylene (EO) including 2:1 and 5:1 were prepared. Stereocomplexes of PDLA-PEG-PDLA and PLLA were prepared by using twin screw extruder at 180°C and fabricated into tubing with thickness of 30 µm in order to improve gas permeability and mechanical properties.

Gas transmission rate results showed that an addition of triblock copolymer with various PDLA/PEG ratios could significantly enhance the O₂ and CO₂ permeability of polymer film. This was probably due to hydrophilicity of the PEG facilitated the permeability of those gases. Tensile strength and modulus of PLLA/PDLA-PEG_{6000(2:1)}-PDLA stereocomplexes were 21% and 4% higher than PLLA with porous material, respectively. Elongation at break of all stereocomplex was slightly higher than that of PLLA and PLLA with porous material. These stereocomplex films can potentially be used as fruit bag for mangos' quality enhancement during growth.

Keywords: triblock copolymer, gas permeability, stereocomplex

AN ADVANCED PREPARATION AND CHARACTERIZATION OF THE PET NANOCOMPOSITES WITH VARIOUS ORGANOMODIFIED LDH AS CATALYSTS

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Various polyethylene terephthalate (PET)/ sulfanilic acid salt-modified layered double hydroxide (LDH-SAS) nanocomposites were prepared via *in situ* interlayer polymerization of bis-hydroxy ethylene terephthalate (BHET). Hydrothermally synthesized modified LDH applied for nanocomposite synthesis. The prepared organomodified LDHs were characterized by different methods, including X-ray diffraction, differential scanning calorimetry, thermogravimetric analysis, and particle size analysis. After that *in situ* synthesized PET/LDH-SAS nanocomposites were characterized by diverse methods, including X-ray diffraction, transmission electronic microscope, thermogravimetric analysis, intrinsic viscosity measurements. The results of the study revealed that the PET/LDH-SAS nanocomposites possess a better state of nanolayer dispersion, thus making it easier for the oligomer to enter the gallery of LDH-SAS layers. Thus, the polymer chains can be included between the LDHs layers during the polymerization of the polymer matrix. The gas barrier, mechanical properties, as well as better thermal properties as compared with the pristine PET. Moreover, the PET/LDH-SAS nanocomposites showed higher crystallization temperature, degree of crystallinity, and maximum degradation temperature than that of the pristine PET.

Keywords: Polyethylene terephthalate, Layered double hydroxide, Polycondensation, Hydrothermal, Nanocomposites.

OC011

PRECURSOR DEVELOPMENT FOR HIGH PERFORMANCE AND LOW COST CARBON FIBRES

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Introduction:

Carbon fibre (CF) is currently undergoing a global renaissance, due to a new range of applications for CF, particularly for the automotive, aerospace and wind energy sectors, and due to the high growth rate in capacity anticipated over the next ten years. The key technical issues that need to be addressed at present are improving the fibre quality and performance, and reducing production cost. It is widely recognised that CF precursor composition and quality determine the ultimate performance of carbon fibres, and also have a significant impact on processing properties and production cost. Polyacrylonitrile (PAN) based precursor is by far the predominant precursor material used globally for commercial production of CF. Our research has therefore focused on the development of new generation of PAN based precursors to address the outstanding quality, performance and cost issues. This presentation reports on the most recent precursor development at CSIRO, which involves a well-defined PAN precursor with a high molecular weight and low polydispersity index for improved CF performance and quality, and a new PAN copolymer that enables low cost processing of quality CF.

Methods: Well-defined PAN precursor polymers with a high molecular weight and low polydispersity index were prepared in a pilot scale by RAFT polymerisation. They were then processed into the precursor fibres by solution spinning in organic and inorganic solvent systems respectively. A new PAN copolymer containing a bifunctional comonomer component was also developed for low cost CF processing. These precursor polymers and fibres were characterized. Comparative studies were also carried out for assessing the properties and performance of the new precursors against their conventional controls.

Results: Comprehensive characterisation results on the new precursor polymers and fibres will be presented, and compared with those of the conventional control polymers and fibres.

Discussion: New and advanced carbon fibre precursors were developed and evaluated. The results of this study demonstrated significant advantages of the new precursors over the conventional controls in terms of their processing properties, fibre quality and/or cost benefits.

Keywords: carbon fibre, precursor, polyacrylonitrile, RAFT, comonomer

TERPENE BASED CHEMICALS: (1) BIO-AROMATICS TO PRODUCE KNOWN MONOMERS -UTILISING PYROLYSIS AND (2) FUNCTIONAL MONOMERS FOR IMPROVED POLYMER PERFORMANCE

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Introduction: Valuable chemicals and polymers modifiers have been produced utilising the cyclic terpenes feedstock's provided by nature. These terpenes (pinene and cineole) have been sourced from woody biomass (pine and eucalypt) sources. We have been able to utilise efficient pyrolysis and downstream chemical conversion to produce a range of aromatic chemicals including 100% Bio-PET. We have also utilised chemical and biocatalysis (fermentation) to produce functional monomers that have been used as modifying monomers for known polymers or to produce novel polymers. Applications for the functional terpene include coatings, resins, plasticisers and engineering polymers with improved mechanical, thermal properties etc

Methods: To produce the Bio-Aromatics we have used gas pyrolysis aided by the use of metal doped gamma alumina catalysts. This predominately produced p-cymene. The p-cymene has been oxidised to produce terephthalic acid which has in turn be used to produce Bio-PET. For the functional terpenes we converted alpha-pinene to produce 2,6 dihydroxy cineole (DHC). This monomer has been utilised in a range of different polymers: Polyesters (PET), Polyurethanes, polycarbonate, high temperature acrylics and to produce plasticisers for PVC

Results and Discussion: Bio-Aromatics: p-cymene intermediate "aromatic platform chemical" was produced from a range of terpene feedstocks (including mixed feeds). Conversion of cineole to p-cymene has been achieved at 95%+ conversion and 100% selectivity. From the p-cymene we have been able to produce good quality 100% Bio-PET (utilising MEG produced from sugar).

Functional Terpene Monomers: DHC modified PET with improved thermal properties ($T_g > 150C$ when replacing MEG with DHC) has been produced. This could have applications in stiffness and hot fill properties for PET based container applications. Fatty acid esters of DHC as plasticisers for PVC have shown to perform as well as DEHP/ DOP (Di-Octyl phthalate:which is under considerable regulatory pressure) DHC and related monomers have been attached to methacrylic acid. The resultant polymers we shown to be tough, clear and scratch resistant with improved thermal properties ($T_g ! 160C$ and $T_m \sim 295C$)

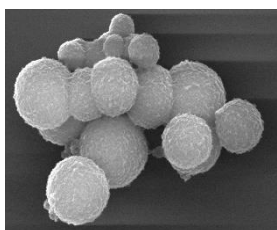
Keywords: Renewable Feedstock's, Sustainability, Biomass, Terpene, Cineole, PET, Polyester, PVC, Polycarbonate, Polyurethane

POLY (MELAMINE-FORMALDEHYDE) MICROCAPSULES FOR SELF HEALING POLYMER COMPOSITES

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In the development of self healing composite through microencapsulation approach, control of capsules shell wall thickness is a key issue because it influences the self healing performance due to following reasons - the microcapsules walls must be resistant to diffuse out of healing agent, capsules with thin walls may leads to loss of core material during storage and causes difficulties in handling during processing of the composites. In this article, we investigated systematically the effect of melamine to formaldehyde ratio taken in the initial feed on the encapsulation efficiency, core content, shell wall morphology, the mean particle size distribution and thermal stability of the microcapsules. Epoxy loaded poly (melamine-formaldehyde) (PMF) microcapsules were prepared by in-situ polymerization using emulsion technique. Structural (FTIR and $^1\text{H-NMR}$), morphological (OM and SEM), particle size distribution and thermal (TGA & DSC) characterization was done to investigate the effect of melamine to formaldehyde ratio on the microcapsules. Free flowing; white coloured, odourless microcapsules with density 1.202 g/cm^3 were obtained by in situ polymerization. Encapsulation efficiency decreased as the formaldehyde content increased. FTIR and $^1\text{H-NMR}$ spectra showed the presence of epoxy as a core material. OM & SEM micrographs showed that the capsules are almost spherical with a perfect periphery, but have a tendency to agglomerate. OM & SEM showed that the capsules synthesized with a lower formaldehyde amount possess the rough, porous and uneven morphology Both OM and SEM analysis revealed that with increasing formaldehyde content the thickness of shell wall increases. Particle size distributions of microcapsules were found in the range of 0.1 to $1000 \mu\text{m}$. The mean particle size distribution of microcapsules decreases (from 83 - $66 \mu\text{m}$) as the formaldehyde ratio increases. Thermal analysis showed that the capsules were thermally stable up to 300°C . Thermal stability increased as the ratio of melamine to formaldehyde increases.



Optimized microcapsules

Keywords: microcapsule, epoxy resins, poly (melamine-formaldehyde) (PMF)

NOVEL THERMOPLASTIC CELLULOSE DERIVATIVES: STRUCTURAL DESIGN, SYNTHESIS, AND PROPERTIES

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Melting processing of cellulose is of special interest, however, it still remains a great challenge, because cellulose has extensive hydrogen-bonded and partially crystallized structure that makes it not melt-processible. Graft copolymerization is a practical way to alter the physical and chemical properties of cellulose. We developed an effective method for grafting L-lactide (LLA) from unmodified cellulose by ring-opening polymerization under homogeneous conditions. By using 4-dimethylaminopyridine (DMAP) as an organic catalyst, cellulose-graft-poly(L-lactide) (cellulose-g-PLLA) copolymers with a molar substitution of PLLA (MSPLLA) in a range of 0.99-12.28 were successfully synthesized in an ionic liquid, 1-allyl-3-methylimidazolium chloride (AmimCl). The amount and length of grafted PLLA chains in cellulose-g-PLLA copolymers could be well controlled by adjusting the molar ratios of LLA monomer to cellulose. When content of grafted PLA in copolymers is above 60wt%, the cellulose-g-PLLA copolymers exhibited thermoplastic behaviour, indicating the intermolecular and intramolecular hydrogen bonds in cellulose molecules had been effectively destroyed. By using a conventional thermal processing method, fibers and disks of cellulose-g-PLLA copolymers were successfully prepared. Using AmimCl as the reaction media, we also synthesized cellulose acetate-graft-PLLA (CA-g-PLLA) and cellulose propionate-graft-PLLA (CP-g-PLLA) copolymers by first acylation of cellulose with corresponding acylation reagents, and followed by ring-opening polymerization of LLA monomer initiated from residual hydroxyl groups in cellulose esters. These two copolymers with proper substituted acyl group content and amount and length of grafted PLLA chains also exhibited thermoplastic behaviour.

Using ionic liquids as the reaction media, we also synthesized a series of novel cellulose esters. The effect of substituents on thermal properties of cellulose derivatives was studied. Compared to cellulose and cellulose acetate, cellulose esters with bulky substituents showed clear glass transition temperatures (T_g) and flowing temperature (T_f). Introducing both soft segment and bulky substituents might further decrease T_g and T_f of cellulose esters. Therefore, homogeneous cellulose esterification in ionic liquids is an alternative practical way to synthesize thermoplastic cellulose derivatives.

Keywords: Cellulose, Ionic liquids, Cellulose graft copolymers, Cellulose esters, Thermoplastic

MECHANICAL PROPERTIES OF PEANUT HUSK COMPOSITE MATERIAL USING INJECTION MOLDING

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Introduction: It is important to use biomaterials for reducing environmental impact. Peanuts are one of the most popular nuts in the world. The peanut husks are removed to get peanuts. The most husks are discarded as an industrial waste. In this paper, the composite materials using peanuts husks and polypropylene (PP) were developed. Higher mass content of peanut husks in the material can reduce the mass of PP. It means that the cost of products can be reduced by increasing the content of peanut husks. The mechanical properties of the material were investigated.

Methods: The pellets of peanut husk powder and PP were produced by using a kneading machine and an extruder. The size of the powder is between 0.25~0.5mm and less them 0.25mm. Hydrogenated styrenic thermoplastic elastomer was used as additive to increase adhesive property. The mass content of peanut husk was varied from 0% to 30%. The pellets were applied to from specimen using injection molding. The tensile and bending tests were performed to evaluate mechanical properties. The test speed was 5mm/min performed in room temperature.

Results: Tensile strength, elongation and Young's modulus are investigated from the results of tensile tests. Fig.1 show the results of tensile test. Bending strength and bending modulus are examined from the results of bending test. Tensile strength is increased with decreasing the content of peanut husks. Elongation is increased with decreasing the contents of peanut husks. Elongation is increased by additives. Bending strength is increased with decreasing the content of peanut husks. Bending modulus is increased with increasing the content of peanut husks.

Discussion: Because of high rigidity of peanut husks comparing to PP, Young's modulus of the composite material is higher than that of PP. More investigation is required to find the reason of decreasing tensile strength and elongation with increasing the content of husks.

Keywords: Peanut husk, Injection molding, Composite material, Biomass

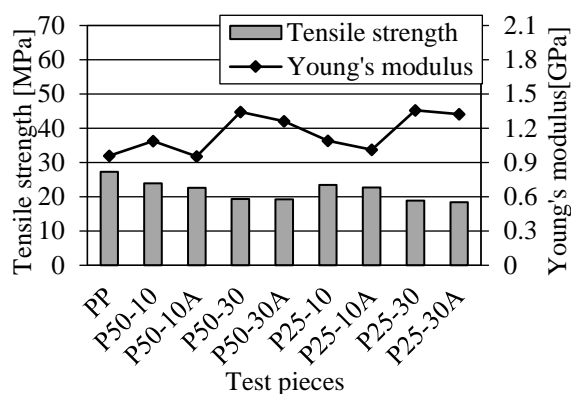


Fig.1 The results of tensile test

TRANSPARENT CONDUCTIVE MATERIALS PREPARED BY POLY(3,4-ETHYLENEDIOXYTHIOPHENE) DOPED WITH POLY(STYRENE SULFONIC ACID) (PEDOT:PSS) AND BISPHENOL COMPOUNDS AS A SECONDARY DOPANT

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Introduction: Indium tin oxide (ITO) has been used as a transparent conductive material since it has high conductivity and high transparency. Nevertheless ITO involves some problems such as poor mechanical flexibility, indium scarcity and high production cost. So a novel conductive transparent material is now required as a replacement for ITO. Many researchers have studied new transparent conductive materials such as conductive polymers, metal nanowires, and nano-carbons such as carbon nanotube and graphene. Among them conductive polymer PEDOT:PSS has been attracting considerable attention, since it has a high conductivity and transparency when it is coated as a thin film. However it has not conductivity enough to be replaced for ITO. Recently lots of secondary dopants such as ethylene glycol, DMF and DMSO have been proposed and applied to PEDOT:PSS films for the conductivity enhancement. The authors have been studying the effect of bisphenol compounds on the conductivity enhancement of PEDOT:PSS and the development of transparent conductive materials with higher conductivity and transparency than ITO.

Methods: PEDOT:PSS and a bisphenol compound such as 4,4'-dihydroxydiphenylsulfone (BPSO), 4,4'-dihydroxydiphenylsulfide (BPS), 4,4'-dihydroxydiphenylether (BPO), 2,4'-dihydroxydiphenylsulfone (24-BPSO) were dissolved in the iso-propanol/water mixture (75/25(w/w)). The product solution was coated on the glass plate and dried on the hot plate (100°C, 0.5 hr). The surface resistivity (R_s) of the thin films was evaluated with the four-point probe device and the volume resistivity (ρ) was calculated from R_s value and the film thickness.

Results: It was found that there was a large difference in the conductivity enhancement effect among the species of bisphenol compounds. The addition of BPSO or 24-BPSO contributed to the conductivity enhancement of PEDOT:PSS, while BPO and BPS showed no conductivity enhancement effect on PEDOT:PSS, but increase of its ρ value. The authors succeeded in the development of transparent conductive materials with superior performance (R_s : 152 Ω/\square , Transmittance(550nm): 88%) to ITO from the composites of PEDOT:PSS, BPSO, other additives and polymer binders.

Discussion: Bisphenol compounds such as BPSO and 24BPSO contribute to the conductivity enhancement of PEDOT:PSS, which is possibly attributable to their good solubility and higher acidity. Conductive films with superior conductivity and transparency to ITO films has been developed from the composites of PEDOT:PSS, BPSO and polymer binders.

Keywords: PEDOT:PSS, ITO, bisphenol, conductivity, transparency

HIGH-PERFORMANCE COBALT-BASED HETEROGENEOUS ELECTROCATALYSTS SYNTHETIZED BY ELECTROSPINNING

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Introduction: The increasing demand for clean and sustainable energy has inspired extensive exploration of advanced energy conversion and storage systems with high efficiency, low cost and environmental benignity, such as supercapacitors, lithium-ion batteries, fuel cells, and metal-air batteries. Nevertheless, the sluggish kinetics of oxygen reduction reaction (ORR) on the cathode side definitely limits the successful commercialization of high efficiency fuel cells and metal-air batteries. Pt-based materials have been considered as the most active noble metal ORR electrocatalysts, but largely constrained in practical applications due to their scarcity, poor durability and severe crossover effects. Therefore, non-precious metal-based and even metal-free electrocatalysts are newly emerging as promising candidates for high-performance fuel cell and metal-air battery applications.

Methods: The self-standing membrane of nitrogen-doped carbon nanofibers (NCNF) has been prepared by the combination of electrospinning, in-situ polymerization and high-temperature carbonization processes. Then, nanocubic- Co_3O_4 coated NCNF composite membranes were prepared by a one-step hydrothermal reaction at 150 °C for 3 h. For comparison, Co_3O_4 powder without the NCNF fiber membrane template was hydrothermally prepared under the same condition.

Results: Under an initial aniline concentration of 0.02 M, nitrogen-doped CNFs (denoted as NCNF-0.02) are obtained, which exhibit an increased fiber diameter of about 215 nm with uniformly distributed needle-like nanoparticles on the fiber surface. After hydrothermal reaction, nanocubic- Co_3O_4 particles with a mean size of 20-30 nm are uniformly distributed on the surface of NCNF-0.02 at a proper concentration of $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$, forming NCNF-0.02@ Co_3O_4 -0.2 composite fibers with an obvious coaxial structure. Electrochemical test indicates that pure Co_3O_4 powder exhibits rather poor current response toward ORR with the E_{peak} potential at -0.37 V. In contrast, CNF fiber membrane with intertwined conductive network structure exhibits higher current response with an obvious E_{peak} potential at -0.35 V. Moreover, introducing nitrogen doping in NCNF-0.02 fiber membrane results in more positive E_{peak} potential (-0.24 V) with largely increased current density. When Co_3O_4 is incorporated under a low initial molar weight (0.05 mmol) of $\text{Co}(\text{OAc})_2$, nanocubic- Co_3O_4 particles with a mean size of 100 nm are sparsely scattered on NCNF-0.02 fiber membrane, resulting in slight positive shift of the E_{peak} potential (-0.21 V). With the molar weight further increased to 0.2 mmol, Co_3O_4 nanoparticles with smaller size of about 20-30 nm are uniformly distributed on NCNF-0.02 fiber surface, thus leading to more positive E_{peak} potential (-0.18 V) with higher current density, which outperforms most of the previously reported Co/carbon based catalysts.

Discussion: The novel self-standing membrane of nitrogen-doped carbon nanofibers (NCNF) has been developed, acting as a three-dimensionally networked and conductive template for immobilization of electrochemically active Co_3O_4 particles. Thus, nanocubic- Co_3O_4 coated NCNF (NCNF@ Co_3O_4) composite fiber membrane with hierarchical structures is obtained, which subtly combines the synergistic effects between the electroactive nanocubic- Co_3O_4 , efficient surface nitrogen doping and highly conductive NCNF network. Therefore, the NCNF@ Co_3O_4 composite exhibits excellent catalytic activity toward oxygen reduction reactions with positive E_{peak} potential, high current density and superior durability over the commercial Pt/C catalyst, being a promising noble metal-free catalyst for practical fuel cell applications.

Keywords: Electrospinning, Nanocomposites, Electrocatalysts

OVER-MOLDED CONTINUOUS WOVEN JUTE/POLYLACTIDE ECO-COMPOSITES

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Introduction: In recent years, the use of natural products, such as natural fibres and polymers, has gained attention in industrial applications due to their low environmental impact. The growth of natural material applications has contributed to the reduction of CO₂ emission. The best match to obtain ecologically friendly composite materials is the combination of natural fibres with a biodegradable matrix. Poly(lactic acid), a biodegradable, renewable, linear aliphatic polyester, is widely used to produce biodegradable composite materials. A new thermoplastic composite technology so called “organic hybrid composites” holds promise to obtain lightweight structural composite components for automotive parts. In this process, a thermoplastic carbon or glass continuous fibre composite reinforcing sheet is over-moulded using matrix polymer in an injection moulding process. By inspiring from that new technology, it is aimed in this study is to process novel continuous jute fibre reinforced over-moulded polylactide composites.

Methods: Continuous jute fibre/PLA composite layers were prepared via film stacking method in a hot press. The fibre orientation was varied as 0°, 45° and 90°. The properties of these continuous fibre reinforced layers were determined via mechanical tests and scanning electron microscopy (SEM). The best combination was determined to use as reinforcing layer for over-moulded composite (OMC) manufacturing. The OMCs were prepared using an Xplore laboratory injection-moulding device. The mechanical, thermo-mechanical and morphological properties were investigated.

Results: Mechanical tests showed that the best properties for reinforcing jute/PLA layers was obtained when 45° orientation was used, therefore this construction was used to produce OMCs. The mechanical tests showed that flexural modulus and strength of OMCs was improved in comparison to neat PLA nearly without altering the density. The DMA results showed that the thermo-mechanical resistance of PLA was enhanced. SEM pictures showed that the jute/PLA interphase needs to be improved to further increase the properties.

Discussion: It was successfully demonstrated that all-ecological continuous natural fibre reinforced over-moulded PLA composites were processed. The one of the biggest advantage of this novel technique is the increase of mechanical properties of PLA without altering the density.

Keywords: polylactide, jute fiber, composites, over-molding, mechanical properties, thermomechanical properties

OC019

DESIGN AND FABRICATIONS OF FUNCTIONAL NANOFIBERS AND NANOFIBROUS MEMBRANES FOR ENVIRONMENT AND HEALTHCARE: CHALLENGES AND OPPORTUNITIES

Autchara Pangon, Nakarin Subjalearndee, Bunnakorn Suntamit, Nipon Phanomkate, Varol Intasanta

presented by Wanee Chinsirikul

Nano Functional Textile Laboratory, National Nanotechnology Center, National Science and Technology Development Agency, Thailand

Introduction: This presentation aims to address research capabilities of Nano Functional Textile laboratory at National Nanotechnology Center (NANOTEC), Thailand by highlighting on-going research on nanofiber fabrication and potential applications. Based on nanomaterials and polymer processing technology, three defined research schemes spanning across the value chain of textile industries are nanofunctional finishing processes, microfiber fabrications and nanofibrous membranes.

Methods: Syntheses, modification, characterizations and processing of multifunctional nanofibers and nanofibrous membranes of PVA based polymers were investigated. First example involves water-based multicomponent solutions containing polymer, UV resistant, antibacterial and water repellent agents. Prepared polymer was electrospun into nanofiber by Nanospider machine. Filtration property of the developed nanofibrous membranes against airborne Tuberculosis (TB) bacteria were studied. Second example is fabrication of chitosan nanofibers through electrospinning. Multicarboxylic acids, as environmentally friendly crosslinkers, were selected and used in chitosan electrospinning for fine-tuning physicochemical properties of the nanofibers.

Results: For the nanofibrous PVA based membranes, unique interplay among the functional components and developed structure of the multifunctional nanofibers led to mechanical-robust, flexible and water-resistant materials with flow-through capacity and size-selectivity towards TB bacteria. In the case of crosslinked water-based chitosan nanofibers, it was found that nanofibers could be effectively developed from solution electrospinning of chitosan/chitin whisker composite in non-toxic solvent system. The study on osteoblast cell biocompatibility revealed a potential use of these nanofibrous chitosans for tissue engineering scaffolds.

Discussion: Key findings of our developed PVA and chitosan nanofibers and/or nanofibrous membranes based on material-process-structure-property relationships will be discussed. Efforts in linking these functional nanomaterials into applications, such as nanofiltration for counteracting the airborne Tuberculosis bacteria and material for tissue engineering scaffolds, will also be covered.

Keywords: nanofibers, nanofibrous membranes, multifunction.

PREPARATION AND PROPERTIES OF ULTRA HIGH MOLECULAR WEIGHT POLYETHYLENE FIBER VIA DRY SPINNING

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Introduction: Ultra High Molecular Weight Polyethylene (UHMWPE) fiber is well known as one of high performance fibers together with carbon fiber and Kevlar. Currently, two routes are applied in UHMWPE fibers production. Decalin is used as solvent in dry route, while mineral oil is used in wet route. Comparing to wet route, dry route has advantages of short process, fast spinning, good product quality, directly recycled solvent and environment friendly. Wet route is widely researched in labs, on the other hand, research of dry route is relatively scarcity.

Methods: In this paper, high-performance UHMWPE fibers with 4 million molecular weight were prepared by advanced dry route pilot line, which was designed independently by Shanghai Research Institute of Chemical Industry (SRICI). The mechanical properties, thermal properties and microstructure of UHMWPE fibers with different draw ratio were characterized by electronic testing machine, SEM and DSC.

Results: Fig.2 showed that crystallinity degree of UHMWPE fibers increased from 49.57 percent to 72.17 percent with increasing drawing ratio. Comparing to as-spun fiber, well-drawn fiber's breaking strength increased from 2.35 cN/dtex to 31.53 cN/dtex, as well as 15.68 cN/dtex to 1054.78 cN/dtex improvement in breaking modulus (As shown in Fig.3).

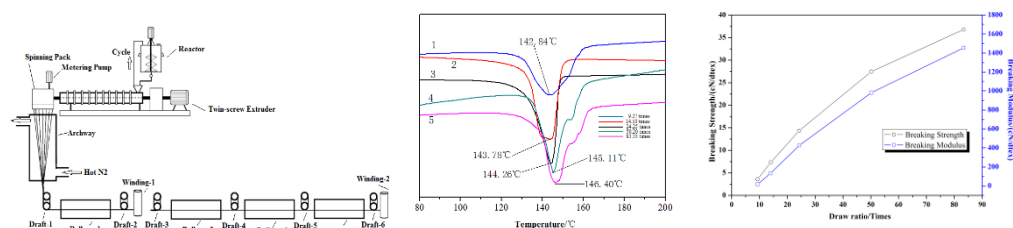


Fig.1 Flowchart of UHMWPE fiber production by Dry Spinning (SRICI) Fig.2 DSC graph of different fibers draw ratio Fig.3 Mechanical properties of different fibers draw ratio

Discussion: In the process, the disorder macromolecular chains was straightened, and amorphous regions turned into crystalline regions gradually, which resulted in well-ordered and compact micro-structure. Highly crystallized fiber showed better mechanical performance because uniform chains required more energy to break.

Keywords: Ultra High Molecular Weight Polyethylene fiber, dry spinning process, draw ratio, crystallization, mechanical performance

Visualization Analysis of Asymmetric Fountain Flow Phenomena by Dynamic Observation of Melt Front Behaviors

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Introduction: In the injection molding of fiber reinforced resins, inappropriate fiber behavior and fiber orientation characteristics during the mold filling process are known to cause deterioration of the surface properties of molded products and reduction of the mechanical strength of the molded products. In this study, we observed the melt front behaviors of long glass fiber reinforced resin in the flow front area, and clarified the relationship between the fiber content and mechanism of asymmetric fountain flow (FF) phenomenon.

Methods: We mounted a high-speed camera to a driving unit to observe the gate side from the open end of the mold cavity and videoed the FF at 500 frames per second while moving the camera according to the flow rate of the resin. Based on the obtained images, we measured the moving speed and movement trajectory of the FF tracer for each content (Refer to Fig.1).

Results: We found that the FF in the case of resins with the fiber content ratio of 0wt% is always symmetrical. However, as the fiber content increased, FF gradually transitioned from symmetrical to asymmetrical, and became asymmetrical for the first time when the fiber content ratio was 3wt% or above. There were no significant changes in the mode of the FF above 30wt% (Refer to Fig.2).

Discussion: We believe that the mechanism by which the melt front behavior changes according to the fiber content is due to the inconsistencies of the fiber length and fiber distribution. Fibers longer than the cavity plate thickness extend so that they cross both the cavities at the stationary side and movable side of the mold diagonally. Then, at the next moment, they are retrained at one side of both cavity walls (e.g. A) and are pulled back. When the amount of fiber increases, it causes the fibers to entangle, resulting in the swinging phenomenon of the entire front part of the flow front and the continuous generation of asymmetrical FF (Refer to Fig.3)

Keywords: Injection molding, Long-fiber reinforced PP, Asymmetric fountain flow, Visualization, Flow front

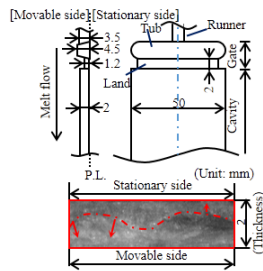


Fig.1 Cavity shape & example of observed image (Fiber content; 50wt%)

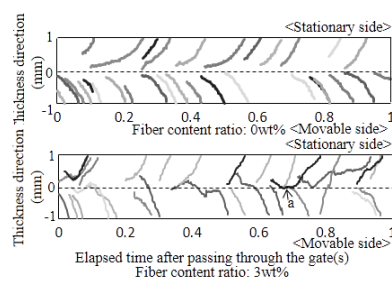


Fig.2 Trajectory variations with time elapse

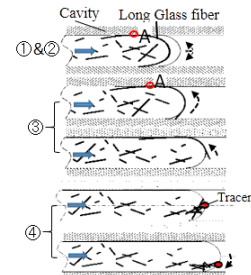


Fig.3 Illustration of fiber movement

DIRECT JOINING OF INJECTION MOLDED POLYMER ON ABRASIVE BLASTED METAL: INVESTIGATION OF MOLDING CONDITIONS

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Introduction: Injection molded plastic can be directly joined with a metal of which surface is roughened (fine-structured) in advance. This process is injection molded direct joining (IMDJ). We focused on abrasive blasting to roughen the surface of the metal. The IMDJ using the abrasive blasting (blasting-IMDJ) is a promising production technology since it can be easily introduced for industrial lines. It is, however, difficult to apply the blasting-IMDJ to the real industry in current state. The blasting-IMDJ has not been well investigated and thus does not hold an enough reliability for the applications. To improve the reliability, this study reports investigations of relationships between molding conditions and joining strength.

Methods: To investigate effects of molding conditions, we produced single lap-joint samples (A5052 aluminum alloy/polybutylene terephthalate) under varied molding conditions. Note that blasting conditions were fixed (Blast media: alumina grain, #150 (100 μm). Size of resultant surface fine-structures: tens μm). In this study, we focused on three parameters of the molding conditions; polymer temperature, injection speed, and cavity pressure. Each condition was controlled separately to avoid interference of the conditions. Since the cavity pressure cannot be controlled directly by the molding machine and can be affected by the polymer temperature, the injection speed, and injection volume, we adjust the injection volume to let the pressure be desired values. The joining samples were evaluated by a tensile-shear test to measure the strength of the joining.

Results: The values of the conditions except cavity pressure had positive correlations with the joining strength in limited ranges. In the investigated range of the polymer temperature, peak strength was obtained at 270°C. If the temperature was higher than this value, the strength decreased. For the injection speed, the strength was saturated over the speed of 100 mm/s. Although these two conditions affected the strength, the cavity pressure had no relation with the strength.

Discussion: The condition-strength relations are similar to relations of a (micro-)replication by injection molding. It means that joining strength could relate with a ratio of replication to the surface fine-structure. The higher temperature and the higher injection speed kept the higher polymer fluidity during molding and thus provided the higher replication ratio. Thermal degradation of the polymer under too high temperature made decrease of the strength. The strength saturation under the high injection speed was obtained by the saturation of the replication (full replication). Regarding the effect of cavity pressure, even the lowest pressure condition could provide full replication; then, the strength was not affected in used pressure range (20-60 MPa for holding pressure, 60-120 MPa for packing pressure). To make clear above discussions, we will observe interfaces of the joining samples and confirm effects of the condition variations on the replication ratios in future work.

Keywords: Metal/plastic direct joining, Injection molding, Roughened surface, Abrasive blasting

OC023

UNDERSTANDING HYDROSTATIC PRESSURE TESTING OF POLYETHYLENE PRESSURE PIPES

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Introduction:

The pressure pipe market is the fastest growing polyethylene market segment both in Australia and globally. Polyethylene pipe has gained market share dominance in applications such as the transportation of drinking water, management of waste fluids as well as the transportation of mining slurries and natural gas.

The design of base polymer resin structure used in polyethylene pipes is fundamental in meeting the demanding engineering requirements of the pipe system which must securely transport the contained fluids under pressure throughout a 100 year service life. Consequently the pipe market is highly regulated and pipes must adhere to comprehensive product and installation standards.

Methods:

In order to qualify polyethylene for use in pressure pipes, the polyethylene compound must undertake a comprehensive hydrostatic pressure testing in accordance with International Standard ISO9080 – ‘Plastics piping and ducting systems- Determination of long term hydrostatic strength of thermoplastics materials in pipe form by extrapolation’.

Results:

This testing consists of pressure testing of a number of polyethylene pipe specimens at varying pressures and temperatures. The results obtained are used to generate pressure curves for use in design of pipe lines.

Discussion:

This presentation aims to provide an understanding of the effect of testing parameters on the hydrostatic performance of polyethylene pipes. The effects of hoop stress, temperature and pipe dimensions on the performance of pipes in hydrostatic pressure tests will be discussed.

A STUDY ON INFLUENCE OF RESIN TEMPERATURE ON FILLING BALANCE OF MULTI-CAVITY MOLD

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Introduction: Filling imbalance, a problem encountered with multi-runners, has been clarified to be closely related to asymmetrical temperature distribution in the secondary runner. In this study, we investigated the effects of a main runner installed with a partition at the center for reducing asymmetrical temperature distribution along the width of the secondary runner by measuring the temperature distribution along the widths of both the secondary runner and main runner.

Methods: Using a system which moves the measurement points through a round rotary block structure installed with an infrared thermometer, the sensor position was moved for each shot to measure the temperature distribution along the width of the runner. In the experiments conducted, the temperature distribution along the widths of the main runner and secondary runner of a two-cavity mold with T-shaped runner was measured under high injection rates in a main runner installed with a partition in the center and a main runner without partition for comparisons (Refer to Fig.1).

Result: The main runner without the partition showed increase in the temperature near the runner wall than that at the center of the runner. In the secondary runner, the temperature was high at the outer circumference, resulting in asymmetrical temperature distribution. In the main runner installed with a partition at the center, the temperature at the center increased significantly. The increase in the temperature at the inner circumference of the secondary runner means that the asymmetrical temperature distribution has decreased considerably (Refer to Fig.2).

Discussion: In the main runner installed with a partition in the center, the temperature of the melt at the center increased due to the shear heating at the partition wall, and this melt flows into the inner side of the secondary runner. These results clarify that the partition installed in the center of the main runner reduces asymmetrical temperature distribution along the width of the secondary runner.

Keywords: Injection molding, Multi-cavity, Filling balance, Temperature distribution

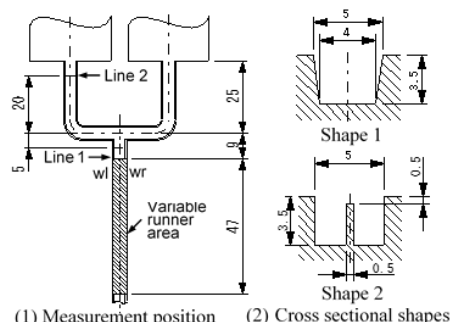


Fig.1 Runner shape and measurement positions (Unit:mm)

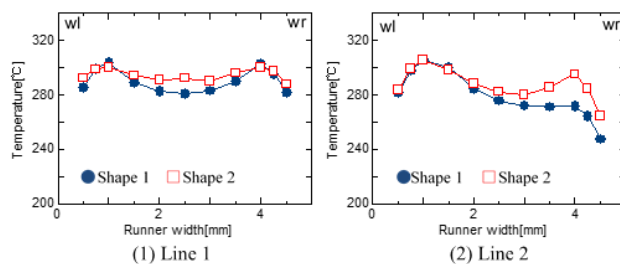


Fig.2 Comparison of temperature profiles in different runner shapes
(Resin: TPEE, Injection rate: 30cm³/s, Resin temperature: 220°C)

MOLD AND MOLDED HISTORY DEPENDENCE OF MECHANICAL PROPERTIES OF RECYCLED AMORPHOUS PLASTICS

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Introduction: Previously, we had investigated systematic evaluation of molecular properties, physical properties and inner structures of recycled semi-crystalline plastics. From the investigation, we had found that main reason of the poor physical properties of semi-crystalline recycled plastics is physical degradation and can regenerate the mechanical properties by selecting the best physical molding condition.

In this time, we investigated the effect of the difference of the mold and molding conditions on the melt viscoelasticity and the tensile properties.

Samples: For the amorphous polymer samples, we used virgin TOPAS (TOPAS ADVANCED POLYMERS Gmb, 5013-L10), recycled TOPAS (From Leiz Advanced Technology Corp.), virgin Polystyrene (TOYO Styrene GP(G200C)) and recycled polystyrene (From Shinei kasei Co. Ltd.)

Methods: We measured viscoelastic properties of these samples and evaluated the change of molecular weight. The longest relaxation time also evaluated from the results. For the tensile properties, we made thin films by compression molding with changing molding temperature and time and compared the results.

Results: From the results, in the case of amorphous polymer, the molecular weight after molding much depends on the type of mold. And also, even after keeping the long enough in molten state than the longest relaxation time, molding history remains. We found that, the physical properties were better than the virgin plastics. Addition, by keeping even more prolonged molten state, it was also found that it would have a ductile characteristics. From the SAXS experiments, we had the suggesting results that the transition comes from the construction of higher structure in the inner structure of amorphous plastics.

Discussion: By this study, we know that the molding history remains much after the longest relaxation time even for the amorphous plastics.

Keywords: Amorphous Plastic, Recycle, Tensile Property, Relaxation Time, Molding history

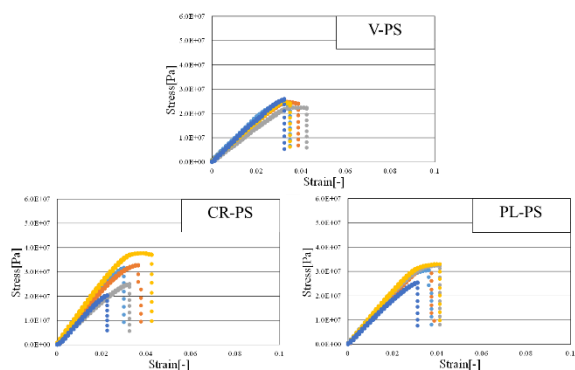


Fig. The tensile test results of thin films of Polystyrene.

Press temperature : 210°C Press time : 2min

TRANSPARENT OIL/WATER SEPARATION FILTER USING POLYESTER MESH

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Introduction: For the practical applications of transparent superhydrophobic surfaces, it is critical that they possess long-term durability of the self-assembled nanostructures against mechanical wear, shear, and liquid flow. In this study, we investigate the possibility of a highly transparent and superhydrophobic thin film with enhanced mechanical abrasion resistance by combining a see-through hydrophobic mesh with a hydrophobic SiO₂ nanoparticle hierarchical structure.

Methods: Superhydrophobic surfaces are fabricated using three steps. (1) Chemical and morphological changes are generated in the polyester mesh by alkaline treatment of NaOH. (2) Second, alkaline treatment causes hydrophobic molecules of perfluorodecyltrichlorosilane to react with the hydroxyl groups on the fiber surfaces forming covalent bonds by using the chemical vapor deposition method. (3) Hydrophobicity is enhanced by treating the mesh with SiO₂ nanoparticles modified with perfluorooctyltriethoxysilane using a spray method.

Results: The results were shown in Fig.1 and Fig.2. The transmittance of the fabricated superhydrophobic mesh is approximately 80% in the spectral range of 400–1000 nm. The water contact angle and the water sliding angle remain greater than 150° and lower than 25°, respectively, and the transmittance remains approximately 79% after 100 cycles of abrasion under approximately 10 kPa of pressure. The mesh surface exhibits a good resistance to acidic and basic solutions over a wide range of pH values (pH 2–14), and the surface can also be used as an oil/water separation material because of its mesh structure.

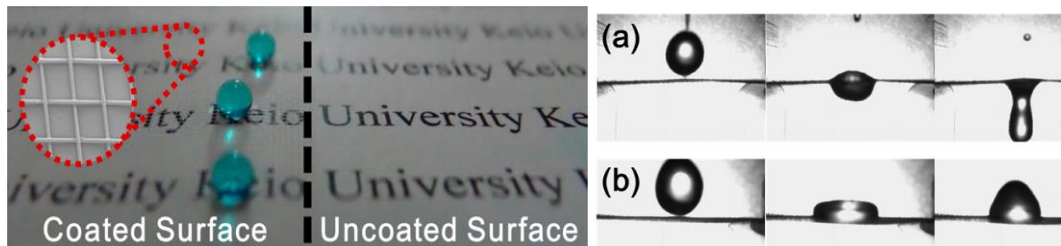


Fig.1 Transparent Superhydrophobic Mesh Fig.2 Oil/ Water Separation (a) n-octane (b) water

Discussion: The combination of the see-through mesh and the SiO₂ nanoparticle hierarchical structure is an effective and simple method for improving the abrasion resistance and transparency of these superhydrophobic films. This simple but novel and effective applications method may be useful for the improvement of highly transparent superhydrophobic surfaces for various applications.

Keywords: superhydrophobic, transparency, polyester, durability, oil/water separation

IONIC LIQUID-ASSISTED ENERGY-SAVING PREPARATION OF STARCH-BASED ELECTRO-CONDUCTIVE MATERIALS

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Introduction: Polymer processing should be developed to be “greener” by minimising the energy consumption. While significant progress has been made to develop various polymeric materials for new and enhanced properties using traditional polymer processing techniques, limited research has been reported on optimising the processing itself to achieve the same or even better polymer melt state. This work concerns the melt processing of a natural biopolymer, starch, which has poor processability. Due to its biodegradability and readiness for modification, starch-derived materials have attracted much interest in the recent decades for various applications. Nonetheless, the processing of starch is mostly carried out using solution methods. Here, we report a novel and simple way to assist the melt processing of starch, which is also instrumental to the processing of other semi-crystalline natural polymers.

Methods: The starch was firstly pre-mixed with aqueous ionic liquid (IL) for *ca.* 1 h. Then using a simple one-step compression moulding process at a moderate temperature (about 50 °C) for the pre-mix, the starch-based electro-conductive films were obtained. The temperature used here was much lower than the widely-reported temperatures for starch processing.

Results: With IL-water treatment, flexible starch-based electro-conductive films were successfully developed at largely reduced temperature. Therefore, a novel energy-saving processing for starch was established. The films displayed electro-conductivity as high as *approx.* 10^{-2} S/cm.

Discussion: For these starch films, the ratio of starch/IL/water, the processing temperature and the relative humidity (RH) could prominently alter the multi-scale structure and thus affect the electro-conductivity. Specifically, the increased temperature and the reduced starch content were preferable for the melting of starch, *i.e.*, the disruption of the granule ghosts, lamellae and crystallites and the formation of denser fractals. These structural changes enhanced the links between starch molecular chains but hindered the movement of electro-conductive ions, leading to an enhanced mechanical properties but a weakened conductivity. With increased RH, more water molecules were trapped into the film especially into its melted starch areas. The water molecules reduced the links between the ghosts and the melted starch, the rearrangement of starch and the compactness of fractal gels, which decreased the mechanical properties but enhanced the electro-conductivity. Again, the water molecules reacted with [OAc]⁻ to release [OH]⁻, and exchanged protons with H-2 of the imidazolium cations; so the conductivity was enhanced. Hence, this work demonstrate that using a cost-effective process starch-based electro-conductive materials can be easily prepared, which have the potential for high-value applications such as biosensors.

Keywords: electro-conductive materials; ionic liquid; energy-saving; compression moulding; biopolymers

IMPROVING CELL MORPHOLOGIES AND MECHANICAL PROPERTIES OF INJECTION MOLDED POLYAMIDE 6 FOAMS BY USING HYDROPHOBIC MODIFIED CELLULOSE NANOFIBER

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Introduction: Foamed resins have many excellent properties such as lightweight, heat and electric insulation, sound and shock absorption, and so on, while their mechanical strength and heat resistance are weaker than un-foamed (solid) resins. In order to improve mechanical properties, it is effective to reinforce foam matrix and reduce bubble size. Cellulose nanofiber (CNF) is expected to be a bland new polymer reinforcement, because of its abundant resources and excellent physical properties. In this study, hydrophobic modified CNF/polyamide 6 (PA6) composites were prepared, and foaming behaviours of them and physical properties of the obtained foams were investigated.

Methods: Foamed test pieces were produced by the physical foaming process with using an injection molding machine, and N2 as blowing agent. Cross section observation of the foams by SEM were done. Flexural tests of the test pieces were also done.

Results and Discussion: Cell morphologies of the foams are shown in Fig. 1. Cell size of modified CNF5% foams was much smaller than that of PA6 and mineral5% foams. This is because that modified CNF, which has an affinity for PA6 increased the viscosity of PA6 and inhibited coalescences of bubbles. Specific flexural modulus of solid and foamed test pieces were summarized in Fig. 2. Modified CNF increased flexural modulus of PA6 higher than mineral. Moreover, decreasing relative density by foaming raised specific flexural modulus of modified CNF/PA6 at the relative density more than 0.7. This is because the bubble of the foams with the relative density less than 0.7 collapsed and the flexural modulus of the foams decreased. It was found that the modified CNF was more effective filler than mineral to decrease the bubble size and increase the mechanical properties of foams.

Keywords: cellulose nanofiber, polyamide 6, foaming, lightweight material

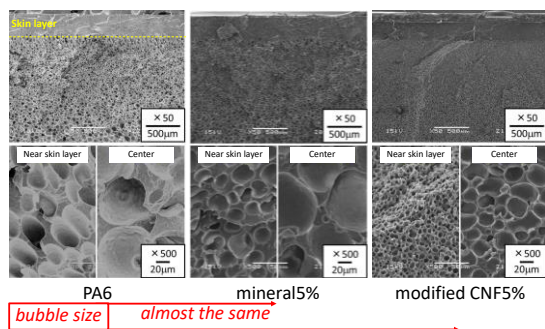


Fig. 1 Cell morphologies of foams

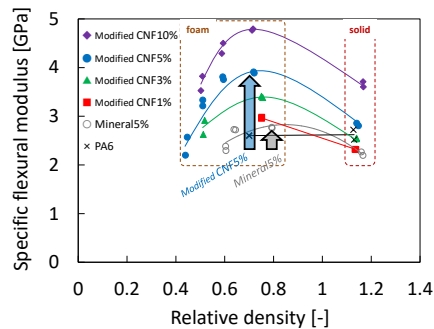


Fig.2 Relationship between Specific flexural modulus and relative density of solid and foamed test pieces

OC029

PROCESS FOR SOLVING HEAT SEALING PROBLEMS IN POLYETHYLENE FILMS

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Heat sealing is a common type of process in the manufacture of packaging from flexible films. The integrity of the process is critical for ensuring the protection of the package contents, while a fast trouble-free process is critical for the commercial viability of the packaging plant. Polyethylenes are widely used in flexible films for packaging due to the excellent sealing characteristics of these polymers.

The presentation will give an overview of the more common problems that may occur during these sealing processes. A procedure for finding the root causes and their solutions will be described. An understanding of the sealing process at the molecular level is a key part of solving problems. Knowledge of the heat sealing properties of the polymers in the film is also important, and this can be gained through examining heat sealing curves. Further valuable information can be gained by collecting information about the occurrence of the problem, using tools such as problem analysis questioning and fishbone diagrams. In this work, these tools have been tailored to be able to be readily used for solving problems in heat sealing. Validation tests for the causes are then conducted.

A number of case studies where this procedure has been used will be described. The problem analysis tools led to different types of validation testing in each case. Examples of the validation testing included Differential Scanning Calorimetry, Hot Tack testing on a laboratory tester, and trials on the industrial sealing equipment. After using the problem solving tools followed by the validation testing, the root causes were quickly identified and solutions were implemented. These examples demonstrate the effectiveness of this problem solving process.

Keywords: Sealing, films, polyethylene

OC030

MASTERBATCH INNOVATION - TOWARDS 2020

Neil Whelan

Axio Masterbatch, Victoria, Australia



I would like to discuss the topic of Innovation and how that relates to our Customers Expectations within the MB Industry today and indeed for the remainder of this decade. Whilst I am going to put some emphasis on my home market, I am also going to talk about this overall region and its global context.

In so doing, I would like to discuss

- 1. Customer Trends**
- 2. Innovation Methods**
- 3. Measures of Innovation & relevance to this region**

Masterbatch as you would all know, is an industry sector exposed to and servicing virtually all sectors of the plastics industry, hence we all have a quite wide involvement and influence within the industry.

Ever increasingly, the reverse also applies – i.e. all sectors of the plastics industry in turn, affect the MB industry – particularly in a strongly competitive environment.

BIREFRINGENCE CONTROL OF POLYMERIC FILMS BY ANISOTROPIC STRUCTURE AND ITS OPTICAL APPLICATION

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¹Japan Advanced Institute of Science and Technology, Nomi, Ishikawa, Japan, ²Nagoya Institute of Technology, Nagoya, Aichi, Japan

Introduction: A technical trend for an optical retardation film is to adjust the retardation in a broad range of visible light. In case of a quarter-wave plate, the retardation has to be a quarter of wavelength. It means that the birefringence of a quarter-wave plate has to increase with wavelength. Although this extraordinary wavelength dispersion is necessary for advanced displays, the orientation birefringence of general polymers decreases with wavelength. In this study, we focus on the form birefringence originated from structural anisotropy in a film to control the wavelength dispersion.

Methods: The films were prepared by a solution-cast method. Cellulose triacetate (CTA) and Diisodecyl adipate (DIDA) were dissolved into a mixture solvent of dichloromethane and methanol with 9/1 weight ratio. The solution containing 4 wt% of CTA was evaporated at room temperature to obtain a film. The films were uniaxially stretched by a tensile machine with a temperature controller. The hot-stretching was performed after holding at the drawing temperature for specific periods. The hot-stretched films were immediately quenched by cold air blowing at a draw ratio of 1.5 to avoid relaxation of molecular orientation. The films were immersed into methanol to remove the plasticizer. Then, they were dried in vacuo at room temperature.

Results: A hot-stretched CTA film containing DIDA shows negative orientation birefringence with ordinary wavelength dispersion, as similar to that of a pure CTA film; the absolute value of the birefringence decreases with wavelength. However, after extracting DIDA from the stretched film by immersion into an organic solvent, the birefringence of the film dramatically changes from negative to positive with extraordinary wavelength dispersion; the magnitude of the birefringence increases with the wavelength. The SEM observation reveals that the film contains numerous ellipsoidal pores elongated to the stretching direction. Moreover, light transmittance of the porous film increases by decreasing heating time and increasing strain rate at hot-stretching process.

Discussion: The results indicate that the ellipsoidal pores provide the form birefringence as a positive value. Therefore, the combination of form birefringence and orientation birefringence provides extraordinary wavelength dispersion. Moreover, the formation of dispersed phase, and thus the form birefringence as well as light transmittance can be controlled by conditions at hot-stretching process.

Keywords: birefringence, anisotropic structure, optical film

PREPARATION OF MULTI-FUNCTIONAL GREENHOUSE COVER FILM WITH UV FILTRATION, NIR REFLECTION AND GOOD LIGHT DIFFUSION

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In tropical areas, cultivation is limited by an excess heat occurring due to high radiation levels. About 40% of the solar radiation reaching the earth surface is the near-infrared radiation (NIR; 700 – 2000 nm) which is the main source of heat load. Planting in the greenhouse is becoming popular since it is possible to manipulate light as well as other environmental factors to improve plant productivity and quality.

The incorporation of NIR reflective particles (pure metal, metal oxide, etc.) into polymer film is an effective way to reduce temperature inside the greenhouse. However, the ideal greenhouse film should not only reflect NIR but also minimize the UV transmission (250 – 400 nm) in order to protect plant cells. Moreover, the film must permit the photo-synthetically active radiation (PAR; 400 – 700 nm) in accordance with photosynthetic requirement of plants.

In this present work, 90% of linear low density polyethylene (LLDPE)/10% of Ethylene vinyl acetate (EVA) based polymer and 0.5 – 1.0 wt% modified TiO₂ and 0.25 - 0.5 wt% aluminium (Al) flake were compounded by twin-screw extruder. The compounding was then blown into film with 30 µm thickness by using blown film extruder. Mechanical properties of the resulting film were characterized using tensile testing machine. Light diffusion was performed by Haze meter and optical properties were measured by UV/Vis/NIR spectrophotometer.

Results showed that 90 µm thickness of 3 layers laminated film exhibiting 14 – 20% NIR reflecting, 56 – 100% UV blocking, 49 – 75% PAR transmission and 40 – 60% of Haze diffusion could be achieved. However, adding those additives adversely affected the mechanical properties of the polymer film.

Keywords: greenhouse film, multi-functional film, NIR reflective, optical properties, mechanical properties

STRESS-OPTICAL BEHAVIOR OF AMORPHOUS FILMS DURING TWO-STEP UNI-AXIAL STRETCHING AND SEQUENTIAL BI-AXIAL STRETCHING PROCESSES

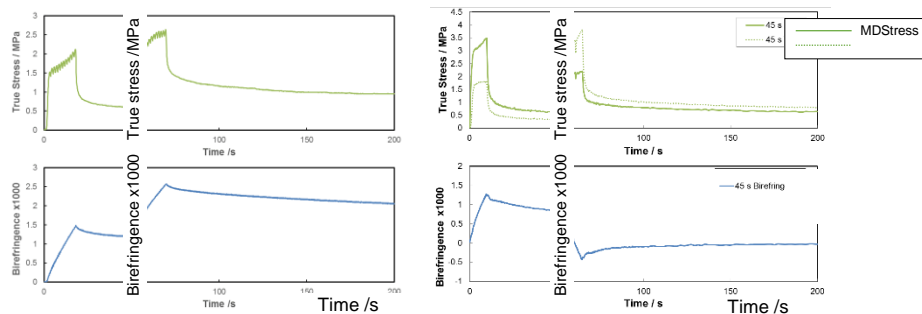
Yuki Takada, Kensuke Nemoto, Wataru Takarada, Takeshi Kikutani

Tokyo Institute of Technology, Tokyo, Japan

Introduction: In the processing of amorphous polymers, structure can be fixed only by cooling the polymer below its glass transition temperature. Therefore, understanding of structure development and relaxation behavior was important. In this research, multi-step processing of amorphous polymers was investigated through the simultaneous measurements of stress and birefringence.

Methods: The films of amorphous polycycloolefine (PCO) was used for this research. The two-step stretching was conducted at 133 °C. During the stretching and relaxation processes, stress and birefringence were measured simultaneously.

Results: The behaviors of stress and birefringence during two-step uni-axial stretching and sequential bi-axial stretching was shown in Fig. 1.



(a) Two-step uni-axial stretching

(b) Sequential bi-axial stretching

Fig.1 The behaviors of stress and birefringence during (a) two-step uni-axial stretching and (b) sequential bi-axial stretching process.

Discussion: In both process, step increase and decrease of stress at the start and end of stretching whereas smooth change at birefringence. Such stress behavior is considered as the appearance of the effect glassy state. Asymptotic values of stress and birefringence indicate an existence of the structure which have long-relaxation time. This asymptotic values were mainly decided by stretching temperature and total stretching ratio but rarely affected by stretching rate and intermittent holding time.

Keywords: Structure development, Relaxation, Birefringence, Film, Stretching

CO₂ LASER PERFORATION OF POLYPROPYLENE FILMS PREPARED BY SIMULTANEOUS BIAXIAL STRETCHING

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Biaxially oriented polypropylene (BOPP) films are widely used in all areas of packaging. Films with high oxygen transmission rate (OTR > 10,000 cc/m²/day) are required for extending shelf life of fresh products such as fruits and vegetables. Laser perforation is one of the techniques for increasing the gas transmission rate through the formation of tiny holes in the film. The shape and the size of microholes on the films depend on laser perforation process parameters (focal length and pulse duration) and materials (degree of molecular orientation and film thickness). Therefore, in this research, BOPP films were produced by simultaneous biaxial stretching of original PP films to various draw ratios. These films were perforated by CO₂ laser under a constant pulse repetition rate of 1 Hz and various pulse energy from 1.8 to 18 mJ, which corresponds to the pulse duration from 30 to 300 microseconds. The diameters of perforated holes increased with increasing pulse energy. For example, for the film sample with the equi-biaxial stretching ratio of $\lambda_{MD} \times \lambda_{TD} = 4 \times 4$, the hole diameters were 103.8, 147.1, 250.4 and 288.3 μm for the pulse energies of 4.8, 6.0, 12.0 and 18.0 mJ, respectively. These micro holes showed circular shape because of the isotropic orientation of molecular chains with respect to the axis normal to the film surface. The volume loss analysed from the cross-sectional shape of microhole also increased with the increase of pulse energy and reached ~400 % for the pulse energy of 18.0 mJ. The size of perforated micro hole was affected not only by the laser pulse energy but also by the focal length of used lenses. When the focal length was increased from 38.1 to 63.5 mm, the average size of microhole increased even though the fluence of laser (energy per unit area) was kept constant. This is mainly due to the increase of focal spot diameter and accordingly the use of higher pulse energy. It is interesting to note that the starting of the opening of hole is mainly controlled by the fluence of laser. Furthermore, for clarifying the effect of film thickness, films with the same stretching ratio and different thickness were also investigated. It was found that the size of microhole decreases with the increase of film thickness. As a whole, these results clearly demonstrated that the CO₂ laser processing is a promising technique for producing the perforated films.

Keyword: laser perforation, biaxially oriented polypropylene, food packaging

ULTRA HIGH MOLECULE WEIGHT POLYETHYLENE FILTRATION MATERIALS

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Introduction: Benefit from its linear long chain structure and high molecule weight, UHMWPE shows high mechanical strength, highly resistance to abrasion and chemicals, low absorption toward water, self-lubricating and nontoxic, which lead to applications including gas and liquid filtration, medical diagnostic test kits, low dielectric constant wrap for microwave cables, batteries and other applications where porosity, toughness and accuracy of gauge are important.

Methods: Herein, we discussed UHMWPE filtration materials by crafting methods: sintering, thermally induced phase separation (TIPS), melt extrusion-stretching, sol-gel methods, etc.

Results: The porosity, as well as the water flux, decrease with increasing initial UHMWPE concentration. On the other hand, rejection rate towards BSA increase with higher initial UHMWPE concentration (As shown in Fig.1, Fig.2). We find the breaking strength increase with the increasing UHMWPE concentration as well as higher stretching ratio (As shown in Fig.3).

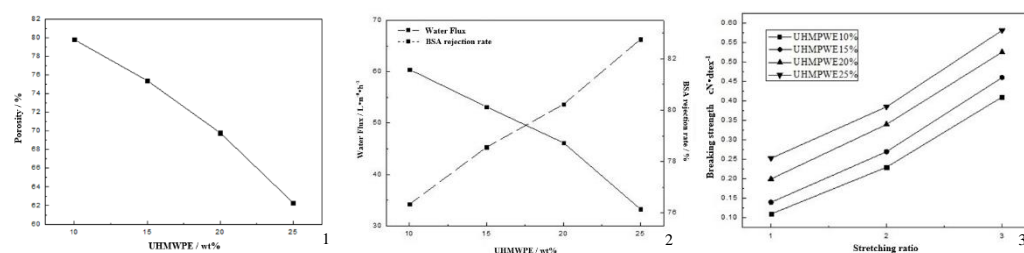


Fig.1 Porosity Changes with concentration of UHMWPE

Fig.2 Water flux and BSA rejection rate changes with concentration of UHMWPE

Fig.3 Breaking strength changes with stretching ratio

Discussion: The main reason is when concentration become higher, the membranes' pore structure grow coarser and thicker. Moreover, when membranes are stretched, the degree of order become larger. Macromolecule get closer to each other and force between them grow stronger, which lead to increasing of membranes strength.

Keywords: UHMWPE, TIPS method, sintering method, porous film, permeability, hydrophilic modification, hollow fiber membrane.

OC036

YALLOURN POWER STATION FLOODING CASE STUDY INTO THE VERSATILITY OF PE100 PIPES

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The urgent diversion of a river through a rapidly constructed pipeline was required to avert a power supply crisis to the state of Victoria. In early June 2012, the Morwell River surged and a levee collapsed allowing the river to flow directly into the open cut coal mine which supplies the Yallourn power station. The flooding caused US\$400m of damage to infrastructure and put the electricity supply to over 1 million homes and businesses at risk.

With limited coal reserves available to keep the power station operational, a rapid response was required to divert the river, dewater the coal mine and reinstate the supply of coal to the power station. Polyethylene PE100 pipe was the only solution that met the project criteria of fast availability, large pipe diameters, adaptable installation techniques, high integrity joints, abrasion resistance and flexibility. A total of 4km of 1600mm diameter PE100 pipe had to be rapidly manufactured and transported to the installation site. Large diameter pipe welding equipment was air freighted from the USA and made operational with a commissioning process that employed destructive weld testing techniques.

The around-the-clock pipeline installation involved welding ten lengths of pipe into 120m strings which were then dragged to the installation site for final joining. In just over 2 months, the tie-ins were completed and the pipeline commissioned so that the entire Morwell River was flowing through the pipeline and the repair of the mine infrastructure could begin. Two years later, the pipeline is still in service ensuring effective operation of the mine and securing the supply of power to Victoria.

Polyethylene, PE100, pipe, Yallourn

WET SPINNING OF PAN-BASED POLYMERS MADE BY RAFT TECHNOLOGY

Jasjeet Kaur, Keith Millington, Jackie Cai, Colin Brackley

CSIRO, Geelong, Australia

Introduction: Wet spinning is a standard method for making high strength polyacrylonitrile (PAN) based fibres as carbon fibre precursors. The PAN based polymers synthesised by using RAFT technology (RAFT PAN) are expected to result in improved carbon fibre precursors, as they have a substantially lower polydispersity index (PDI) than those made by conventional free radical polymerisation. A low PDI PAN polymer means a high consistency in its polymer structure, and it is free from very high molecular weight fractions, which may cause difficulties in polymer dissolution, filtration and spinning. This also indicates that the RAFT PAN may have different rheological properties, and require different spinning conditions to maximise the potential of this advanced polymer. To date, very limited information is available on wet spinning of RAFT mediated PAN polymers. In this study, we investigate the special characteristics of the RAFT PAN polymers and their effects on spinning performance and resultant precursor fibre properties.

Methods: We studied the rheological properties and wet spinning performance of both conventional and RAFT PAN polymers using dimethyl sulphoxide as a spinning solvent. The precursor fibres were obtained under various wet spinning conditions, and their morphology, crystallinity and mechanical properties were characterised. .

Results: The results of this study show that RAFT PAN polymers exhibit unique rheological properties. RAFT PAN solutions have much lower viscosity values than the conventional polymer solutions at same conditions. They also show more liquid character and gel slower than their conventional controls. These special rheological properties enable a higher polymer content in a spinning solution, which still remains spinnable. Due to the different rheological behaviour, RAFT PAN polymers require to be spun at different spinning conditions. By the time of this abstract submission, we have produced the RAFT PAN precursor fibres under various wet spinning conditions. Further optimisation of the spinning parameters is ongoing.

Discussion: The unique rheology of RAFT PAN polymer offers a wider working window for wet spinning operation and allows a higher solid content in the spinning solution. This is probably attributed to the absence of very high molecular weight polymer fractions. The improved spinning performance of the RAFT PAN polymers are expected to result in improved quality and performance of carbon fibre precursors.

Keywords: wet spinning, polyacrylonitrile, RAFT, rheology, carbon fibre

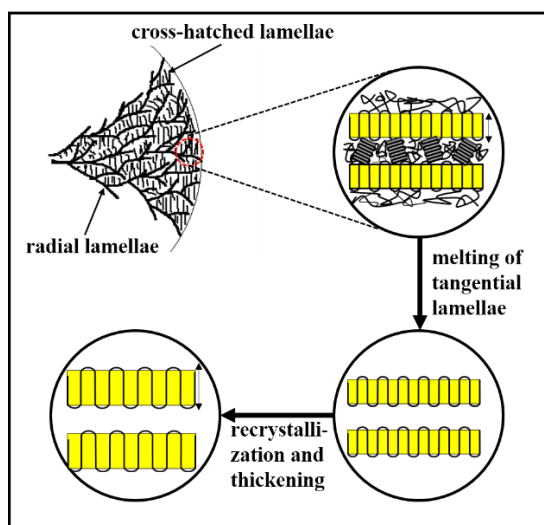
LAMELLAR THICKENING AND THE RESULTANT ABNORMAL MELTING BEHAVIOR OF ISOTACTIC POLYPROPYLENE FILMS

Zhenzhen Zhou, Huihui Li, Shouke Yan

Beijing University of Chemical and Technology, Beijing, China

In this work, an abnormal melting behavior of isotactic polypropylene (iPP) was observed by polarized optical microscopy (POM), i.e., the melting of α -iPP spherulites isothermally crystallized at 135 °C could occur prior to that of α -iPP spherulites crystallized at 125 °C when heated by 2 °C/min, but the case was opposite if the heating rate was 10, 20 or 50 °C/min. The changes of lamellar organization during heating process (2 °C/min) were investigated by scanning electron microscopy (SEM), wide-angle X-ray diffraction (WAXD) and in-situ synchrotron small-angle X-ray scattering (SAXS). The occurrence of lamellar thickening could be revealed by in-situ synchrotron SAXS, and it can be found that the initial lamellar thickness of α -iPP spherulites grown at 135 °C is over that of spherulites grown at 125 °C, whereas the latter will exceed the former when heated over 160 °C by means of lamellar thickening. SEM micrographs of iPP sample can also demonstrate the occurrence of lamellar thickening. Moreover, the change of mixed α -iPP spherulites to negative ones could be observed by POM during melting process, which could indicate that the amount of tangential lamellae in α -iPP decreased remarkably. Thus, the abnormal melting behavior should be attributed to the lamellar thickening, which can be described as the following, i.e., the tangential lamellae in the spherulite will melt and recrystallize on the radial lamellae during heating process, yielding much thicker radial lamellae with higher perfection. Moreover, the amount of tangential lamellae in spherulites crystallized at 125 °C is more than that in spherulites crystallized at 135 °C, and average lamellae thickness of spherulites grown at 125 °C can exceed that of spherulites grown at 135 °C via lamellar thickening process. Consequently, the spherulites crystallized at 135 °C will melt before that of spherulites crystallized at 125 °C. A schematic diagram showing the lamellar thickening process is present in the following.

Keywords: isotactic polypropylene, lamellar thickening, tangential lamellae, radial lamellae



A schematic diagram of iPP melting process with low heating rate.

COMPARATIVE INVESTIGATIONS ON DIFFERENT SHEAR THICKENING SUSPENSIONS FOR IMPACT ENERGY DISSIPATION

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The transition from viscous liquid to solid state as a function of shear rate in dense suspensions has been of interest, over a decade. Although suspensions comprising of nano or micro sized particles in a Newtonian liquid medium are ubiquitous, those suspensions showing shear thickening have to fulfil certain criteria to be a shear thickening fluids (STF). Several physical and chemical properties of both suspended particles and the liquid medium independently stimulates the uniqueness of the liquid-solid transition. However, even with the development of scaling relations and predictive models for rheological parameters, the contribution of forces of interactions present in suspensions responsible for their flow behaviour is still ambiguous. In the current study we consider 3 systems as comparative studies correlating with their rheological properties. We present the occurrence of shear thinning with a high yield stress at large particle concentrations in cornstarch–water, fumed silica-PEG and silica-PEG suspensions. We compare the forces of interactions responsible for all the systems. For 50 wt% of cornstarch in water we observed a continuous shear thickening with a critical shear rate of 21.5 s^{-1} . The viscosity rises from 0.026 to 0.239 Pa.s post critical shear rate. However, with the addition of PEG to water (1:1 ratio) at same cornstarch concentration, a shear thinning behaviour is observed with high yield stress. Similarly, 19 wt% of fumed silica in PEG 400 and 62 wt% of surface functionalized silica in PEG 400 also showed a shear thinning behaviour with high yield stress. The dynamics rheology of all the suspensions under study however showed high elastic modulus in the frequency range of 0.1 to 100 s^{-1} . The amplitude strain under which linear viscoelastic behaviour observed is less than 0.1% for all the suspensions. Further, we also present impact behaviour and energy dissipation analysis for shear thickening suspensions using Abaqus. The direction of energy displacement after the impact is visually perceived by this simulation technique. By combining the experimental and simulation results, we provide a route where one can choose the suspensions for suitable applications.

Keywords: Shear thickening suspensions, shear thinning, yield stress, energy dissipation

DEGRADABLE POLYMERIC NERVE GUIDES PRODUCED BY FIBRE EXTRUSION, KNITTING AND ELECTROSPINNING

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Introduction: Injury to nerve tissue in the Peripheral Nervous System (PNS) results in long-term impairment of limb function, pain, often with associated psychological effects. Minor injuries can be left to regenerate. The challenge has resulted in the development of nerve guides or conduits produced from various polymers to improve nerve repair. This study describes the design and fabrication of a nerve regeneration conduit using polylactic acid (PLA) and (PLA):poly(lactic-co-glycolic) acid (PLGA) and Polyurethanes produced by direct Reactive Extrusion from HDI (hexamethylene di-isocyanate) and PLA and polycaprolactone polyols and short diol chain extenders. The nerve conduits produced consisted of : an electrospun fibre and knitted outer sheath, shaped profile guidance fibres surrounded by a neurotrophin-enriched alginate hydrogel.

Methods: Polymers and Nerve Conduit Design: The polymers were first evaluated for cell compatibility using petri dish evaluation methods. Assembly and Sterilisation: The extruded fibers and knitted/electrospun conduit sheath were fabricated separately to facilitate sterilization for implantation. Implantation Studies: The nerve guides were implanted into the 12mm gap in the hamstringsciatic nerve. Analysis of Regeneration: The rats were monitored for 4 weeks, euthanased and the conduit inspected for cell regrowth.

Results and Discussion: Our results support that the conduit restored some sensation in the axotomized limbs through support of effective repair of the transected sciatic nerve. However, more detailed functional analyses (eg nerve conduction) are still required to fully quantify the extent of functional repair promoted by the conduit designed in this study.

Keywords: Biomedical Polymers, Degradable, Nerve Guide, Reactive Extrusion, Fibre, Knitting, Electrospinning, Implant

PREDICTING THE SHAPE AND BOND STRENGTH OF INJECTION OVERMOLDED STAMP FORMED COMPOSITES

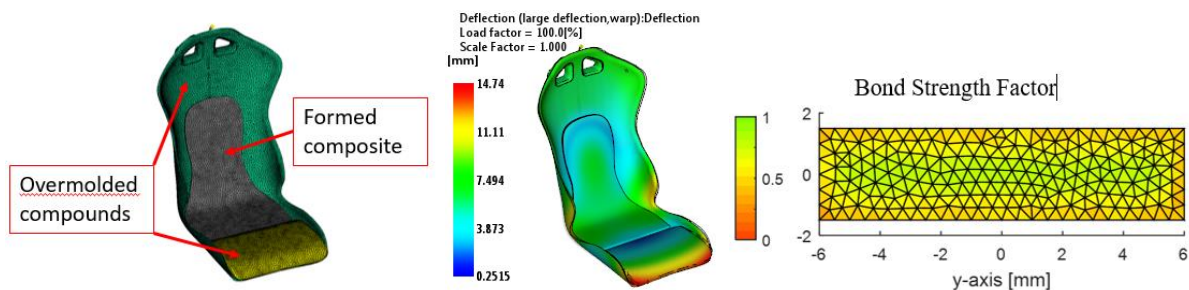
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Introduction: CAE tools are developed to assist the design validation of hybrid thermoplastic composite parts containing stamp formed continuous fiber composite sheets which are overmolded by an injection molding process to add functional design elements and reinforcements.

Methods: A method is developed for coupling the output (residual stresses, anisotropic properties and thickness changes) of an existing stamp forming simulation software tool (Aniform) into the combined finite element warpage simulation of the injection overmolding process (Autodesk Moldflow). The stamp formed composite is represented in the warpage simulation as a part insert, with local (elemental) anisotropic mechanical properties computed from the orientation of fibers resulting after the stamp forming process. In a similar way, the fiber stresses induced by the forming process are mapped into the warpage simulation as element level initial strains in the part insert (formed composite) as well as the thermo-mechanical strains which will occur in the insert due to cooling to room temperature. These combined initial strains, along with the in-mold residual stresses and cooling strains of the molding compound are the drivers of warpage in the combined part. A model of the interface bond strength between the stamp formed composite and the injection molding compound is also developed based on the temperature history at the bonding interface. Outcomes from the warp simulation and bond strength model will be compared to experimental results.

Results: A warp deflection result for a combined part having a formed composite inner frame and surrounding injection overmolded compounds is shown. The properties of the formed composite are anisotropic. A bond strength factor ranging from zero (no bonding) to one (full bond strength) is computed from the temperature history at a rectangular overmolding interface for a PA6 polymer. The perimeter of the interface shows lower bonding effectiveness due to the weaker heating of the insert in regions close to the (cooler) mold surface.



Discussion: Final part shape (warp) prediction is achieved for a combined stamp formed composite and injection overmolding process. A model for interface bond strength is available for use in a subsequent structural performance analysis.

Keywords: Injection overmolding, continuous fiber composites, warpage, bond strength

NEW RESINS FOR 3D PRINTING OF RAPID PROTOTYPES

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Introduction: Due to its speed and ability to make complex objects with minimal waste, 3D printing is predicted to revolutionise manufacturing. For this to occur, a wider range of polymer resins with a broader range of properties are needed. Here we report on our efforts to develop new 3D printable resins suitable of Digital Light Projection (DLP) 3D printers.

Methods: Formulation stability was measured by simple visual inspection over time. Cure time of the resin was investigated using photorheometer (ARES). A photocure 3D printer, Titan 1 (Kudo 3D) was used for all 3D printing. A gradient cure of a tube was used to determine optimum layer exposure time. Small and large parts were then printed to assess design reproduction. Metrology and physical properties of the 3D printed parts were investigated.

Results & Discussion: Translation of simple photocure chemistry to a DLP printer is not as straight forwards as expected. There are many factors which need to be considering when developing resins for DLP printers, namely: formulation stability, cure time for each layer, plastic deformation, successful initiation of crosslinking reactions, and inhibition of unwanted curing. Here we discuss these requirements and the interplay between them. Soft elastic materials are particularly difficult to 3D print due to their low modulus and ease of plastic deformation during the printing process. We have successfully printed soft elastic pasts (Fig. 1) using an in-house developed siloxane based resin as well as hard parts with high thermal stability using another in-house developed resin.

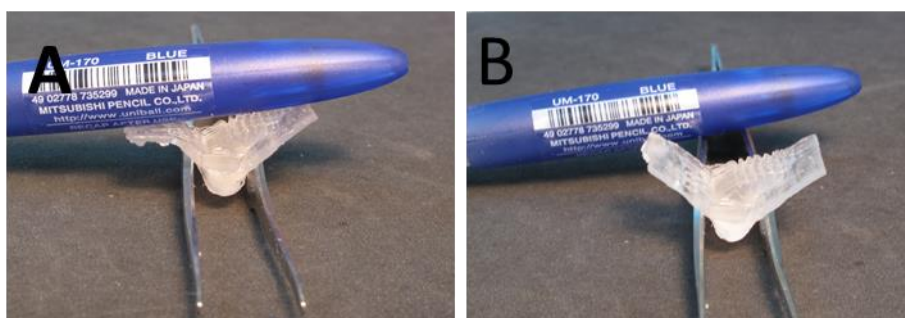


Figure 1: (A) Siloxane based 3D printed part deformed. (B) Siloxane based 3D printed part returns to natural shape post deformation, demonstrating its elasticity.

Keywords: 3D printing, rapid prototyping, photocure, siloxane, photorheology

NANOPOROUS OF PILLARS-LIKE STRUCTURE OBTAINED BY POLYMER BLENDS – PRECISE MOLDING SYSTEM

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Introduction: Recently, the fabrication of nanopillar arrays structure with the nanoporous surface (NPPs) has aroused great attention because of their applications such as sensing materials, drug releasing control, filtration materials, catalyst scaffolds etc. As compared to other fabrication methods, thermal nanoimprint combined anodic aluminum oxide (AAO) template has been proved to be outstanding performance technique for precisely, short time, and high efficiency. In order to produce the nanoporous structure, polymer blend with nano-domain segment having a great potential for the precursor materials that induced the porous by the selective removal of the nano-domain segment. However, it is still a challenge to fully understand the formation and the transformation of the obtained porous structure.

In this study, AAO-thermal nanoimprint was selected to produce NPPs of the polystyrene (PS) using PS/polyvinyl alcohol (PVA) blending materials. The PS/PVA blended having PVA micro/nano-domain size of the multiphase structure in PS matrix was prepared by the high shearing process. The porous structure was obtained using water-selective removal of the imprinted structures. Here, we mainly focused on clarifying the effect of PVA micro/nano-domain on the formation and the transformation of the obtained porous structure. Moreover, we have reported the hydrophobicity and friction property of the obtained structures.

Methods

1. **Materials:** Commercial grade polymers were used: PS1 ($T_g = 87\text{ }^\circ\text{C}$, MFR = 18.0 g/ 10 min at 200 °C GPPS 679; PS Japan Corp.) and PS2 ($T_g = 100\text{ }^\circ\text{C}$, MFR = 11.9 g/ 10 min at 220 °C, G210; Toyo Styrene). The PVA ($T_g = 26\text{ }^\circ\text{C}$, MFR = 4.0 g/10 min at 190 °C, CP-1210; Kuraray Co., Ltd.) was also used in polymer blend system.

2. **Experiment:** The high shear machine (NHSS2-28, Niigata Machine Techno Co., Ltd.) were used for blending process of PS/PVA (90:10 and 70:30 w/w %) materials by 500 rpm at 180 °C. The neat PS and PS/PVA films with the thickness of 500 μm were fabricated by hot/cold press method. The polymer films were imprinted with AAO-thermal nanoimprint machine (Izumi Tech) at 120-220 °C, 5.0 MPa for 30 min. The process diagram is shown in figure 1.

3. **Characterization:** The obtained structures of selected blended materials and nanopillars were observed by field emission scanning electron microscopy (FE-SEM). The chemical property of blending precursors was analysed by attenuated total reflectance (ATR), and the thermal property of obtained polymer films was characterized by modulated differential scanning calorimetry (M-DSC).

Results and Discussion: The transformation of the nanopillars structure were presented in Figure 2. The nanoporous could be obtained on the PS nanopillars surface after the water selective removal by using PS/PVA 90:10 w/w% precursor because of the blended precursor having the smaller PVA nano-domain size (30-1,000 nm) compared with 70:30 w/w% (300-1,000 nm) that able to flow in AAO. In the case of 70:30 w/w% was found the fibers breakup behaviour that nanofibers transformed to nanoparticles. Increase of the imprinting temperature could be induced the surface transformation of the pillars structures.

Keywords: Nanopillars, Nanoporous, Nanoimprint, Polymer Blends.

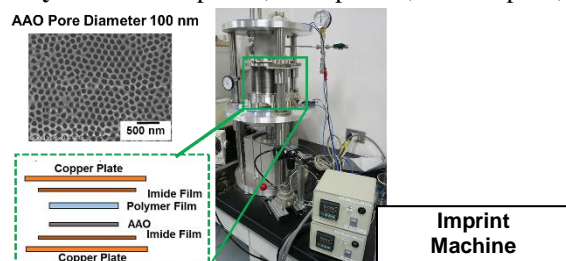


Fig. 1 AAO-thermal nanoimprint system

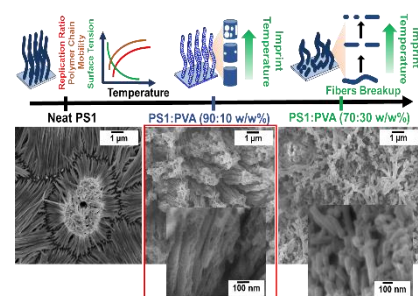


Fig. 2 Transformation of nanopillars structure.

PHYSICAL REGENERATION OF RECYCLED RESIN AND APPLICATION TO THE REAL CONTAINERS AND PACKAGING RESIN

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Introduction: The poor physical properties of material-recycled plastics are believed to be due to the chemical degradation that results from the heat, ultraviolet light or oxidation in the recycling process. Such chemical degradation is thought to be irreversible because it is associated with the breaking of molecular chains. However, our recent research indicated that the molecular properties of material-recycled plastics are restored to the same levels as those seen in virgin plastics.

In this time, we will report the processes to improve mechanical properties of pre-consumer recycled polypropylene (Pre-RPP) and real wasted containers and package plastics.

Methods: Table 1 shows the compressed conditions of pre-consumer recycled polypropylene pellet and real wasted containers and package plastics pellet (polypropylene : polyethylene = 5 : 5).

Table 1 Hot press and cooling conditions producing films.

Hot press conditions	210°C, 230°C, 250°C, 2min, 6min, 10min
Cooling conditions	Slow Cool (SC), Quench
Experiment contents	Tensile test, SEM observation

Results: Fig.1 shows results of Pre-RPP samples after tensile test. Mechanical properties of Pre-RPP improved dramatically by changing hot pressing and cooling conditions. Fig.2 shows samples of real wasted containers and package plastics after tensile test. For the real recycled resin, the mechanical properties also improved by changing the compression condition.

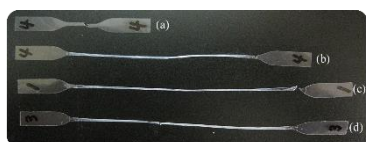


Fig.1 This picture of samples after tensile test.
(a) Pre-RPP 210 °C 2min Slow-Cool, (b) Pre-RPP 210 °C 10min Slow-Cool, (c) Pre-RPP 210 °C 2min Quench, (d) Pre-RPP 250 °C 10min Quench



Fig.2 This picture of samples after tensile test.
(a) 180 °C 2min Slow-Cool, (b) 180°C 2min Quench

Discussion: This result suggests that the main factor of the degradation of recycle plastics is physical degradation and we can regenerate the physical properties by selectin the molding condition. The results suggest that we can establish a new recycling process for the wasted plastics.

Keywords: pre-consumer recycled polypropylene / real wasted containers and package plastics / hot pressing and cooling conditions / mechanical properties / physical degradation

OC045

EFFECTS OF MICROSTRUCTURE ON THE MECHANICAL PROPERTIES OF HIGH DENSITY POLYETHYLENE FOR THE INJECTION MOLDING APPLICATION

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The high density polyethylene (HDPE) is one of the most commonly used thermoplastic polymers due to its low cost, good mechanical properties and versatility. The properties of polyethylene are strongly influenced by its molecular weight, molecular weight distribution, and branching. We developed the HDPE resins with high processability and performance by controlling the microstructure for the injection molding applications such as cartridge, pail, and bottle cap. In this presentation, we show the relationship between the microstructure and processability/performance of HDPE. The presence of long-chain branches in the high molecular part leads to significant shear thinning in viscosity, which reduces the injection molding cycle time. Such high processability, as compared to the conventional products, also allows for a further increase in the polymer molecular weight, improving the mechanical properties such as impact strength and environmental stress-cracking resistance (ESCR). However, when injection molded pails filled with water are vertically and horizontally dropped from a height 2.0 m, they crack at the side. It is found that this is due to the flow-induced alignment of polymers during the injection molding process. The proportion of high molecular weight tail is thus reduced to decrease the relaxation time of polymer, eventually allowing for resolving the crack issue of pails.

Keywords: polyethylene, injection molding, microstructure, mechanical properties

NETWORK STRUCTURES AND PHYSICAL PROPERTIES OF CROSSLINKED METALLOCENE EPDM

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Introduction: Ethylene-propylene-diene terpolymer (EPDM) is a synthetic rubber which is used for various industries, such as automotive, electrical, and construction industries. The olefin polymerization catalysts are necessary for the polymerization of EPDM. In 1953, the Ziegler-Natta catalyst was found. Industrialization of our EPDM by using the Ziegler-Natta catalyst began in 1969. And then, a new catalyst, metallocene catalyst was discovered in 1980. Industrialization of our EPDM by using the metallocene catalyst began in 2008. The metallocene catalyst has excellent performance in controlling branch structures of polymer chains. We can get branched EPDM by a metallocene catalyst and linear EPDM by another metallocene catalyst. In this study, we prepared linear EPDM and branched EPDM, and network structures of the EPDMs crosslinked by peroxide initiated reactions were investigated by small-angle neutron scattering (SANS) and small-angle X-ray scattering (SAXS).

Methods: Table 1 is a list of the EPDMs used in this study. Sheets of the EPDMs vulcanized by peroxide were obtained by compression molding. The sheets were immersed in deuterated xylene and swollen sufficiently. The network structures of the EPDMs in the sheets were investigated by SANS and SAXS. The SANS experiments were performed at the BL20 Materials and Life Science Experimental Facility of J-PARC under a user program (Proposal No. 2014AM0009). The SAXS experiments were performed at the BL03XU SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2013B7266, 2015A7216).

Results: Figure 1 shows the SANS profile. Branched-V-EP showed larger upturn scattering in the smaller-angle regions ($q = 0.02 \sim 0.05 \text{ \AA}^{-1}$) than other two linear EPDMs.

Discussion: The SANS result indicates that the network structures of the linear EPDMs are more homogeneous than those of the branched EPDM. We will show excellent practical properties of Linear-V-EP having homogeneous network structures at the venue of AWPP 2016.

Keywords: EPDM, metallocene, neutron scattering, X-ray scattering

Table 1. Characteristics of EPDMs used in this work.

Name	Diene type	Structure of polymer chains
Linear-V-EP	VNB	linear
Branched-V-EP	VNB	branch
Linear-E-EP	ENB	linear

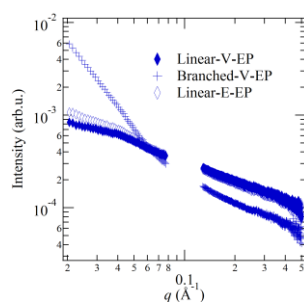


Figure 1. SANS profiles for samples shown in Table 1.

SYNTHESIS AND CHARACTERIZATION OF NOVEL POLYURETHANE ELASTOMERS CROSSLINKED BY ROLYROTAXANES

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NAGASAKI UNIVERSITY, NAGASAKI, JAPAN

Introduction: Crosslinking structures of polyurethanes (PUs) play critical roles for the appearance of their elastic properties. Polyrotaxane (PRX) is a macromolecule in which plural ring molecules are interlocked by a linear axle polymer with bulky terminal cap groups. One of unique properties of PRX is free sliding of the ring molecules along the linear chain. Therefore, introduction of PRX into the PU structure results in addition of the sliding effect. In this presentation, we describe the synthesis and characterization of PUs crosslinked by PRXs where the length and filling ratio of the ring molecule are different.

Methods: PRXs, which consist of α -cyclodextrins (CyDs) and poly(oxyethylene)glycols with different chain lengths (PEG1500, PEG4000, and PEG6000), were synthesized. The filling ratios of CyD are 75 % for **PRX1500**, 63 and 43 % for **PRX4000s**, 50 and 37 % for **PRX6000s**, respectively. The hydroxy groups in CyD were partially methylated to enhance their solubility. The PUs were synthesized by a prepolymer method. The prepolymer was prepared with 4,4'-diphenylmethanediisocyanate (MDI) and poly(oxytetramethylene)glycol 2000 (PTMG2000) with a ratio of $[\text{NCO}]_{\text{MDI}}/[\text{OH}]_{\text{PTMG}} = 3.0$. Subsequently, the prepolymer and the PRXs were reacted with a ratio of $[\text{NCO}]_{\text{total}}/[\text{OH}]_{\text{total}} = 1.5$. The name of PUs are abbreviated as **PUa(b)** in which a and b indicate the lengths and filling ratios of PRXs, respectively.

Results & Discussion: All of the PUs left their gel components in DMSO, indicating the existence of the crosslinks between the PRXs. ATR-FT-IR spectral results revealed that the formation ratio of hydrogen bond between the PU chains increased with increase in the filling ratio. DSC and dynamic viscoelastic measurements and tensile tests for the PUs revealed that (i) reorganized-crystallization of the PTMG chains of **PU1500(75)** easily occurred; (ii) the thermal and physical behaviours of **PU6000(37)** and **PU4000(43)** were almost the same because CyDs as the crosslink points disperse; (iii) **PU6000(50)** and **PU4000(63)** with the moderate filling ratios shows slow reorganized-crystallization of the PTMG chains. **PU4000(63)** also showed better tensile performance than the other PUs. In conclusion, we have demonstrated the synthesis and characterization of five PUs crosslinked by PRXs with different chain lengths and different filling ratios of CyD. Characteristics of the PUs depend in large part on the filling ratio of CyD in the PRX.

Keywords: Polyurethane elastomer, Polyrotaxane, Crosslink

SURFACE CONSTRUCTION ON THERMOPLASTIC ELASTOMERS WITH MICRO/NANO-FABRICATION TOWARDS BIOMEDICAL APPLICATION

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Introduction: The purpose of the abstract is to describe the preparation of blood-compatible biopolymers based on thermoplastic elastomers with micro/nano-fabrication and establish the basic mechanism for the selective reaction between blood and constructed surface of thermoplastic elastomers.

Methods: Micro/nano-patterning technique and reactive electrospinning were used to construct the surface on the thermoplastic elastomers to investigate the selective reaction between blood and the surface and develop blood-compatible biomaterials. The surface structure, blood-compatibility, selective reactions between the blood and biopolymer surfaces and function of the biomaterials were investigated.

Results: The surface on the thermoplastic elastomers was constructed with micro/nano-patterning technique and reactive electrospinning. The micro/nano-patterned surface and hierarchical architecture on blood-contacting surface were developed to reduce thrombus and hemolysis of red blood cells (Figure 1). The surface was also well manipulated to control the selective reactions between biopolymers and blood. The selective and specific reaction between patterned surface and blood plasma in the fresh blood (without adhesion of blood cells on the patterned surfaces) was realized to investigate the adsorption behaviour of blood plasma on the surface. Then, the selective reaction between the surface and blood cells was obtained to capture blood cells from the blood in the harmless manner. Finally, the smart selection reaction between blood and surface that could capture and release the blood components on the surface was achieved to establish the reaction mechanism of blood and surface/interface. Based on the fundamental research, we set up the new technology for preparation of blood-compatible biomaterials and some biomaterials had been applied in industry.

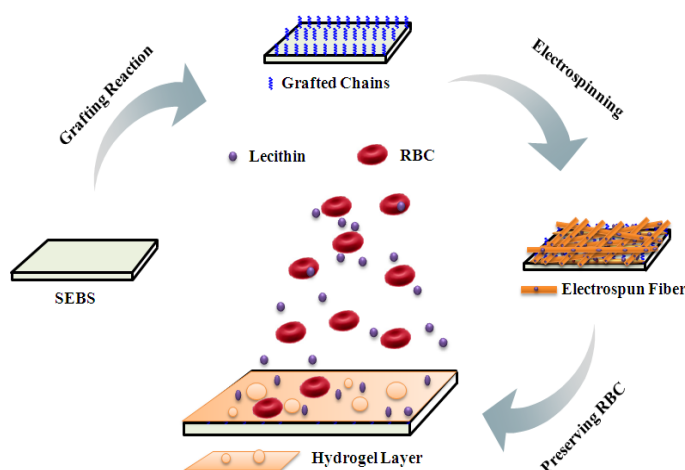


Figure 1. Constructing blood-compatible surface on thermoplastic elastomer with micro/nano-fabrication

Discussion: The current knowledge of blood-contacting biopolymers and devices were summarized. In addition, how the surface structure affected the selective reactions between blood and biopolymers, blood-compatibility of biomaterials were detailed discussed. The perspective of biomaterials prepared by micro/nano-fabrication was proposed.

Keywords: Micro/nano-fabrication, selective reaction, blood-compatibility

PHASE SEPARATION IN THERMOPLASTIC PROTEIN/POLYESTER BLENDS

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Novatein thermoplastic protein is produced from bloodmeal and is used for applications in the meat processing industry. Currently, it has insufficient impact resistance and energy to break for other applications. Blending Novatein with other polymers is a suitable strategy to overcome this. By tailoring phase morphology it could be possible to improve impact resistance and energy to break. Novatein was blended with poly(butylene adipate-co-terephthalate) (PBAT) in varying compositions between 2 - 30 wt. % PBAT. Poly(2-ethyl-2-oxazoline) (PEOX) dissolved in water was blended with Novatein prior to melt blending with PBAT using a twin screw extruder. Poly[(phenyl isocyanate)-co-formaldehyde] (pMDI) was used as a secondary compatibilizer added to the Novatein-PBAT blend before injection moulding. Uncompatibilized blends were also produced as a control. Uncompatibilized Novatein-PBAT blends show an asymmetric phase inversion whereby a co-continuous morphology is seen at low PBAT content. The coalescence of PBAT at low wt. % can be attributed hindrance of minor phase break up due to interfacial tension and a high viscosity ratio. In comparison, the addition of compatibilizer caused a reduction in PBAT phase domain size, and shifted the onset of co-continuity of the minor phase to a higher wt. %. This was attributed to the presence of the dual compatibilizer delaying the onset of minor phase coalescence. Novatein presents a unique challenge during blending with other polymers due to the abnormally high viscosity ratio which leads to the asymmetric phase morphology seen here and in other Novatein-polyester systems.

Keywords: PBAT, Novatein, Co-continuous, Phase Morphology

SMALL- AND WIDE-ANGLE X-RAY SCATTERING STUDIES ON METALLOCENE EPDM AND EBDM

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Introduction: Ethylene-propylene-diene terpolymer (EPDM), is a synthetic rubbers which is used for automotive, electrical, and construction industries. Industrialization of our EPDM by using the metallocene catalyst instead of the Ziegler-Natta catalyst began in 2008. The metallocene catalyst has excellent performance in copolymerization. For example, we can use a new monomer, butene instead of propylene, and get ethylene-butene-diene terpolymer (EBDM).

The new monomer butene influences crystal structures and amorphous structures, which results in excellent softness at very low temperature. In this study, these structures were studied by using synchrotron radiated small- and wide-angle X-ray scattering method and rheology.

Methods: We prepared EBDM and EPDM having various ethylene contents by using the metallocene catalyst. The glass transition temperatures (T_g s) of each polymer were measured by using differential scanning calorimetry (DSC). Sheets of each polymer were obtained by compression molding, and hardness (Shore-A) of each sheet was measured according to JIS. Small- and wide-angle X-ray scattering (SAXS and WAXS, respectively) measurements under uniaxial stretching were simultaneously carried out using the beamline BL03XU at SPring-8, Hyogo, Japan. The SAXS and WAXS measurements were conducted with the approval of SPring-8 (Grant Nos. 2011B7270, 2012A7219 and 2012B7269).

Results: When the T_g s were compared at the same ethylene contents, the T_g s of EBDM were about 10 °C lower than those same contents of EPDM. Fig. 1 shows WAXS patterns obtained at $\varepsilon_n = 0, 256, 523$ %. Here, ε_n is strain of uniaxial stretching imposed on specimen. In both EPDM and EBDM, scattering intensities along the direction perpendicular to the draw direction increased with increasing strain.

Discussion: Low T_g indicates that EBDM has excellent softness even in very low temperature. WAXS result indicates that strain-induced ordering occurred. Degree of the increasing of intensities was smaller in EBDM than in EPDM, indicating that EBDM is softer than EPDM under deformation.

Keywords: Ethylene-propylene-diene terpolymer (EPDM), metallocene, X-ray scattering

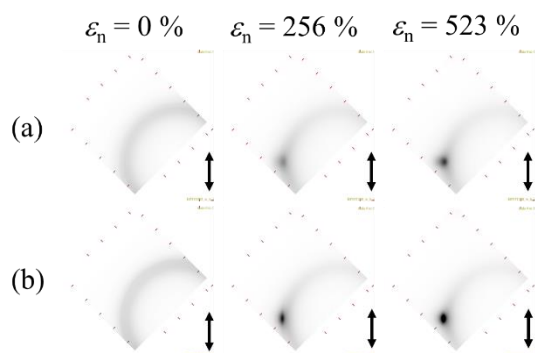


Fig. 1 2-dimensional WAXS patterns of EBDM (a) and EPDM (b) obtained at $\varepsilon_n = 0, 256$ and 523 %. Arrows indicate the draw direction.

OC051

A CASE STUDY IN CABLE EXTRUSION - UNDERSTANDING RHEOLOGY WITHOUT RHEOMETERS?

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Introduction: In wire and cable industry, polymer insulation and sheath are applied over conductors by extrusion. Specially designed “cross-heads” are used to continuously coat the wires or laid-up cores that move in a perpendicular direction, relative to the extruders. Rheology of polymer compounds utilised in this process needs to be understood, in order to optimise production speed and product quality. While developing a highly filled compound for a break-through fire resistant application, the material’s interesting rheological behaviour was studied. This allowed composition optimisation for improved processing.

Methods: A typical industrial R&D project was initiated after observation of batch to batch variation and poor processability of certain batches. Two significantly variant batches were analysed and their flow curves obtained by a capillary rheometer. The material’s extrusion behaviour was studied by using a Brabender Plasticorder / lab extruder, equipped with a cross-head. Modified compositions were compounded using a Buss kneader. Torque and pressure readings were taken and correlated to filler content.

Results: The batch with poor processing behaviour was found to be less viscous than the ‘good’ batch, as a consequence of a lower level of filler. Relatively quick QC methods were identified to assess these two properties of the studied compounds, namely %ash (solid residue after calcinations at 900°C) and melt flow index (MFI). Good correlation was found between MFI, extruder torque and die pressure. This was especially the case in a series where a single component was varied. More complex situation occurred when trying to correlate data from the whole study, where more than two components were varied.

Discussion: Based on this study, two goals were achieved: - a simple procedure was acquired for compound quality control and – a new compound was developed, with improved processing behaviour and ultimate properties. Apparent paradox that compounds with lower melt viscosity showed poor processing behaviour was explained by the need to achieve sufficient die pressure to exude low molecular weight components to the surface (external lubrication).

Keywords: rheology, extrusion, cable, insulation

A TALE OF TWO MOTORS

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A new rheometer platform based on two electronically commutated (EC) synchronous motors was introduced recently, representing a large step in rheometer innovation. By integrating two independently controlled air bearing motors on one rheometer, a range of testing possibilities become available (Fig.1). Combined motor transducer (CMT), separate motor transducer (SMT) and counter rotation are just some of the measurement modes to elucidate material behaviour across a large strain and stress range. The modular design allows the lower motor to be exchanged for another accessory much like a standard rheometer.

In the counter-rotation mode the motors are set to rotate in opposite directions. The pre-set speed is split between the two motors, whereas the torque and normal force are measured at the upper motor unit. This mode is an invaluable tool for microscopy applications – the creation of a stagnation plane allows the real-time observation of structural elements under shear fields (Fig.2). Further typical applications of the counter rotation mode are extensional rheology and the investigation of flow instabilities under different shearing conditions.

In the SMT mode the upper motor is operated solely as a torque transducer, while the lower motor functions exclusively as a drive unit. In this configuration it is possible to measure low elasticity contributions in samples with low viscosity, as well as extending the shear rate or strain range in measurements of polymeric samples.

The aim of this paper is to describe new technologies involved and to present measurement results and examples relevant for polymer rheology and dynamic mechanical thermal analysis (DMTA).

Keywords: Rheology; Stress controlled; Strain controlled; Counter rotation; Superposition

THE POLYMORPHIC CRYSTALLIZATION BEHAVIOUR OF POLYMER IN THE NANOPOROUS ANODIC ALUMINA OXIDE TEMPLATE

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Introduction: Recently, crystallization of polymer in the cylindrical templates has been widely investigated. Inorganic templates such as anodic aluminum oxide (AAO) membranes have been mostly employed. The studies can be divided into several categories. The first one is to study the nucleation mechanism of polymers in the nanopores. The second one is to study the orientation of the polymer crystals in the pores. The influence of nanopores on the crystal phase is rarely explored. Considering many polymers have polymorphic crystals which plays an important role on the final properties of materials, it is worthwhile to study the phase transition and polymorphic behaviour in the confined one dimensional spaces.

Methods: The polymorphic polymer was infiltrated into the AAO templates with different pore diameter and polymer nanotubes with different diameters were prepared. The crystallization and phase transition behaviour of the obtained polymer nanotubes capped in the nanopores have been explored by using X-ray and DSC.

Results: As for the poly(butylene adipate), it has α phase which form at high crystallization temperature and β phase which form at lower crystallization temperature. In the nonisothermal crystallization process, only α crystals form in the bulk sample. However, predominant β -PBA crystals form in the AAO templates with the orientation of b-axis parallel to the long axis of pore in narrow pores. During the melt recrystallization process, the critical temperature, below which pure β -crystals form, is 20 °C for bulk PBA. It drops down significantly with the decrease of pore diameter. Phase transition behaviour of β phase in the heating process depends on pore size. In narrow pores, it melts directly. Only in large pores, it has the chance to transit to α phase.

The poly(vinylidene fluoride) exhibits different crystallization behaviour in the AAO. In the nonisothermal crystallization process, only α crystals form both in bulk and AAO. The AAO template affects the nucleation types and melting temperature. The melting temperature of PVDF crystals in the AAO is much higher than that of bulk which can be attributed to the presence of an interfacial layer of PVDF on the template inner surface. The interaction between PVDF and AAO template produces the interfacial layer. In the melt recrystallization process, for the bulk, γ crystals show up when the crystallization temperature is above 158 °C. γ crystals are completely depressed in AAO when in the pore with the diameter is smaller than 200 nm.

Discussion: The phase transition and polymorphic behaviour of polymer in the confined one dimensional spaces can be well controlled by the AAO pore size, interfacial interaction and thermal treatment methods. Smaller size decreases the melting temperature and interfacial interaction improves the melting temperature.

Keywords: polymorphic behaviour; melt; AAO template; pore size; confinement

DIRECT PREPARATION OF NANOFIBER YARN BY NEEDLELESS MELT ELECTROSPINNING WITH ASSISTED SUCTION WIND AND ROTATING COLLECTOR

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Melt electrospinning is drawing more and more attention in recent years since it doesn't introduce any toxic solvent, is suitable for insoluble thermoplastic materials, and avoids any obvious whipping. Oriented nanofiber and even twisting yarn by melt electrospun fiber are urgently wanted for potential applications in high end textile, high sensitive sensors, and other anisotropic areas. In this work we proposed a needleless melt electrospinning with assisting suction wind and rotating collector for nanofiber yarn continuous and direct preparation. Well oriented nanofiber strands were prepared by applying suction wind as the multiple jets ejected from the self-made nozzle. Then continuous nanofiber yarn was successfully prepared after the strands were collated by collecting roller and a rotating disk (the base of the collecting roller). It was found the twisting degree of the yarn can be precisely controlled by the adjusting the ratio of speed of collecting roller to speed of rotating disk (SCTSR). X-ray diffraction (XRD) results showed that the crystallinities increased with the increase of the yarn degree.

melt electrospinning, needleless, nanofiber, yarn,

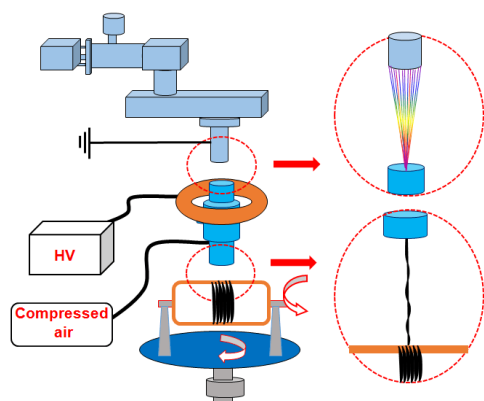


Fig 1 Schematic diagram of experimental set-up

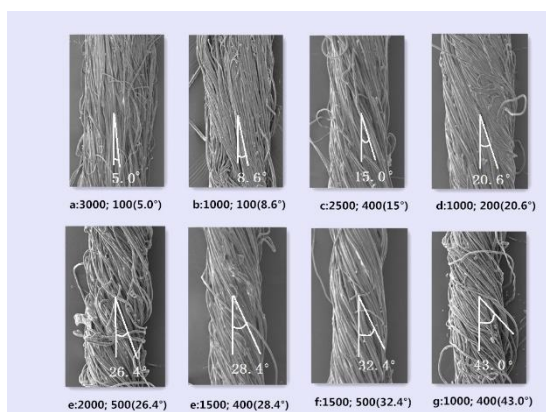


Fig 2 SEM images of several kinds of roller speed collection and rotation disk speed ($\times 500$)

CELLULOSE NANOFIBER-ASSISTED DISPERSION OF CELLULOSE NANOCRYSTALS@POLYANILINE IN WATER AND ITS CONDUCTIVE FILMS

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Recently, polyaniline (PANI) has received a high amount of attention from the nanoscience and nanotechnology fields. This is because PANI exhibits superior chemical sensing capabilities, unique photothermal effect, and thermoelectric effect, stem cell differentiation promoting effect, catalytic activity, and high electrochemical performance. However, PANI nanomaterials tend to aggregate and precipitate in water. Additionally, the application of PANI nanomaterials has been restricted by its poor ability to be processed and relatively poor mechanical properties. Therefore, finding a facile, efficient, and controlled method to disperse PANI nanomaterials is highly desirable undertaking. Currently, there are a number of studies that have focused on the dispersion of PANI nanomaterials. Methods for dispersing PANI nanomaterials have included controlling the reaction, controlling the PH of dispersion, doping it with phosphate ester, adding ionic liquid based polymer electrolytes, and adding poly(sodium 4-styrenesulfonate) .

The goal of this paper is to investigate the possible use of cellulose nanofibers (CNFs) as dispersants to disperse and stabilize cellulose nanocrystals@polyaniline (CNCs@PANI), which will result in the preparation of high strength and conductive films. It is potential applied to conductive ink, sensors, flexible supercapacitor, biomedicine, etc.

In this work, a facile and efficient method for dispersion of cellulose nanocrystals@polyaniline (CNCs@PANI) nanocomposites in an aqueous solution is reported by using cellulose nanofibers (CNFs) as a dispersant. To begin, the CNCs@PANI was prepared for the experiment by using CNCs as a template and controlling the reactions to obtain fiber-like nanocomposites. The nanocomposite was then dispersed in water with the CNFs by means of mechanical shearing. Centrifugal and storage stability of the nanocomposite were measured, as well as the zeta potential. The results showed that the CNFs had special dispersion assisting effects for dedoped CNCs@PANI. The CNCs@PANI dispersion showed good centrifugal and storage stability, and a zeta potential of -30.7 ± 0.2 mV was recorded with the addition of 10 wt% CNFs. This CNCs@PANI dispersion was casted to obtain a conductive film, which had tensile strength and electrical conductivity of 26.7 MPa and 104.7 S/m, respectively.

Keywords: cellulose nanocrystals, cellulose nanofibrils, polyaniline, mechanical property, conductivity

MECHANICAL PROPERTIES AND MICROSTRUCTURE OF POLYMER FILMS WITH LASER WELDING METHOD

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In this investigation, we demonstrate various polymer film sealing by using Laser Advanced Welding of Plastics (LAWP) method and try to evaluate the mechanical properties and internal structure of sealed parts. PE, PP, and PTFE films with 50 μm thickness were used as sealing films, and CO₂ laser was applied for laser welding of polymer films. As a result, it became to seal for PP film during 4.5-7.5 W of CO₂ laser irradiation power and for PE film during 2-4 W of that. Therefore, we suggest that some kinds of polymer films can be sealed by controlling the irradiation laser power. The relationship between mechanical properties and microstructure in sealed parts of polymer films will be discussed on the basis of the results of peel test, optical microscope, AFM observation, and micro-Raman spectroscopy.

Introduction: In recent years, laser welding has become established as a new joining technique in plastics technology, because it selectively exploits certain advantages over conventional methods. Particular mention can be made of the contact-free energy introduction to the laser welding methods that have been developed and the high flexibility that they frequently offer. It is expected that a number of these processes will not only replace conventional plastic joining processes in the future, but will also serve to open new fields of application and design that are made possible only through laser welding. Laser advanced welding of plastics (LAWP) method is one of the laser welding methods, and it is very excellent sealing method for medical plastic bags and various polymer films. LAWP method is with transparent heat sink in order to cool the excessively heated polymer films by high power laser irradiation. In addition, this method does not need to dope a dye or to set an absorption layer. Accordingly, the surface feature of sealed parts is clear and smooth as compared with general heat sealing methods.

Methods: PE, PP, and PTFE films with 50 μm thickness were used as sealing films. The CO₂ laser (Firestar V30, Synrad Co., Ltd., USA) was used, and the irradiation scans speed was 1 mm/s. The irradiation power was varied in the range of 4 to 15 W.

Results: It became to seal for PP film during 4.5-7.5 W of CO₂ laser irradiation power and for PE film during 2-4 W of that. PTFE film was also sealed as same as the power of PP films, although the peel strength was very weak as compared with PE and PP films. Therefore, we suggest that some kinds of polymer films can be sealed by controlling the irradiation laser power.

Discussion: Crystallization in sealed part of each film is promoted by irradiation energy of laser. Accordingly, the relationship between mechanical properties and microstructure in sealed parts of polymer films will be discussed on the basis of the results of peel test, optical microscope, AFM observation, and micro-Raman spectroscopy.

Keywords: Laser welding, CO₂ laser, polymer film, sealing

OC059

PLATEAU TO POWER STATION TO PADDOCK THE MIDLANDS IRRIGATION SCHEME

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Delivering water from the Highlands of Tasmania to transform drought prone grazing land in southern Tasmania is a complex engineering project on its own merits with economic benefits to small communities and family farms. Then delivering power for up to 5000 homes as the water travels down from the plateau, enhances the project above similar irrigation schemes.

The scheme started with the desire to deliver water 365 days per year to more populous areas in southern Tasmania, from an area with over 1000 mm of rainfall. From the source at Arthurs Lake, water is piped in 1.2 diameter high pressure gravity pipeline (60 bar) to a mini hydroelectric station (5.5MW) at the foot of the plateau at 12 bar. From the station, water is delivered via low pressure pipelines and natural waterways to farms to enhance agricultural land from lower value grazing to higher value cash crops and dairy.

David Kenworthy, managing director of Zezt Pty Ltd, recounts this engineering feat of the longest pipe line in Tasmania (124 km) transferring 38,500 ML of water from the Tasmanian Highlands at 900m AHD, irrigating 55,000 ha through HDPE pipe, DICL pipe, 5300 welds, 4800 tonne of polyethylene, 1 power station, pump stations and dams. The innovation used on the project has seen the scheme and its engineers recognized nationwide as an outstanding engineering feat, including individuals working on the project.

Keywords: Polyethylene, Tasmania, Engineering, Irrigation



FABRICATION OF ORGANIC FIELD EFFECT TRANSISTOR BASED ULTRAVIOLET SENSOR USING AZOBENZENE-CONTAINING FILMS AS GATE DIELECTRICS

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Introduction: Many research group have investigated tailoring OFET devices by incorporating functionalities into OFET devices for the advanced application such as gas sensor, optical switching, memory, and light-emitting devices. In this study, We functionalized gate dielectric in OFET device to have UV light sensitive property by using azobenzene derivative.

Methods: In 2011, Faupel group has reported that the capacitance of the azobenzene and PMMA blend film can be reversibly and largely changed upon irradiation with UV light. So, we presented a OFET device with azobenzene containing film as a photoactive gate dielectric layer for the UV photosensor application.

Results: We fabricated the azobenzene-containing film based OFET by simple method. The device showed reversible photo-response upon alternating irradiation with UV light and visible light as shown in Fig. 1.

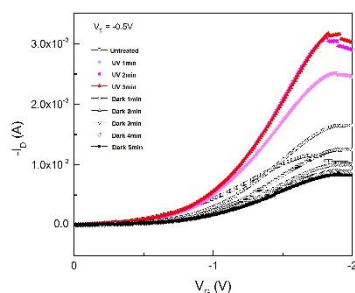


Fig. 1. Transfer characteristics of phototransistor under Dark and UV light.

Discussion: In this work, we successfully synthesized azobenzene derivative and prepared azobenzene-containing blend film. We fabricated OFET UV sensor by simple spin-coating method with the blend film as gate dielectric layer. The OFET device showed UV light sensitive property which can be used for UV sensor application.

Keywords: azobenzene, UV sensor, OFET

DEFORMATION BEHAVIOUR OF NOTCHED THERMO-PLASTIC ELASTOMER SPECIMEN -ANALYSIS OF YIELD STRESS - STRAIN RELATION -

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Introduction: An analysis of large deformation behaviours of polymeric materials is one of the important issues in design of molded products. However, it is difficult to explain large deformation behaviour of polymeric materials by conventional measurements, because of an onset of necking, and heat generation due to plastic deformation. Thus, we observed simultaneously true stress-true strain relations and heat generation in the tensile tests by using DIC (Digital Image Correlation) and thermal tracer for thermo-plastic polyester elastomer (TPEE) ^{1) 2)}. In the large deformation region, the stress-strain relation of TPEE is influenced by both strain rate and heat generation. On the other hand, in the small deformation region near a yield point, heat generation can be ignored, so that we can separate the influence of strain rate from that of heat generation. In this study, we report relationships between yield stress and strain rate of TPEE, and their dependencies on ambient temperature.

Materials and Methods: Thermo-plastic polyester elastomers (PELPRENE™ made by TOYOBO) with surface hardness D57 (ASTM D2240) were used for tensile tests (including high speed conditions). The specimens with R-shaped notch were used to control a necking position. Strain analysis using DIC was conducted by Visual Imaging Correlation-2D (VIC-2D) image analysis system. Digital gray scale images for DIC were obtained via CCD camera (GRAS-20S4M : Grasshopper). Thermotracer (TH9260 : NEC Avio infrared rays technology) was used for the temperature measurements. With using above setups, we observed the true stress-true strain relations with various tensile speeds. From these data under various ambient temperatures, we obtained the relationships between yield stress and tensile speed, and their temperature dependencies.

Results: Figure 1 shows true stress- true strain relations of TPEE. Stress has tendency to increase with increasing tensile speed. Figure 2 shows the relationships between tensile speed and yield stress in different ambient temperatures. For each ambient temperature, the yield stress tends to increase when tensile speed is raised. In addition, a point of inflection was observed in each ambient temperature. This positions move to faster side of tensile speed with increase of ambient temperatures, as shown by an arrow in Figure 2.

Discussion: The inflection points in the relationship of yield stress and tensile speed appear at the high-speed tensile conditions. These inflection points correspond to induction of yield mode having a faster relaxation time. Relaxation time of this fast yield mode depends on the ambient temperature. Thus, time-temperature superposition principle can be applied to analyse it. To explain these phenomena effected by both heat generation and strain rate, we have planned to numerical analysis in the future.

1) T. Kanatani et al., Preprints of Seikei-Kakou Annual Meeting '14, p.203 (2014).

2) Y. Isogai et al., Preprints of Seikei-Kakou Annual Meeting '14, p.205 (2014).

Keywords: Thermo-plastic elastomer, Thermal imaging, DIC, yield stress -strain rate

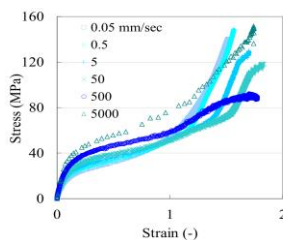


Figure1 True stress- true strain relations with various tensile speeds of TPEE.

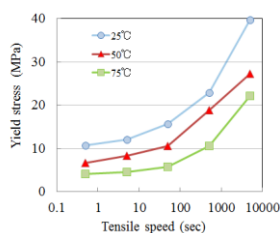


Figure 2 Relationships between yield stress and tensile speed in various ambient temperatures.

OC062

ADVANCED COOLING SIMULATION TECHNOLOGIES FOR THE INJECTION MOLDING PROCESS

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Introduction: Cooling circuit design in plastic injection molding has become the focus for improving part quality and reducing cycle times. In order to achieve both high quality parts and low cycle times; technologies such as, rapid heat cycling, conformal cooling, induction heating and thermocouple controlled cooling are commonly used.

Methods: This paper gives an extensive introduction to a 3D finite element method based solver which has been developed to simulate these molding technologies. Rapid heating and cooling cycling heats the mold surface prior to mold filling using steam, hot water or heater cartridges and cools the mold during the cooling phase with chilled water resulting in a very good surface finish without compromising the cycle time. In order to achieve the same effect but in a more efficient way induction heating uses an alternating electromagnetic field to heat the mold during filling. With conformal cooling, the cooling channels follow the precise geometry of the part in the mold. This aids in creating a uniform temperature distribution across the surface of the part by targeting hot spots. Thermocouple controlled cooling has a temperature sensor in the mold that controls the flow of the coolant in the cooling channels. This results in shorter cycle times and lower costs.

Results: Case studies of some of these technologies are presented in this paper and discussed.

Discussion: Advance cooling technologies can now be simulated with confidence by plastic injection molding engineers. The results presented in the paper comparing the results of the 3D finite element simulation models to actual experimental data show that an excellent correlation is achieved.

Keywords: Injection molding, Autodesk Moldflow, advanced cooling technologies