



# Symposium 15

## Foam Processing and Applications



## Effects of Dissolved Carbon Dioxide and Organoclay on the Crystallization of Poly(lactic acid) as Probed by Ultrasonic Measurements

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While low-density foam extrusion of amorphous poly(lactic acid) (PLA) has been successfully performed, foaming its semi-crystalline counterpart still remains problematic, since blowing agents such as carbon dioxide affect significantly the crystallization kinetics of PLA. Similar kinetics enhancement has also been observed with the addition of organoclays. The effects of dissolved CO<sub>2</sub> molecules on the crystallization kinetics of PLA, as well as the effect of addition of organoclay to the polymer, were investigated using an original device that combines ultrasonic and volumetric measurements. Contrarily to high-pressure DSC measurements, applied pressure and CO<sub>2</sub> concentration can be studied independently with this device. Ultrasonic parameters such as sound velocity and attenuation are very sensitive to crystallization and were thus used to monitor the crystallization kinetics. The crystallization rate was found to tremendously increase with a moderate addition of CO<sub>2</sub> and the results are compared with classical DSC measurements. Glass transition temperature was found to decrease non-linearly as the CO<sub>2</sub> concentration increases. Impact on foam processing is also finally addressed.

S15-915

## Fabrication of porous 3-D structure from poly(L-lactide)-based nano-composite foam via enzymatic degradation

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In order to prepare the porous three-dimensional (3-D) structure in biodegradable polyester materials we have conducted the enzymatic degradation of a poly(L-lactide) (PLLA)-based nano-composite foam having nanocellular structure, using proteinase-K as a degrading agent at 37 °C. The surface and cross sectional morphologies of the foam recovered after enzymatic hydrolysis for different intervals were investigated by using scanning electron microscope. The nanocellular took up large amount of water, which led to the swelling of the foam due to the large surface area inside the nanocellular structure, and facilitated the enzymatic degradation of matrix PLLA as compared with the bulk (pre-foamed) sample. Consequently, we have successfully prepared a porous 3-D structure as a remaining scaffold in the core part of the nano-composite foam, reflecting the spherulite of the crystallized PLLA.



## BIODEGRADABLE THERMOPLASTIC FOAMS FROM SYNTHETIC AND NATURAL RESOURCES

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Due to their unique properties, biodegradable thermoplastic foams have been widely used in applications that require good mechanical properties coupled with reduced density and allow the control over energy and mass transport, e.g. acoustic and thermal insulation, high energy or mass absorption, microfiltration and tissue engineering. The physico-chemical properties of the polymeric matrix and the architecture of the porous structure of these foams are key parameters in designing their performance, and therefore their potential applications. From the point of view of processing techniques, gas foaming has shown great potential in the design of the porous network of the foams, in particular porosity, pore size and degree of interconnection. This technique allows a fine control over the extension of porous pathway of the biodegradable foams by the modulation of the processing parameters and by using environmentally friendly, non-flammable and low cost blowing agents. In this study, we investigated the gas foaming of biodegradable polymers derived from synthetic, poly( $\epsilon$ -caprolactone), and natural resources, gelatin and zein, with the aim of preparing biodegradable thermoplastic foams characterized by well controlled porous micro-architecture. Before foaming, the natural polymers were processed to achieve thermoplastic materials by using suitable plasticizers and melt-mixing processes. The thermoplastic materials were subsequently foamed with mixtures of CO<sub>2</sub> and N<sub>2</sub> as blowing agents, in a batch foaming apparatus, at different temperatures, pressures and pressure drop rates. Results indicate the chance to prepare biodegradable foams with fine controlled porous architecture by the opportune selection of materials and foaming parameters. Moreover, the possibility to extend the results to obtain foams with multi-scaled porous architecture and enhanced interconnectivity was exploited by using PCL-TG and PCL-TZ heterogeneous blends.

S15-138

## Change in the Critical Nucleation Radius and Its Impact on Cell Stability

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The critical radius of cell nucleation is a function of the thermodynamic state that is uniquely determined by the system temperature, the system pressure, and the dissolved gas concentration in the polymer/gas solution. Since these state variables change continuously during the foaming process, the critical radius is varying at the same time despite the traditional concept that this critical radius is fixed as a thermodynamic property for a given initial state. According to the Classical Nucleation Theory, the critical radius decides the fate of the bubbles. Therefore, the change in the critical radius during foaming has a strong impact on the stability of the formed cells, especially in the production of microcellular or nanocellular foams. In this work, the continuous change of the critical radius is theoretically demonstrated under an isothermal condition while the pressure is dropping and the gas is diffusing into the nucleated cells. The experimental results observed from the visualization cell are used to support the theoretically derived concept. Sustainability of the nucleated bubbles is also discussed by comparing the actual bubble size and the critical radius.



## Novel cellular materials by foaming nanostructured blends and nano-reinforced polymers

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Cellular polymers are nowadays used in manifold areas, e.g. for light-weight structural parts and thermal insulation, but also for biomedical applications. Independent of their final use, both scientific and industrial approaches aim at controlling the cell size over multiple length scales, the foam density as well as the intrinsic properties of the polymeric matrix. Thus, one intends to comply with the steadily growing requirements towards the materials performance. In this study, the foaming behaviour of nanostructured polymer blends and nano-reinforced polymers was systematically investigated, as a promising approach to meet the demand for novel cellular materials. With regard to blends, immiscible blends of poly(2,6-dimethyl-1,4-phenylene ether) and poly(styrene-co-acrylonitrile) (PPE/SAN) were melt-compatible with triblock terpolymers. Furthermore, carbon nanofibre reinforced polyamide 6 (PA6) and SAN were used as nanocomposite materials. As processing techniques, batch foaming and foam injection moulding were performed. Electron microscopy provided a detailed insight into the foam morphology and the structure of the cell walls, and highlighted interesting features. Via controlling the morphology as well as the properties of the individual blend phases, a wide range of different foam structures became readily available, including bimodal cell size distributions. In the case of the nanocomposites, heterogeneous nucleation activity of the nanofibres led to significantly reduced cell sizes. In addition, the cell walls and struts were reinforced by nanofibres. The correlation with the morphology of the non-foamed material, and its thermal and rheological properties, finally allowed to establishing relationships between the foaming characteristics and the cellular morphology. In the light of these results, novel routes for processing sub-microcellular and nano-reinforced foams are derived by systematically exploiting the characteristics of multiphase materials.

S15-269

## Extruding PP foam sheets by means of a new blowing agent injection device

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In foam extrusion with physical blowing agents, long single screw extruders with a length in the range of 40 D are needed in order to obtain a homogeneous melt/blowing agent mixture and a proper melt temperature. By means of a new blowing agent injection device, which is mounted between extruder and die, standard extruders can be used for physical foaming of polymers. In first studies with this new injection device, polypropylene (PP) was foamed with carbon dioxide (CO<sub>2</sub>) as blowing agent. During these experiments, blowing agent content, nucleating agent content and die pressure were varied. The results show that fine-celled foam sheets with a density as low as 150 kg/m<sup>3</sup> can be achieved with this new technique.



## THE EFFECT OF MOLD TEMPERATURE ON MORPHOLOGY, TENSILE AND TORSION PROPERTIES OF INJECTION MOLDED HDPE STRUCTURAL FOAMS

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In this study, HDPE structural foams were produced by injection molding using different mold temperatures between 30 and 80°C to determine the effect of this variable on foam morphology: average cell dimension, cell density and skin thickness. Samples were also produced by setting independently the temperature of the fixed and moving plates of the mold to impose a temperature gradient while processing the foam. The resulting foams were also characterized in terms of mechanical properties including tensile and torsion tests. The results show that for homogeneous mold temperatures, skin thickness was uniform on both faces and increased with decreasing mold temperature. On the other hand, by keeping one mold face at a constant temperature and varying the other one, non-symmetric skin thicknesses were obtained. The degree of asymmetry was found to increase as the temperature difference between both molds increased. Furthermore, decreasing mold temperature was found to produce a small increase in average cell sizes while reducing cell density. As a general trend, both tensile and torsion moduli of the structural foams increased with increasing skin thickness.

S15-324

## Foaming of Thin Films of Fluorinated Ethylene Propylene Copolymers Using Supercritical CO<sub>2</sub>

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A versatile but not widely used process for manufacturing cellular polymers is the foaming by supercritical gases. This process was applied to fluorinated ethylene propylene copolymers (FEP) which has not intensively been investigated so far. In contrast to the amorphous polymers mainly researched in the literature, the selected FEP is a semi-crystalline material, i.e. its foaming is carried out in a partially-molten state ( $T_m \sim 260^\circ\text{C}$ ). The foaming behaviour was characterized in dependence on the variation of process and material parameters. The resulting density of the foam can be related to the pressure level during the saturation of the films with gas. The size of the cells generated mainly depends on the foaming temperature, whereas their number per volume is determined by the magnitude of the applied pressure. Furthermore, the pressure drop rate influences the resulting cell sizes in a way that significantly fewer but larger bubbles are found for longer times of depressurizing. Three linear FEP with different average molar masses were characterized. They showed a differing foaming behaviour with respect to the cell sizes generated and their dependence on the processing parameters temperature and pressure drop. These findings could be related to their diverse stiffness during the process. Optimized material properties and process conditions led to microcellular foam structures. Applying a pneumatic valve with a high pressure drop rate very thin films with thicknesses down to 50  $\mu\text{m}$  could be foamed. By a following biaxial stretching of the foamed films, a variation of the spherical to a lense-like bubble shape could be achieved. One motivation behind foaming thin FEP films is their promising application as ferroelectrets with high thermal and temporal stability, e.g. as membranes for audio systems. Due to a charging process in an electric field, these nonpolar cellular polymers are able to exhibit piezoelectric properties.



## Effects of Cold Crystallization on Cell Morphology in Microcellular Polyphenylene Sulfide Foam

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Microcellular of Polyphenylene Sulfide (PPS) was produced with carbon dioxide by a two-stage batch process. The emphasis of the study was paid to particularly on the effects of cold crystallization on the foam structure. The effect of cold crystallization on cell structure was investigated by varying foaming temperature in range of 353K, below the crystallization temperature of PPS to 523K, which was slightly lower than the melting temperature. The PPS foam shows a closed cellular structure with uni-modal cell size distribution in cell diameter when the foaming temperature was below the crystallization temperature and it shows a bimodal one when the temperature was above the crystallization temperature. The bimodal cell size distribution possesses one peak at around 10μm and the other at around submicron scale in diameter. We found that the growing crystals in cold crystallization process acts as an active nucleating agents of bubbles. Thus, the submicron cell structure was created by bubble nucleation in the heat quench foaming process when crystals were growing in the heating process.

S15-478

## Visual Observation of Polymer Foaming in Mold Cavity during Core-back Type Physical Foam Injection Molding Process

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Polymer foaming behavior in mold cavity was observed using a core-back type foam injection molder and a mold with visual quartz windows. A 35 ton Mucell machine was used to foam several kinds of polypropylene physically either with nitrogen and carbon dioxide. Foaming behavior together with cavity pressures and temperature was monitored through the windows and recorded by a CCD camera and a computer. The pressure profile in mold cavity was measured by ejector pin type pressure gauges and the temperature by infrared sensors equipped on the mold wall. The effects of core-back procedure and difference of physical blowing agents on foaming behavior was investigated. The obtained data showed some important behaviors of foaming process. The concentration of the physical blowing agent and the pressure depression rate played quite important roles for controlling the cell structure. The N<sub>2</sub> blowing agent provided larger bubble density and smaller bubble size than CO<sub>2</sub>. The foaming process can be optimized in accordance with polymer rheology through the information obtained by the above mentioned visualizing and sensing techniques.



## Foaming behaviour of HMS –PP/Clay nanocomposites

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Subhendu Bhattacharya, Rahul. K .Gupta, Sati.N. Bhattacharya\* Rheology and Material Processing Centre, School of Civil, Chemical and Environmental Engineering RMIT University, Melbourne, Australia Email: sati.bhattacharya@rmit.edu.au The foaming behaviour of HMS PP and HMS PP/Cloisite 20A clay nanocomposites was studied in a batch process. HMS –PP clay nanocomposites with 2, 4, 8, 10 weight percent clay were prepared in a twin screw extruder. The morphology of the nanocomposites was studied using XRD and TEM. Subsequently foaming experiments were conducted using supercritical CO<sub>2</sub> as the blowing agent in a batch process and foams with cell sizes varying from the sub micro meter to the micro meter range were prepared. The foam cell nucleation and growth of HMS PP based nanocomposites were studied at various clay contents. The effect of variation in saturation pressure, saturation temperature, foaming temperature, foaming time and quench temperature was determined experimentally. The physical relation of the above mentioned parameters to the final foam morphology was also determined. Dynamic rheological measurements were made to relate the influence of nanocomposite morphology on foam cell growth and nucleation. The foam structure was characterized using SEM and image analysis technique. Further a heterogeneous nucleation and a foam growth model was used to validate experimental results. It was found that the nucleation efficiency of clay reduces with increase in clay loading.

S15-638

## Optimisation of extruded polymer foam by the Resident Time Distribution approach

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We have studied the processing of polystyrene foam by extrusion. The blowing agent used is CO<sub>2</sub> and obtained by thermal decomposition of an organic molecule called Chemical Blowing Agent (CBA). To obtain an optimal foam, i.e. lower density, the process parameters of the extruder must be adapted to the CBA kinetic of decomposition. In this work, we used the Residence Time Distribution (RTD) approach to study the foaming during an extrusion process. A single screw extruder associated with a gear pump constitutes our extrusion line. The gear pump mounted between the extruder and the die was used to increase the pressure gradient in the extruder. The RTD is measured by transit experiments by injecting a small quantity of an inert tracer at the feed hopper of the extruder. The dye concentration evolution is analyzed continuously in real time by a light transmittance technique. The extrusion line with and without a gear pump is simply and quantitatively described by three continuously stirred tank reactors with recycling loops and one plug-flow reactor. The CBA kinetics of decomposition was quantified by measurement of the releasing volume of CO<sub>2</sub> with an home made apparatus. The range of pressure necessary for an accurate working of the pump strongly limits the domain of screw rotation speed explored. Nevertheless, the results in terms of mean residence times shown the efficiency of the pump. The extrusion process is correctly described by the RTD model. These results were confirmed by comparison of the density of the foams obtained and the theoretical density determined by the total decomposition of the CBA in the extruder. Moreover, it appears that the mean time residence is the critical parameter to optimize the foaming process.



## **Foam Extrusion and Dielectric Properties of Polyethylene Foams at medium and high frequencies**

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The increasing transfer rates in data communication has increased the performance demands on the insulation of communication cables. By foaming the insulation material, the dielectric constant of the insulation can be lowered. This paper addresses the influence of bubble shape on the dielectric properties of expanded polyethylene. Two commercial materials were extruded using a single screw Brabender laboratory extruder mounted with a slit die and a take-off system. Foams with different bubble shapes could be produced by adjusting the haul-off rate, distance from die to haul-off, temperature profile in the extruder and die temperature. Dielectric properties of the produced foams were measured and a relationship was found between the dielectric constant and bubble shape. The results showed an expected decrease in dielectric constant and electric loss modulus with increasing porosity. Flat bubbles showed a transition from a parallel equivalent circuit to a series equivalent circuit around 28% porosity for both 100 kHz as well as for 11.2 GHz. The electric loss modulus decreased with increasing porosity regardless of bubble shape both at 100 kHz and 11.2 GHz. These findings are important as a lower dielectric constant for a given porosity lowers the decay of a signal through a cable. Thus, less energy is used to transmit a signal. The dielectric losses derive from the polymer matrix, as the reduction in loss factor with increasing porosity suggests. Therefore, the dielectric behaviour of some selected commercially available materials was studied. Influence of molecular weight, molecular weight distribution, crystallinity, melting point, type of additive package, content of different short side chain branches, comonomer and catalyst type was evaluated against the dielectric properties of 11 commercially available materials measured at 1.8 GHz on compression molded plaques. The results showed a significant correlation with some factors and no correlation with others.

S15-810

## **The Investigation of the Effect of the Glass Microsphere on the Mechanical Properties of the Thermoplastic Foam**

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The microcellular closed structure of thermoplastics can be achieved by using a wide range of technologies for example injection moulding process with chemical or physical blowing agents. There is also possible to use different glass microsphere to create a microcellular structure with defined cell size. In the last years the recent researches had been demonstrated the mechanical properties of composites with glass microsphere in resins matrix, ceramic or metal. Because of their low density, higher isostatic crush strength and smaller size of the newest glass microsphere make them optimal alternative to conventional fillers for thermoplastic matrix. Due to the other benefits like reduce part weight, improved dimensional stability and flow, have a wide range of applications from aerospace and automotive to cosmetics, electronics as well as construction products. The effect of “foaming” on the morphology, density and mechanical properties of PP filled with different concentrations of microsphere are presented and discussed. It was investigated that the relationship between kind of glass microspheres, volume fraction and wall thickness of them and mechanical properties of thermoplastic foam. It was found that, their mechanical characteristics, tensile and flexural E-moduli depend on the volume fraction and spatial distribution of the microsphere in the finished part. Scanning electronic microscopy was applied to investigate microsphere properties such as morphology, shape, size, and distribution of them. It was found that with increasing of the glass microsphere contents in the polymer matrix, the flexural and tensile modulus increased.



## Morphological Characterization of Bio-Fiber Composite Foam

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As one of eco-friendly bio-fibers, wood-fiber has been incorporated in plastics to make wood-fiber/plastic composites (WPC) with increased stiffness and durability and with lowered cost. Foaming of WPC with a uniform, fine-celled structure can lower the density, increase the strength-to-weight ratios, improve insulating properties, and decrease the material cost. The cellular structure (i.e., cell size distribution) is closely related to their properties. In establishing the processing-structure-property relationship of WPC foams, it is necessary and helpful to quantitatively characterize the cellular structure for determining the functional relationship. However, there are very few studies on quantification of cellular structure in the literature. This paper presents a simple and effective method, based on 2D scanning electron micrograph, for quantitatively characterizing the cell morphology of WPC foams, in terms of cell size and cell size distribution uniformity. In addition, the 3D images of the foamed and unfoamed samples generated from a micro-computed tomography (MicroCT) scanner will be displayed to demonstrate the effectiveness of the proposed structural characterization method. Keyword: characterization, bio-fiber, composites, foam

S15-1092

## Characterisation of Low Density Polyethylene Foams Modified with Montmorillonite

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Consideration is given to the influence of nanocomposite filler on the structure and properties of low density cross-linked polyethylene foams, made by a nitrogen gas expansion process. Formulations were prepared containing 0, 5, 10, 15 and 20 wt. % additions of maleic anhydride grafted LDPE compatibilising agent together with up to 5 wt. % montmorillonite. All samples were crosslinked using a peroxide crosslinking agent. Polymer crystallinity was determined using XRD, which together with TEM also revealed levels of clay exfoliation in the materials before and after foaming. Image analysis of SEM micrographs was used to quantify cell density and cell wall thickness within the foams. DSC revealed changes in sample crystallinity with the addition of the nanoclay. DMTA, hot modulus and compression testing were used to examine the modulus of the materials, which in some cases surpassed their conventional non-reinforced counterparts.



## INJECTION FOAM MOLDING OF WOOD-FIBER/PLASTIC COMPOSITES

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This paper investigates the feasibility of injection molded wood-fiber/high-density polyethylene (HDPE) composite foams that substitute for injection molded HDPE solids in industrial applications. The study compares physical properties and cost of wood-fiber/HDPE composite foams with those of solid HDPE. The experimental results show that wood-fiber/HDPE composite foams with 20% weight reduction have superior physical properties, such as density, dimensional properties and mechanical properties. Furthermore, the cost analysis exhibits that wood-fiber/HDPE composite foams are much less expensive than HDPE. Therefore, it is concluded that wood-fiber/HDPE composite foams are strong candidates that can replace current injection molded HDPE products.

S15-1130

## Mechanical Property Characterization of Highly Porous Injection-Molded Biodegradable Polymer Matrices

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A novel approach has been applied to injection molding to manufacture highly porous biodegradable polymer foams that have the potential to act as tissue engineering scaffolds. In order to determine relevant application areas for the resulting polylactide foams of approximately 80% porosity, mechanical properties such as compressive strength and modulus of unfilled and filled foams is presented. The filled foams contain varying amounts of hydroxyapatite, a mineral that is naturally occurring in bone and provides bone's compressive strength. The incorporation of hydroxyapatite combined with this unique injection molding process may allow for highly porous biodegradable matrices with mechanical properties similar to that of bone to be manufactured cost-effectively with complex geometries. This research has the potential to significantly impact current therapies available for orthopedic applications.



## Physical Blowing of an Epoxy Foam

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Foams are ubiquitous low density materials used for a variety of applications including shock, thermal, and vibration isolation of electronic component, disposable containers, and energy production. Despite their many uses, foams are still not well understood at a fundamental level. Two major categories of foam exist: chemically blown foams and physically blown foams. Chemically blown foams expand via reactions that produce a gas phase during polymerization, e.g. polyurethanes, while physical blown foams begin with a dissolved blowing agent that boils to produce cells either by increasing the temperature or decreasing the pressure. For our applications, we are interested in a blown foam that starts off as an emulsion of Fluorinert blowing agent in epoxy monomer and curative. Once this emulsion is formed, the foam precursor is injected into the mold and inserted into an oven to boil the Fluorinert and produce foam. The complex interplay between heat transfer, polymerization, boundary conditions and nucleation of Fluorinert can make predetermination of the final foam density and amount needed to fill the mold difficult. A series of experiments, ranging from temperature instrumented flow visualization studies to examination of single droplets, have been undertaken to understand the likely nucleation mechanisms, effects of air entrained during mixing, foam rise rates under different conditions, and the flow properties of the rising foam. With input from these experiments and microscale models, we are developing a homogenized continuum-level model based on a finite element discretization to help understand and predict the foaming process. \* Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

S15-172

## Microwave assisted foaming of melamine-formaldehyde resin

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Polymeric foams have gained scientific interest in modern technology due to their light weight, excellent strength/weight ratio, superior insulating abilities, energy absorbing performance and comfort features of polymeric foams. Foams can be prepared from virtually any polymer, however due to the unique properties of melamine-formaldehyde (MF) resins their foams can be applied in demanding environments. MF resins are dimensionally stable under a wide variety of conditions due to their rigid network structure, which is formed by curing. To produce a foam from a thermosetting polymer the reactants must be foamed while only partially reacted and still fluid, followed by curing to solidify and stabilize the foam. For the purpose of foaming physical blowing agents, such as low-boiling liquids, are commonly used. To enable the foaming (boiling agent evaporation) and curing (MF condensate curing) process an external heat source must be used. Conventionally used convectional heating provides high temperature gradients inside the growing foam resulting in its non-uniform structure and consequently its poor applied properties. To avoid such problems the use of microwaves as an alternative heat source has been proposed. In the present study, the microwave assisted foaming process of MF condensate was studied and compared to the convectional heating process. The composition of the foaming system, which consisted of MF condensate, catalyst, emulsifier and blowing agent, and the process parameters were varied to study the effects of heating source on foam microstructure and mechanical properties. By the use of microwave heating uniform foams were obtained and their properties were controlled by the intensity of the microwaves.



## Zero Flash Micro-Embossing

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It has been demonstrated and studied by several investigators that embossing with localized heating of conventional polymer substrates can generate relatively high quality features, but in all cases, flash is generated. In this work, foamed substrates were studied using localized heating and the resulting features were free of flash. In this study, ultrasonic heating was selected and features embossed on several different microcellular foamed polymer substrates. Samples were fabricated to mimic standard compact discs (CD) that could be used for bio-sensing. Patterns of micro features were designed and developed to function as devices such as valves, mixing chambers for sample/reagent loading.

S15-247

## Batch-foaming of multiphase blend systems varying the viscosity ratio

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Polymer foams face steadily increasing requirements demanding for the development of novel materials. The aim of the present study was to systematically investigate the foaming behaviour of multiphase blend systems. As a reference system, two phase blends composed of poly(2,6-dimethyl-1,4-phenylene ether) (PPE), polystyrene (PS) and poly(styrene-co-acrylonitrile) (SAN) were selected. As PPE and PS show complete miscibility over the whole compositional range, but immiscibility with SAN [1], the viscosity ratio as well as the difference in glass transitional behaviour between the two phases (PPE/PS and SAN) could be systematically controlled. In order to obtain cellular blends, batch-foam processing was performed as a function of foaming temperature and time, using carbon dioxide as physical blowing agent. Subsequently, the morphology of the unfoamed blends, as investigated by transmission electron microscopy (TEM), was correlated to the rheological characteristics and the structural features of the foamed material observed by scanning electron microscopy (SEM). In addition, a more detailed foam analysis was performed including the evaluation of the cell size and the cell size distribution. This analysis highlighted a multimodal cell size distribution, which results of the difference in cell nucleation and growth of both blend phases. Based on these results, pathways for tailoring cellular polymers were derived. As a next step, the up-scale from batch-foaming to foam extrusion will be studied in order to investigate the transfer to commercially relevant processing techniques. Reference: [1] Hachiya, H.; Takayama, S.; Takeda, K. Journal of Applied Polymer Science, 70, 2515 (1998)



## Mechanical properties of a co-injection molded structural TPO foam

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We examined the influence of the morphology at the interface of a co-injection molded TPO foam part. Co-injection molding was selected to manufacture a foamed part with 20% reduced weight while maintaining a Class A surface finish. A thermoplastic polyolefin (TPO) comprised of 70% isotactic HiVal polypropylene (HiVal, MFR 12) and 30% Engage elastomer (Dow Chemical) was prepared on a 27mm Leistritz twin-screw extruder. The endothermic chemical foaming agent used was Hydrocerol HK40E (Clariant), supplied in masterbatch form. Apparent density was calculated using the volume displacement method. The flexural modulus was determined using the 3-point method according to ASTM D790. The tensile modulus and strength were determined according to ASTM D638. Impact behavior was studied using a dart impact tester by fracturing a part at room temperature, and comparing the internal structure to that of a brittle fracture, in order to determine the fracture mechanism for the part. The foam structure was characterized by cell diameter, skin thickness, and cell density using an optical microscope. Scanning electron micrographs were obtained from gold-sputtered cross-sectional surfaces that were exposed by cryogenic fracture. It was observed that mechanical properties could be dramatically affected by the core's cellular structure close to the interface of the A-B-A sandwich composite. SEM analysis shows that at room temperature, impact fractures have some plastic deformation, and occur within the cellular core structure. The density of the parts examined had a maximum density reduction of nearly 40%, however, only density reductions of less than 20% maintained most or all of their tensile and flexural moduli, and were as visually appealing as a similar, solid injection molded part. The co-injected foamed core parts were similar to those of integral foam, with the exception of the location of the cracks.

S15-310

## Mechanical behavior of asymmetric structural foam

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Mechanical behavior of structural foams is directly related to their morphology. In the past, parameters such as skin-core thickness and density reduction were used. In this work, careful studies on the complete morphological characterization of symmetric and asymmetric structural foams were performed using density profiles through X-ray densitometry. The mechanical properties in tension and flexion were also measured and compared for both types of structural foams. The results show that the direction of applied load has a significant effect on the mechanical properties of asymmetric foams while this is not the case for symmetric ones. Some models are presented and discussed in order to predict the measured mechanical properties.



## Micro Bubble Letters on Plastics – a technique of ink-free printing

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The letters and lines are printed on plastic films and plates by microscale size bubbles. The microscale bubbles are created by focusing the laser irradiation power on the plastics in which CO<sub>2</sub> or N<sub>2</sub> is dissolved a priori. The strong light reflectability of microcellular structure provides the bubble letters whiteness, high contrast and clear shape. The effects of laser irradiation power, scanning rate (irradiation time) and dissolved gas concentration on bubble density and size were investigated. We found that the cell size decreased and the cell number density increased as laser irradiation power decreased, the high CO<sub>2</sub> gas concentration increased or molecular weight of plating polymer increased.

S15-486

## Development of Polystyrene/ nano-CaCO<sub>3</sub> composite foams

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Foaming of Polystyrene and Polystyrene/ nano-CaCO<sub>3</sub> composite foams has been studied using a Supercritical Fluid technique. For preparing microcellular composite foams, 0.5%, 1% and 3% nano-CaCO<sub>3</sub> were mixed with pure Polystyrene. Morphology of cell size and cell distribution of different concentrations of these Polystyrene/ nano-CaCO<sub>3</sub> composite foams were compared with those of the pure Polystyrene foam. Solubility of liquid CO<sub>2</sub> under supercritical condition in Polystyrene/ nano-CaCO<sub>3</sub> composite foams with soaking time at 1, 6, 12, 24 and 48 hrs were compared. The number of bubbles/cm<sup>3</sup> of polymer increased and the cell size decreased with increasing in concentration of the intercalate Polystyrene/ nano-CaCO<sub>3</sub> composite foams.



## EFFECT OF COMPOUND FORMULATION ON THE PRODUCTION AND PROPERTIES OF EPOXIDISED NATURAL RUBBER (ENR-25) FOAMS

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Epoxidized Natural Rubber (ENR-25) formulations were compounded and tested for the feasibility of obtaining stable expandable rubber foam as well as to determine the foam cell physical morphology and its mechanical properties. Several formulation parameters were varied by employing different ratio of rubber blend between ENR-25 and natural rubber (SMR-L), different amount of blowing agent which is Sodium Bicarbonate and different ratio of accelerator between Tetramethylthiuram-disulfenamide (TMTD) and N-cyclohexyl-2-benzothiazolsulfenamide (CBS). Cure characteristics of the compounded rubber were determined using Monsanto Rheometer. The rubber foams were produced using compression molding technique with utilization of heat transfer process. The generated foam cell morphology was analyzed using image analyzer and SEM while the mechanical properties of the foams were examined by using Instron machine to determine their compression stress-strain curves. Beside that, swelling test using the Flory-Rhener equation was also conducted to measure the crosslink density of the rubber and subsequently used to support the mechanical properties results. The results showed that at the ratio of 60 phr ENR-25 and 40 phr SMR-L, stable rubber foams could be produced. Furthermore, increasing the amounts of blowing agent evidently increased the foam cell size, inducing smaller cell in between the foam cell wall, decreasing the value of compression stress and compression set. It was also discovered that increasing the ratio of CBS in the rubber compound will definitely increase the cell size, creating thicker foam cell wall, increasing the value of compression stress and compression set.

S15-664

## Processing of PP based Composite Foams in an Injection Molding System

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A continuous production of polypropylene (PP)/carbon fiber composite foams is achieved on an injection molding system. In this work, a conventional injection-molding machine was modified and gas (N<sub>2</sub>) was injected into the upstream side of a special injection nozzle consisting of static mixer and a shut-off nozzle to form a single-phase solution as against barrel injection where the screw and barrel were modified for gas insertion. The foams were generated due to the thermodynamic instability i.e. sufficient pressure drop from the plastification unit to the mold to assist rapid nucleation, duly controlled by process parameters. The effectiveness of foam molding system developed in this study was verified. In particular, the effect of injection speed and pressure on cell morphology of PP composite foams were examined and discussed. It is observed that cell size decreases and cell density increases with an increase in injection speed.



## **Effect of Processing Parameters and Fiber Orientation on the Mechanical Properties of Microcellular Polypropylene (PP) /Glass Fiber Composites**

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In this study, the effect of processing parameters and fiber orientation on the mechanical properties of microcellular polypropylene (PP)/glass fiber composites are investigated. In microcellular injection molding, nitrogen (N<sub>2</sub>) gas at super critical level is used as a physical blowing agent into the molten polymer and thereby injected into the mold to produce microcellular-foamed samples. PP neat resin and 30% glass fiber filled PP were injection molded into ASTM test bar samples with both conventional and microcellular injection molding system. These samples were then subjected to scanning electron microscope (SEM) imaging as well as tensile and impact testing to study how the process conditions and presence of fibers affect the cell structure and mechanical properties of the microcellular injection molded samples. The detailed discussion leads to the effect of fibers on cell size as well as cell distribution. The orientation of fibers also affects the mechanical properties as well as cell growth in the vicinity of fibers.

S15-757

## **ELASTIC PROPERTIES OF PACKAGING CUSHION FOAM : VISCOELASTIC RELAXATION IN THE LONG TIME LIMIT AND CUSHION CURVES**

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The compression set of the cushion foam for packaging is very important to characterize the behavior of the material in the long time limit. Actually, a compression set experiment is a viscoelastic relaxation stress experiment from a rheological point of view. In the present work, compression set experiment was thus simulated in a usual stress controlled rheometer by applying a different compression uni-axial strain. The time dependent variation of the stress was recorded over ten hours from the normal force transducer. The frequency spectrum and the relaxation behavior are studied for different compression strain. Furthermore, it is well known that elasticity properties depend on the structure of the foam, the size or air cells. However the microstructure of those cells is another sensitive parameter. In order to understand the influence of these two parameters on the final elastic, different types of foams usually used in packaging. The Young relaxation modulus  $E(t)$  was modeled thanks to physical laws in foams. The compression set behavior was then predicted at different time of loading. The results were compared between the different materials and qualitatively explained by considering the microstructure of the foams. And to conclude this work, those rheological experiments were compared to classic tests in packaging for packaging wedging foam like the cushion curves. We will thus compare the dynamic damping to the compression set under static stress.



## SYNTHESIS OF NOVEL HIGHLY POROUS BIOMATERIALS BASED ON ALGINATE

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Alginate is a negatively charged polysaccharide widely used both in industrial and biomedical field, due to its ability to form stable hydrogels, to its biocompatibility, biodegradability and non-immunogenicity. The preparation of highly porous monolithic biomaterials as supports for cell (or tissue) cultures is an emerging area of research activity. We have developed two novel methodologies to prepare highly porous scaffolds based on alginate, characterized by widely interconnected cavities (1-2). First procedure is based on high internal phase emulsion (HIPE) technology (polyHIPEs). To improve the biocompatibility of the process and the morphology of alginate polyHIPEs, we tested the novel gas foaming methodology. Macroporous supports can be formed by incorporating in situ developed inert gas (CO<sub>2</sub>) bubbles within a biopolymeric solution and its following cross-linking. In both cases, we employed two biocompatible cross-linking methodologies: the calcium-induced gelation and the auto-cross-linking (EDC/NHS system mediated). Moreover to improve the alginate scaffolds affinity towards hepatocytes, we introduced galactose moieties on the polymeric chains, which are recognized by specific hepatic receptors. The average cavities and interconnections sizes of alginate PolyHIPEs, valuated by SEM and optical microscopy, resulted to be of 80 and 20 mm respectively. Using gas foaming methodology, we obtain scaffolds with bigger average pores and interconnections sizes of 260 and 80 mm respectively. Hepatocytes growing tests demonstrated, by cellular adhesion and proliferation, a very good alginate scaffolds biocompatibility and high degree of colonization. REFERENCES: 1 E. Barigelli, Tesi di laurea in Chimica Industriale: "Sintesi e caratterizzazione di materiali altamente porosi a base di alginato", 2007. 2 A. Barbetta, M. Dentini, Italian Patent RM2003 A00037, (2003), 1-43.

S15-855

## TWO DIFFERENT ROUTES FOR THE PRODUCTION OF HIGHLY POROUS SCAFFOLDS FOR TISSUE ENGINEERING APPLICATIONS

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The results of a research project, concerning the synthesis and characterization of novel biomaterials (scaffolds), suitable for tissue engineering applications, are reported. Two different techniques have been used for the production of biopolymeric scaffolds of monolithic type (polyHIPEs) or solid foams. PolyHIPEs with highly porous and fully interconnected morphology have been synthesized by means of emulsion concentrated techniques (HIPE) starting from biopolymers like gelatin, hyaluronic acid and chondroitin sulfate. Two different procedures of biopolymers cross-linking have been adopted: radical polymerization of methacrylate functionalities, previously introduced onto the polymer chains, and enzymatic cross-linking promoted by the microbial transglutaminase. These matrices have been tested in the culture of hepatocytes for liver regeneration. All the scaffolds are resulted suitable for cell colonization but those obtained by radical polymerization have a better defined and more robust structure, while the enzymatically cross-linked matrices result in less cytotoxicity and the hepatocytes express a better differentiated phenotype. However from an application point of view, these polyHIPEs can be use only for some tissue regeneration because the average pore and interconnect diameters are 35-40 µm and 15-20 µm respectively, as valuated by optical microscopy. We have improved the morphology using a new technique based on the in situ generation of inert gas (N<sub>2</sub>) inside an aqueous solution of a biopolymer (gelatin) and carrying out its reticulation reaction in the aqueous phase. This process allows the synthesis of porous scaffolds characterized by pores and interconnects of 250 and 100 µm respectively. The foams have been tested in the culture of mesenchymal stem cells which have great potential in bone tissue engineering.



## Expandable polystyrene beads for monitoring of bed fluidization

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In this work, expandable polystyrene beads were used to determine of residence time in a fluidized bed. HDPE is usually prepared in fluidized bed reactors. Distribution of residence time actually affects on final properties of obtained high density polyethylene specially on its molecular weight. By using of expandable polystyrene beads and amount of expanding of beads through the bed, it is possible to monitor residence time and residence time distribution of particles inside the bed.

S15-1200

## Structural foams having moderate thickness and ultra-low density prepared by a new injection molding process

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This paper is going to present new achievements in the field of structural foam moulding concerning the achievable density range. Typical parts prepared by low-pressure structural foam moulding offer small density reductions in the range below 20 %. High pressure techniques like the breathing mould technology allow 3-fold expansion factors, but only if the initial part thickness is not below 2 to 3 mm, which leads into parts offering an overall thickness of 6 to 9 mm and a high mass per unit area. In foam extrusion or bead-foams, densities in the range of 100 kg/m<sup>3</sup> and lower are state of the art. In injection moulding, these limitations can only be overcome if one could control the relevant physical foaming conditions like the foaming temperature, the pressure and the volume given for expansion. So far, that was not possible. In the newly developed structural foam moulding process a mould design was chosen, fulfilling the above mentioned requirements in terms of foaming conditions. A breathing mould as well as gas-counter pressure allow the control of volume and pressure of the gas-laden melt. In conventional injection moulding the mould temperature is typically kept low, to achieve good and fast cooling of the moulded part and to limit cycle time. But cold moulds induce temperature gradients in the melt, leading to inhomogeneous cell structures and the expansion ratios are limited due to the prematurely freezing melt. Active and fast changes of the mould temperature between melt and room temperature in each cycle helps to overcome these restrictions. Hence, one can avoid the solidification of the polymer when foaming, which opens a completely new field in density range for structural foam moulding. This allows moulding of parts with densities in the range of 100 kg/m<sup>3</sup> at a relatively thin final part thickness. These low-density parts are pretty attractive in terms of light-weight automotive parts as well as for durable packing applications.



## **Foaming of PP composites filled with cellulose fibers**

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There is an worldwide increasing interest in the light weight materials for transport applications, which results from a shortage of crude oil availability and high prices of this raw material. In parallel to an increasing implementation of plastic parts at automotive industry an increasing extent of research has been focused at the innovative solutions and applications of cellular plastics. Foaming of PP composites filled with cellulose fibers has been investigated. The parameters of a cellular structure generation in these composites were investigated. Influence of selected modifiers on the melt strength, foam structure and mechanical properties have been discussed and possible improvements at the foaming technology were suggested.

S15-1282

## **Polymer/Organoclay Nanocomposite Foams with Interconnected Cell via High Internal Phase Emulsion Polymerization**

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Reinforced open-cell microstructured foams have been prepared by the polymerization of high internal phase emulsions incorporating inorganic thickeners. Organoclays were used as oil phase thickener, and sodium montmorillonite was used as aqueous phase thickener. Rheological properties increased as the oil phase thickener concentration and agitation speed increased, due to the reduced drop size reflecting both a competition between the continuous and dispersed phase viscosities and an increase of shear force. Drop size variation with thickener concentration could be explained by a dimensional analysis between the capillary number and the viscosity ratio. Compression properties, such as crush strength and Young's modulus were measured and compared. Among the microcellular foams prepared in this study, the foam incorporated with an organoclay having reactive group showed outstanding properties. It is speculated that the exfoliated silicate layers inside polystyrene matrix are the main reason why the foam has superior properties.



## Extrusion and foaming of polymers: an on-line method for rheology monitoring

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Polymer foams gained scientific interest in modern technology due to their excellent properties. The motivation for any rheometrical study is often the hope that observed behavior in industrial situation can be correlated with some easily measured rheometrical function. On the other hand the study of the system polymer/chemical blowing agent (CBA) requires conditions that are not achieved in standard rheometers. For preparing bi-phase system, Hydrocerol BIH (0.3, 0.9, 1.2 %wt) was mixed with pure LLDPE in an extruder. The presence of the screw in the extruder lets possible to obtain a homogeneous melt/CBA mixture and a proper melt temperature. Furthermore at the end of the extruder an homemade instrumented apparatus was mounted. This setup is realized adding from one to three identical cylinders, so varying the length of the capillary die, in which, using Dynisco sensors, pressure is measured and temperature is controlled by heat resistances. The temperature and pressure signals are sampled and recorded by a data acquisition system and an ad-hoc software code. The polymer melt flows down the die and the pressure gradient generated along it is calculated using Bagley correction, performing experiments at different flow rates varying the screw rotational velocity. The shear stress is calculated using Rabinowitsch correction, and thus viscosity is measured in a large window of operating conditions, varying pressure, temperature and flow rate. In order to validate this technique the pure LLDPE is used also in standard rotational and capillary viscometer obtaining viscosity measurements at low and high shear rates, respectively. The viscosity measurements with the on-line setup covers five decades of shear rate and the comparison with standard viscometers is satisfactory. The setup is used to systematically investigate the rheological behavior of the polymer/CBA system as function of concentration of CBA, revealing important indications on the evolution of the process.

S15-1346

## Flame retardant Expandable Polystyrene Beads

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Thermal degradation of polystyrene causes temperature-dependent emission of CO and aerosol free styrene and benzaldehyde are also released. The aerosol contains e. g. oligomeric polystyrene chains. Based on this, it is probable that the most important aspect of polystyrene smoke toxicity is its aerosol and CO content. Burning polystyrene causes a dense black smoke which impairs visibility and may hinder rescue work. The health effects of smoke particles from polystyrene are not known to the detail while in a comparative study they were assessed to be less harmful than smoke from wood, cork, leather or rubber (The toxicity of polystyrene smoke is most clearly associated with the concomitant CO exposure. One of the mechanisms seems to include changes in the hemoglobin oxygen-binding capacity. in this work we have been study on th effect of flame bromo copmpounds such ad HBDC( hexa-Bromo dodeca cyclohexane) on CO aerosol releases or any other toxic materials in EPS using in buildings and so on.



## **Polyurethane foams processing: improvement of nanoclays dispersion through microwaves applications**

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Polyurethane nanocomposite foams, both rigid and viscoelastic ones, were synthesised by in situ-polymerisation using both pristine and organically modified layered silicates. The effect of synthesis conditions, in particular the effect of different dispersing methods on morphology of polyurethane nanocomposite foams was studied. Clays were dispersed both in polyols and isocyanate, using a well known method (ultrasonication) as well as new dispersion method (based on microwave), in order to obtain very good clays dispersion. The raw materials so filled were used for foams preparation. The morphological characterisation of the foams, using X-Ray diffraction (WAXD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM), proved that the technology based on microwave processing is able to provide very good silicates dispersion and requires very short application time to be effective. Moreover, the results show that the better dispersion are achieved using polyol as dispersing medium, although also isocyanate may be successfully employed. Further confirmations of the importance of the clay organo-modifier are still present.