Symposium 13

Process Monitoring, Control & Sensors

S13-452

Terahertz Spectroscopy – A New Tool for Monitoring Compounding Processes

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In 1989, the first devices, capable of generating and detecting terahertz waves with frequencies between 100 GHz and 3 THz, were invented. The rich interest in this research field led to a rapid development of systems and components, which allows the introduction of terahertz technology to a variety of applications ranging from security scanners and biological imaging to quality control and production monitoring. Another promising field is the monitoring of the additive content and the degree of dispersion of polymeric compounds. Most plastics exhibit very low absorption at terahertz frequencies and their dielectric properties significantly differ from the ones of commonly used additives. First, we demonstrate inline measurements, where the terahertz system is located at a die after the extruder, which contains two quartz glass windows. The quartz glass is fairly transparent for frequencies smaller than 500 GHz. In between the windows, the polymer melt is located in a narrow slit. With pre-obtained dielectric parameters of the glass available, the exact index of refraction and absorption coefficient of the polymer melt can be determined. We characterize the effect of temperature and pressure on the measured transmission data for different polymers. Furthermore we verified several compounds with varied additives in order to obtain reliable information about the additive content of the polymer melt. Additional offline experiments on samples with differing degrees of dispersion are performed. A clear distinction between well and badly dispersed samples is possible, demonstrating the high potential for future inline characterization. We conclude that terahertz spectroscopy is a high-potential technique for non-destructive testing of polymeric compounds. Both, the degree of dispersion and the additive content can be characterized directly without the need for costly random sampling and offline testing procedures.

S13-912

Visualization Analysis of In-cavity Demolding Phenomena in Injection Molding of Fine Prism-Patterns Assisted by Dissolved CO2 Gas

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This research aims to investigate the influence or effect of the high-compressed CO2 gas filled in the mold cavity and the dissolved gas into the melt upon the in-cavity demolding process of injection molding. The experiments were conducted using 50 um-pitch prism patterns depending on the prismatic-glass inserted mold with a highly sealing structure and a high-speed video camera. In-cavity demolding process was directly visualized to capture the demolding patterns inside the whole cavity area, where the start timing and the subsequent penetration process of demolding phenomena were clearly detected.Comparing between the distribution patterns of transcription ratio in the molded sample and the demolding process observed, it was revealed that there were obviously some differences between the CO2-filled process and conventional molding process, in terms of the start timing as well as the end timing of in-cavity demolding process, supposed under the same transcription ratios. In the experiments, the existence of compressed CO2 gas proved to apparently accelerate the demolding process. Furthermore, the results obtained under the vacuum pumping conditions were also compared with the CO2-filled process and the conventional molding process, and discussed in this paper.

S13-1239

In-situ Characterization of Microstructure Development During Melt Spinning: Synchrotron x-ray Diffraction Studies From an Industrial Scale Fiber Spinning Process

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Spinning is a highly non-equilibrium process, in which filaments are extended as they cool from the melt. Fiber properties and structure depend crucially on the details of the spinning conditions. Molecular orientation and crystallization setup during spinning influence the structure developed in further processing downstream. Detailed characterization of this complex spinning process should lead to an understanding of the limits in properties practically achievable. The high brilliance of synchrotron x-ray sources makes it possible to study with relative ease the structure of a fiber during the spinning process. An industrial scale fiber spinning process was setup at the Advanced Photon Source, Argonne National Laboratory, which allowed x-ray characterization of orientation and crystallinity to very high spinning speeds. Nylon 6,6 was used in this study, and spinning speed ranged from 500 to 4400 mpm, with all other factors unchanged. Information was extracted from the x-ray data on the amount of the amorphous phase, the mesophase, and the crystalline phase, in addition to orientation. At the highest spinning speeds, a small amount of oriented crystalline material is formed from the highly oriented chains. These crystallites are rather small and crystallize with pseudohexagonal symmetry. The amount of oriented mesophase shows a plateau at intermediate speeds. The position of the peak associated with the oriented mesophase depended on speed, or residence time in the chimney. A fraction of unoriented amorphous material remained constant regardless of the speed. This immutable, unoriented, amorphous fraction imposes an undesirable upper limit on the strength of melt-spun fibers.

S13-216

Instrumentation of Injection Stretch Blow Moulding

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The injection stretch blow moulding process is commonly used to manufacture PET bottles for the carbonated soft drinks and still water industry. Bottle properties and performance are highly dependent on the balance between the stretching of the polymer and blowing times as well as the level of the air pressure inside the bottle. A portable wireless data acquisition system capable of accurately measuring the process conditions within an industrial environment has been developed and deployed within an industrial machine (SIDEL SB01). Sensors for force, pressure, displacement and temperature have been incorporated in a stretch rod whilst the kinematics of the blowing is also assessed by measuring the contact timing of the polymer with the mould by incorporating contact switches along the mould side wall. Statistical analysis reveals correlations between the stretching force, displacement of the rod, the pressure inside the bottle with the machine input parameters and provides valuable information for a better understanding of the process as well as for validation and development of numerical simulations.

S13-272

In-line monitoring of injection moulding materials and parameters using near infrared spectroscopy and chemometric analysis

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In recent years, near infrared spectroscopy has become a versatile analytical tool and has gained acceptance in a large range of production processes and industrial sectors, such as the petrochemical, pharmaceutical, environmental, biomedical, and automotive sectors. This study demonstrates the potential offered by near infrared spectroscopy for the injection moulding industry. A near infrared transmission optical fibre sensor has been developed and applied for in-line monitoring of colour changes, humidity levels during injection moulding polymer processing. In addition, near infrared spectroscopy was found to be a useful tool not only for in-line monitoring of the polymeric materials but also for in-line monitoring of the injection moulding parameters such as temperature and injection speed.Currently, the focus of the injection moulding industry is on improving and tightening the variation of injection moulding parameters and developing new monitoring and controlling systems and less on monitoring the material during its manufacturing. As previously emphasised in other studies, the monitoring and controlling of materials should be the priority when producing high quality products with specific functions. Therefore, this study shows that 1) the in-line spectroscopy system can successfully detect and monitor polymer changes (due to colour or moisture presence) and that 2) modifications of the injection moulding parameters can be translated into molecular and structural polymeric changes, identifiable by infrared spectroscopy.Different chemometric tests (conformity index test, cluster analysis test and PLS method) has been employed to assess the polymeric changes recorded through the near infrared data.

S13-549

Realtime Monitoring of Particles and Inhomogeneities in Flowing Polymer Melts at Extrusion and Injection Molding Processing

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In this paper an unique optical sensor system will be presented which is able to provide realtime information about particle and inhomogeneity situation in different flowing polymer melts by an inline or online process adaptation. By applying different kinds of signal evaluation in combination with a matched optical sensor setup a broad range of particles sizes and particle concentrations can be covered. This gives the opportunity to use that sensor system for a versatile number of applications in polymer processing. Thus phase morphology in flowing polymer blend melts can be evaluated. There are also first results on estimation of the dispersion degree of (nano-)filler in a flowing polymer composite melt as result of an extrusion compounding. From continuous particle sensor measurement a highly effective process optimization can be established to achieve high and stable product performance and to minimize waste from plastics processing. For unfilled amorphous polymers (PC, PMMA) only a minimum amount of disturbing particles is tolerable for certain high performance products (e.g. optical parts like glasses or lenses, displays, optical storage media). For this reason this optical sensor system can be applied for control of melt purity by an realtime detection of unwanted single particles in terms of process control and quality assurance. The presentation will give an overview about the applied measuring method. The optical sensor system and the possible sensor configurations to fit to different measuring tasks will be explained in detail as well as the possibilities and limitations of the method. Thereby the main focus is set on some of the most relevant results achieved at extrusion processing from laboratory to pilot plant to industrial scale. Furthermore first results at injection molding processes will show the potential of the method as well as further sensor developments which are necessary to meet the demands of this discontinuous polymer processing.

S13-783

Monitoring of the sample electrical conductivity during temperature cycling of polyethylene/CNT composites

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The electrical conductivity of a series of polyethylene / carbon nanotube (CNT) composites with different CNT concentrations (2.5, 5, 7 wt %) has been measured during multiple thermal cycling between room-temperature and 115°C.The CVD grown multi-walled nanotubes have an entangled structure, average diameter of 40-50nm and lengths of up to 100 µm, respectively. The nanocomposites were prepared by melt-mixing in a micro-twin screw extruder. High density polyethylene (HDPE) and low density polyethylene (LDPE) have been chosen as matrix. Very different behaviour has been found for the development of the conductivity with increasing temperature cycle number: while for the HDPE/CNT composites a continuous increase in conductivity has been observed with a saturation after about 6 cycles, for the LDPE/CNT composites a sudden decrease during the first cycle has been found, with only minor changes during further temperature cycles. In the stabilized temperature-conductivity characteristics after multiple cycles two different regimes can be distinguished. Up to about 70°C the conductivity increases slightly with increasing temperature due to the intrinsic temperature-dependent properties of the carbon nanotubes. For higher temperatures a positive temperature coefficient (PTC) regime has been observed, as often reported for filled polymers [1], related to the weakening of the nanotube network interconnections due to the volume increase of the composite. By changing the applied voltage during cycling, we could exclude that the continuous conductivity increase during cycling was due to field-induced alignment of nanotubes, as previously reported for linear low density polyethylene/CNT composites [2].[1] J-H.Lee, S.K. Kim and N. H. Kim, Scripta Materialia, vol. 55 (2006), 1119[2] M. Ferrara, H.C. Neitzert, M. Sarno, G. Gorrasi, D. Sannino, V. Vittoria and P. Ciambelli, Physica E, 37 (2007), vol. 66

S13-851

Quality surveillance for the water injection technique

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The water injection technique (WIT) is a special injection moulding process which is closely related to the well-established gas injection technique (GIT). It allows channels to be formed in an evolving moulding by injecting water into the still molten core. Due to the considerably more efficient cooling effect of water, cycle times can be reduced significantly. Since its development in 1998 the technology has gained significantly importance and has been used for several serial applications. One significant drawback of the technology is, that no specific to WIT adapted quality surveillance systems are available. Thus, different instruments for WIT process control have been developed, applied and evaluated. Apart conventional process control instruments, such as detection of cavity pressure, temperatures and volume flow rates, an ultrasound system was used for an online wall thickness measurement in the cavity. For an inline part analysis an IR-thermography system was additionally used. As test geometry a water cooling pipe with an outer diameter of 35 mm was produced using fibre reinforced Polyamide 6.6 as well as a virgin Polypropylene. As process variants the short-shot and the full-shot process were investigated using conventional WIT as well as the process combination of WIT and sandwich injection moulding. The investigations showed that characteristic values of the course of the cavity pressure in combination with the course of the volume flow rate offer a good correlation with typical part defects. For the online wall thickness measurement by means of ultrasound the process parameters water holding pressure, water delay time and mass temperature have to be taken into account for a sufficient correlation between time-of-flight and residual wall thickness. Furthermore the investigations showed that IR-thermography is not only capable for the detection of major part defects but also can be used for a control of the wall thickness.

S13-913

Application of Capacitive Transducer for melt front rate control in injection molding

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In injection molding, melt front rate (MFR) during filling phase is a key variable that determines the final product quality. A number of studies have shown that an acceleration or deceleration of MFR could result in hesitation flow which produces visible flow defects on the surface of a molded part. Variable velocity at melt front also induces variable orientation which in turn leads to non-uniform shrinkage and thus part warpage. A constant MFR during mold filling is recommended by most researchers. In practice, due to the lack of the approach to measure MFR, screw injection velocity is commonly chosen as the controlled variable during filling instead of MFR. However, experiments show that even with a constant screw velocity, MFR can be varied. A novel measurement device - capacitive transducer has been proposed and developed with a simple construction and low cost. The transducer can online and continuously measure the melt front position in mold cavity. With such device, it is possible to implement direct in-mold MFR control. This paper, based on the study of the characteristics of MFR, developed a proper controller through which MFR can be controlled constant well. Experimental results have shown the effectiveness of the sensor and the developed controller.

S13-937

Study on Transcription Improvement by Visualization of Melt-transcription Molding Process

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This paper deals with the experimental study on the improvement of the surface transcription in the melt-transcription molding process. First, the melt-transcription molding machine was used to replicate the mold stamp surface structure of 10 microns scale. The test material in the first experiment was cyclic olefin copolymer. The result showed that the transcription ratio was improved by increasing the stamp temperature, though the ratio was not improved very much by increasing the melt temperature. It was also considered that keeping the pressure of the molten polymer during the spreading process was important to obtain better transcription. To precisely discuss this point, a test transcription molding system for the visualization was made to simulate the process, and the flow condition and the pressure variation were measured. The test fluid in the second experiment was water solution of carboxymethyl cellulose. The result showed that the wedge shaped die-lip increased the fluid pressure during the spreading process.

S13-1042

In-situ Polymerization of Cyclic Poly(butylene terephthalate) : Ultrasonic and DSC Monitoring

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The in-situ polymerization of cyclic poly(butylene terephthalate) oligomers into PBT was investigated under various conditions of temperature, pressure and time using an original device that combines ultrasonic and volumetric measurements. The material was kindly provided by Cyclics Inc. as a two-component system where the catalyst was pre-blended within the CBT oligomers. In a typical ultrasonic monitoring test the CBT sample is heated at a rate of 2 oC/min to the polymerization temperature where it is held for a certain time under low or high pressure (30 to 400 bars). The sample is heated again to about 240 oC and cooled at the same rate to room temperature. Melting and crystallization of the resulting PBT polymer are identified from the simultaneous measurement of sample volume and the ultrasonic velocity and attenuation at a frequency of 2.8 MHz. Differential scanning calorimetry (DSC) tests at different heating rates were also conducted following the same procedure as the ultrasonic tests, albeit under no pressure. The results from both techniques will be discussed.

S13-1326

Investigation of Relationship Between Warpage of Injection Molded Productsand Temperature Distribution inside the Mold

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Warpage is a problem seen in the box-shaped injection molded products. The difference in temperature between the movable mold side and stationary mold side causes inconsistent shrinkage in molding, which is considered one of the reasons causing warpage. Measures to prevent this include changing the shape of the mold cooling channel or changing the mold material. In this study, we built a temperature distribution measurement mold, in which sheath thermo-couples are installed in each part of the mold, in the aim to measure in-mold temperature distribution when mold cooling channel patterns or mold materials are changed. We also investigated the correlation between the measurement results and the warpage amount, and obtained the following findings. (1)There exists an evident correlation between warpage and temperature difference between the movable mold side and stationary mold side. Consequently, increase in temperature difference increases warpage. (2)Changing temperature cooling channel patterns and mold materials to decrease this temperature difference decreases warpage by 20 to 30%.

S13-1332

Influence of extrusion conditions on the in-line measured electrical conductivity of polymer– multiwalled carbon nanotube composites

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In recent years, the applications of carbon nanotubes (CNT)-polymer composites has grown rapidly. Although, first CNT-polymer composites are commercially available a major restraint in market acceptance is the wide variations in electrical conductivity as function of the processing conditions. In order to study the influence of extrusion conditions on electrical properties experiments have been performed in a slit die flanged to the outlet of a twin screw extruder. The measurement slit die contains two electrodes in plate-plate geometry [1]. AC conductivity and the related complex permittivity were measured in the frequency range between 20 to 10E6 Hz for different extrusion conditions (melt temperature, screw speed, throughput etc.) and after stopping the extruder. The investigations have been performed during melt processing of polycarbonate (PC) and polyamide-6 (PA6) containing 0.5 – 2 wt% and 0.7 -2.7 wt% of multi walled carbon nanotubes (MWNT), respectively, for different processing temperatures. It was found for all samples that during extrusion the conductivities were in the order of magnitude of the matrix polymer. After the extruder was stopped (model experiment for injection molding) the conductivity shows a tremendous increase with time (conductivity recovery after shearing). This process was more pronounced for samples with CNT content close to the percolation threshold. The finding can be explained by the reorganization of the conducting network-like filler structure which was (at least partially) destroyed under shear. The reformation kinetics of filler clusters is due to a cooperative aggregation [2], which is related to the kinetics of cluster-cluster aggregation in a percolating system. [1]Alig I., Lellinger, D., Dudkin, S., Pötschke, P., Polymer 48 (2007) 1020 [2]Alig I., T. Skipa, Lellinger, M. Engel, S., Pötschke, P., Physica Status Solidi, 244 (2007) 4223.

S13-108

Nanofiller dispersion in rubber blends – a characterization method based on the online electrical conductance

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The present work introduces a new method based on the online measured electrical conductance for an online characterization of the dispersion of the nanofillers like carbon black, carbon nanotubes and nanoclay in the nanocomposites during the mixing process. The mechanisms of nanofiller dispersion in rubber matrix were studied by the analysis of the morphological development on macro and micro scale. The results from the morphological assessment were correlated to the time dependent electrical conductance measured directly from the equipment. It becomes obvious that the conductance-time curves of filler-rubber mixing show basicly the characteristic course in dependence on the structural feature of the fillers. The mechanical properties of the nanofiller-rubber composites were determined. A correlation between the online conductance, micro dispersion of the nanofiller and the mechanical properties was found. Further more, the online conductance can be applied to monitor the nanofiller immigration process in heterogeneous rubber blends and the development of the blend morphology. The investigation shows that nanofillers can be controlled included into rubber matrix materials using the method of online measured conductance during the mixing process. Online measurement of electrical conductance is a suitable tool for the identification of dispersion mechanisms and control of mixing process.

S13-254

FT-NIR as a inline qualitative determination method for polymer nanocomposites

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Nowadays, the X-ray diffraction and microscopic techniques are the most used tools for characterization of dispersion level in polymer nanocomposites. However, these techniques are pretty time consuming with a need of expensive scientific equipment. As shown in our previous publications, rheotens measurements are much faster and cheaper. But this method is still offline and reflects only a random sample. For industrial use there is a need for a fast, reliable and first of all inline method. Near-Infrared (NIR) spectroscopy is a non-destructive, optical method to determine information on the composition of samples. This method measures the absorbance of light due to excitation of molecular vibrations of the substance under investigation. By placing the sample in the light path, the substance present in the sample absorb NIR radiation at specific frequencies according to their molecular structure, resulting in NIR sample spectra.The position of the absorbance bands in the NIR spectrum provides the information for identification of substances and the existence of specific chemical functionalities present in the sample. NIR is also capable of providing information on mechanical properties as these properties are generally linked to the chemical state of the sample. The parameter of interest is the level of reinforcement in the nanostructured materials produced by filling a polymer matrix with nanoparticles in different processing ways using the co-rotating twin-screw extruder. The silicate platelets form different levels of 3D physical network in the polymer matrix, i.e. different physical crosslinking and bonding between polymer chains and organoclay are formed. This change in polymer and nanoparticle structure is detected by NIR and shown in NIR spectra. For predicting the mechanical properties and the reinforcement of the polymer nanocomposites the NIR measurements were correlated to results of X-ray diffraction-, tensile-, and rheotens tests.

S13-314

Real Rheology - and MFI On-Line

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The Melt Flow Indexer has been a useful tool to characterize an aspect of a polymer's molten flow behavior. Although simple to use, its limitations of length of time between samples, difficulty of automatic operation, inability to determine shear rate sensitivity, and narrow range of operability have frustrated its use for process control. The Helical Barrel Rheometer (HBR) circumvents these limitation and provides on-line data of viscosity versus shear rate, enabling direct determination of MFI. For agiven polymer type and process, the HBR viscosity versus shear rate raw data are collapsed onto a common master curve when converted to (viscosity)x(MFI) versus (shear rate)/(MFI). The HBR software calculates the extent of axes shift required for superimposition of the raw data for an unknown sample to match the master curve, which automatically determines the MFI.

S13-503

Potentiometric sensing of chloramphnicol based on molecularly imprinted polymer

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Despite the existence of a variety of analytical methods for controlled drugs detection, there is still a strong need in the development of portable sensor devices which would allow quick and effective analysis of a whole range of target compounds. Molecular imprinting technology is a synthetic approach to imitate natural molecular recognition. The imprinting process is performed by polymerising functional and cross-linking monomers in the presence of a template molecule. The subsequent removal of the imprint molecule reveals binding sites in the polymer network, which are complementary to the template in size and shape. That allows the highly specific rebinding of the template. Furthermore, usually MIPs are reusable, need low cost of preparation, exhibit a high mechanical and chemical stability and are applicable to a vast amount of operating conditions potentiometric sensor based on a molecularly imprinted polymer was fabricated for recognition and determination of drugs. The response characteristics of this sensor were evaluated by measuring the response potential to chloramphenicol which was used as a model material. The sensor showed high selectivity and a sensitive response to the template in aqueous system. The MIP-modified electrode exhibited Nernstian response in a wide concentration range, high performance, high sensitivity, and good long term stability. The method was satisfactory and used to determine the concentration of chloramphenicol in human serum and urine.References:1. Elena V. Piletska ,Maria Romero-Guerr ,Iva Chianella ,Kal Karim, Anthony P.F. Turner, Sergey A. Piletsky, Analytica Chimica Acta 542 (2005) 111–117.2. Raphael Levi, Scott McNiven, Sergey A. Piletsky, Soo-Hwan Cheong, Kazuyoshi Yano, and Isao Karube, Anal. Chem.1997, 69,2017-2021.3. G. D'Agostino , G. Alberti, R. Biesuz, M. Pesavento, Biosensors and Bioelectronics 22 (2006) 145–152.

S13-553

Potentiometric sensing of cetirizine dihydrochloride based on molecularly imprinted polymer

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A potentiometric sensor based on a molecularly imprinted polymer was fabricated for recognition and determination of cetirizine dihydrochloride. A molecular imprinted polymer(MIP) was prepared with cetirizine as the template and methacrylic acid (MAA) as the functional monomer. Acetonitrile was used as the porogen with ethylene glycol dimethacrylate (EGDMA) as the crosslinker and 2,2'-azobis(sobutyronitrile) (AIBN) as the initiator. Cetirizine hydrochloride, an antihistamine, is a major metabolite of hydroxyzine, and a racemic selective H1 receptor antagonist used in the treatment of allergies, hay fever, angioedema, and urticaria. The synthesized polymers were characterized by IR spectroscopy, X-ray diffraction and thermal analysis techniques. The proposed electrode shows good selectivity and wide linear dynamic range with a Nernstian slope. Control polymers (CPs) were prepared under identical conditions without the template. The applicability of the sensor was tested in potentiometric titration of cetirizine dihydrochloride in tablet pharmaceutical form and human plasma.Reference:[1] Susan Sadeghi, Fatemeh Fathi and Javad Abbasifar. Sensors and Actuators B. 122(2007) 158-164[2] G.M. Murray, A.L. Jenkins, A. Bzhelyonsky, O.M. Uy, JohnHopkins APL Technical Digest 18 (1997) 464–472.[3] Y. Zhou, B. Yu, E. Shiu, Anal. Chem. 76 (2004) 2689–2693.

S13-592

In-line monitoring of PP/MMT nanocomposites formation during melt compounding

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The aim of this work is to monitor in-line, using an optical detector fitted at the extruder die exit, the formation of PP/MMT nanocomposites during polymer melt compounding. The intensity of the detector´s signal is dependent of the clay type (organofilization treatment), concentration and particle size, and so it is possible to monitor the presence of the clay tactoids due to their light extinction. The MMT clay is added as a pulse with constant weight into the PP extrusion flow and its presence is followed by the optical detector at the die exit. The data comes out as the common residence time distribution curves having its maximum intensity dependent upon the clay average particle size. On clay exfoliation the size of the tactóides are reduced to levels below the minimum particle size for producing light extinction and so the signal intensity reduces as the nanosize composite is formed. The detector´s intensity reduces following the sequence: “not treated clay” > “organofilized clay” > “organofilized clay + compatibilizer”, which represents the level of particle size reduction. The paper will discuss the effect of clays with different organofilization treatment (Closite 15A, 20A, Na+) and compatibilizer (PP-g-MA) in the in-line PP/MMT nanostructure characterization.

S13-693

Consideration of anti electrostatic agents in the polymerization mediums with low dielectric constant solvents

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In the polymerization of some polymers such polyolefins , low dielectric constant solvent such as hexane, heptane and… must be used. As a result of solvent movement and its friction with system components electrostatic charges generates. Because of low electrical conduction of these solvents the generated charges is accumulated in the polymerization medium. Consequently repulsive force between same charges effects the growth of polymer particles and causes to form Fine .Fines have no commercial cost and lead to process problems. In this article role of anti electrostatic agents in increasing of electrical conduction of medium and its effect on particle size distribution (PSD) and final properties of polymer (polyethylene) is discussed.

S13-820

Identification of raw material condition by process monitoring and derived control strategies in injection moulding

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State of the art electromechanically driven injection moulding machines are equipped with several sensors to ensure an adequate control performance. But still a lot of information available in the injection moulding machine in running production is not utilized. Substantial information on the processing behaviour of the raw material can be gained from the resulting torque or the resulting forces respectively. Assuming that the injection moulding machine has a suitable reproducibility in its movements and the ambient conditions (including the processes in the mould) can be kept at constant level, the only source of disruption is the raw material. Faults coming with the feed pellets can be of different types. Common faults are a varying humidity contend of the material, the discontinuous admixture of regrind and also batch variation effects. The impact on the process can be indentified monitoring the process data. In this work special index numbers and integrated measurands that are mainly based on energy balancing are shown. These parameters are determined for each moulded cycle and are shown with its characteristic reaction on the specific disturbance. Furthermore the gained information on the processing behaviour can be used to propose a change in the relevant machine settings in order to keep the part quality at constant level. The selected representative quality criterion here is the parts mass. In future an intelligent injection moulding machine could be able to compensate the disturbing effects coming with the raw material automatically.

S13-917

Visualization Analysis of Transcription Molding Process in Microscale Line and Space Patterns

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The direct observation of how molten resin fills into a cavity is considered as the most efficient method of clarifying the transcription process. The aim of this study was to visualize the transcription process of microscale line and space patterns to clarify the transcription process and its relation with various influencing factors. Using rectangular grooves fabricated by the LIGA (Lithography Galvanic Forming) process, the resin filling behavior was visualized using the combination of a long-distance microscope and high-speed video camera. From the visualized images, the area where the resin contacted the groove bottom was taken to be the boundary between the dark and bright regions, and a model of the resin behavior in the grooves was proposed. The resin contacted the groove bottom shifted from the center of the groove to the downstream side. As a result, the resin flows to the micro rectangular groove asymmetrically, and the resin touching the bottom moves toward the opposite side to the gate. Changes in the filling ratio of the molten resin into the rectangular grooves were also quantified with time, under different injection rates, holding pressures and groove widths. The transcription width at each injection rate was found to increase markedly up to several ms, confirming that the higher the injection rate, the faster the resin touches the groove bottom and the better is the transcription ability. On the other hand, the influence of the holding pressure appeared for a period of several hundred ms.

S13-1004

Measurement of Demolding Resistance in High Transcription Molding of Line and Space Micro Grooves

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The higher the transcription ratio in injection molding of fine patterns, the more difficult the demolding becomes without any troubles. Therefore, how to separate the molded sample and the mold cavity surface can be regarded as a very important subject in the field of high transcription molding. In this study, we developed a new measuring mold which can realize the precise measurement of the demolding resistance with high repeatability. On the basis of the tool, we successfully conducted the measuring experiments under different molding conditions and analyzed the correlation between the transcription molding and demolding resistance. The fine patterns used for the experiments were rectangular line and space grooves with 50 micrometers in height and width. Through the experiments, it was confirmed that the demolding resistance increased according to the increase of transcription ratio, whereas the existence of the compressed dissolved CO2 gas in the molten resin drastically decreased the demolding resistance even though the transcription ratio was remarkably improved under the same holding pressures as those in the conventional injection molding.

S13-1125

Near Infrared Spectroscopy for In-Line Control of Biopolymer Processing

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Near infrared (NIR) spectroscopy has become an analytical tool for material and process control leading to substantial quality improvements of the output material. In the field of polymer processing NIR spectroscopy has been increasingly applied for an on-/in-line monitoring mainly at lab scale process development but also for production of high value materials. In this paper the bio-polymer polylactide was investigated by NIRS in the range of 1.2 to 2.4 µm in order to identify modifications induced by additives of nanofil® and nano titanium oxide. Although these additives do not strongly absorb NIR radiation, there influence can be analysed by use of chemometric codes. A twin screw extruder (Haake Polylab PTW 16) was used to extrude polymer pellets mixed with the additives. The extruder was fed by a single screw metering feeder. The optical sensor probes (glass fibers) were placed between screw and nozzle to achieve a good optical throughput and to withstand typical temperature and pressure conditions during the extrusion (up to 220°C and a few 10 MPa). The in-line NIR spectroscopy enabled a real time information which might be used for process control or feeding control on the compound characteristics continuously. The chemometric data evaluation was able to quantify the measured values, especially the content of the additives in the compounds. The established statistical models predicted the actual values with high correlation coefficients (>0.99). The additives altered viscosity of the melt and mechanical properties (Young´s modulus) of injection moulded test samples of the compound. The evaluated NIR spectroscopic data could be directly correlated to the measured mechanical properties.

S13-1191

On-line measurement of light transmission using photodiodes during crystallization in calorimetric experiments

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Spherulite growth kinetic is often followed microscopically. Starting from these direct observations, the idea of associating light intensity measurements to crystallizion of polymers is always attracted many reaserchers. In this work the phenomena associated with the growth of spherulites on cooling molten polypropylene and their decay on raising the temperature, together with the associated changes in the light transmission of the sample is studied. The apparatus used consisted of a stabilized light source to illuminate the sample in a special calorimetric cell using a microscope, with crossed polarizers. A photodiode was placed over the ocular tube of the microscope to record the depolarized light from the crystallizing specimen. The polymer samples (ca. 100 to 200 micron), between microscope slide cover slips, were melted and rapidly cooled to follow isothermal crystallization. The method, used also by Magill in the sixties, embraced a wider crystallization range than microscopically observations of spherulite growth kinetic. The simultaneous calorimetric and optical measurements allowed to relate directly the observed structure to an overall crystallinity degree.

S13-1325

Investigation on the tensile deformation behavior of the semi-crystalline polymer

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The tensile deformation behavior of three typical semi-crystalline polymers, including the low density polyethylene (LDPE), the high density polyethylene (HDPE) and the polyamide 6 (PA6), was investigated in the present work. It has been found that the different semi-crystalline polymers exhibit markedly different tensile deformation behaviors, including the shape of stress-strain curves and the so-called heterogeneous-homogeneous transition. For LDPE, it deforms homogeneously and shows no obvious necking process during the overall tensile process. While for HDPE and PA6, clear necking is observed when samples are uniaxially tensile deformed. When considering the effect of strain temperature on necking process, significant difference of plastic deformation between HDPE and PA6 sample emerges. The heterogeneous necking disappears and the homogeneous deformation occurs with strain temperature increasing. For HDPE, this transition takes place in the vicinity of the melting temperature, while for PA6, it takes place close to the glass transition temperature instead. Combining the Brereton analysis and Considère construction, the local stress maximum of nominal stress-strain curve can be measured quantitatively. However, this method is limited in the necked obviously and instantaneously yielding materials. In addition, with respect of the double yielding of materials, it seems that this special phenomenon can be demonstrated simply by plotting the compression residual strain-applied strain curves of samples.

S13-1327

Improvement on Injection Molded Products Appearance by Induction Heating Mold

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In injection molding, surface defects such as weld-line and silver streaks are serious problems which trouble engineers. It is also known that these surface defects can be prevented by heating the mold cavity surface to above the resin melting point Tm or glass transition point Tg. In this study, we designed and built a rapid heating and cooling mold applying induction heating, which is capable of continuously heating and cooling the cavity surface rapidly. This mold has a cavity insert with coils and cooling channels arranged inside the mold base. First the heating and cooling characteristics of the cavity insert were studied. Next, using the mold, heating and cooling molding of high impact polystyrene and normal molding without performing heating and cooling were performed and effects on the external view and surface properties of rapidly heated and cooled molded products were investigated. The following findings were obtained. Observation of the surface of the product molded using this mold showed that in normal molding, the formation of V-shaped weld-lines is seen on the molded product surface. On the other hand, when heating and cooling is performed in molding, no weld-lines are found, and compared to normal molding, a highly glossy molded product surface has been confirmed, verifying that this mold helps improve the external surface of molded products. However, if the holding pressure is low, sink marks are found to generate on the molded product surface, but when too high, flashes are found in the vicinity of the molded product, suggesting that there is a need to set the holding pressure carefully.

S13-1355

Evaluation of the rheological data's by flow of a molten polymer through a double - stream die

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The determination of rheological data's of molten polymers usually requires the measurements realized by use of various types of rheometers, like capillary, shearing, elongational or/and rotating devices. A comprehensive description of the characteristic rheological values includes between others the Weissenberg – Rabinowitsch correction, with the n and k – values from the power low equation. In our investigation the rheological data's were determined during extrusion flow of molten polymers through a special die, characterized by a double (multi) flow with various cross sections. Based on the die swell values and the actual cross sections, precisely measured and recorded in a real time by an optical observation system, the n and k – value may be estimated. Two procedures were applied in our case, e.g. constant linear flow velocity or equal linear pulling rate of both (multi) streams of extruded molten polymer. Particularly, the measurements of the streaming polymer cross section were performed by means of a visualization technique, coupled with the special computer program (BARUS 2002), giving the possibility to observe and to record the diameter in real extrusion conditions. The polymers used in this study were commercial polyolefin (LDPE, PP and the mixture of both). The theoretical prediction of the extruded cross section and the results of the measurements, together with revealed corresponding rheological data's will be presented and discussed.

S13-1366

THE DUAL-MODE SENSOR OF THE VISCOSITY FOR COAGULATION LIQUIDS.

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Many technological liquids (for example, some type of the oil) at statistic conditions have clear the tendency for formation the structure. This phenomenon have been determined, in particularly, the time for safety stoppage of the oil pipelines. In this case, the energy of the ties particles there are at initial milestone of the process, that mechanical influences there are sufficiently for breaking its. The sourses of such influence there are the technological devices as the mixer and the pump, sometimes there are the vibratory dispersants. Arising the problems for transport of such liquids take place attention to the processes of reversible the mechanical breaking and reconstruction of the structure of thixotropic colloid solutions and gels. In this case, there are the oil dispersion systems. At this time, the technical means of measuring for the kinetic coagulation work out scanty For the solution this problem were worked out the dual-mode vibration sensor, it is possible to use at regime of the breaking structure and dynamical not the breaking dimension with continuous the registry of the running values of the rheological characters. At the result of the consistent switching regimes there are possibility to registering of the dynamic to restoration the coagulation structure. There are the possibility to determine from such kinetic dependence: 1.The viscosity of the breaking structure. 2.The viscosity of the forming structure. 3.The thixotropic period – the time for reversible of the coagulation.

S13-1395

Online characterization of the intercalation/exfoliation process of nanoclay in polymer materials

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A new online method based on the electrical conductance measured directly in the mixing chamber of the internal mixer has been developed to characterize the nanoclay dispersion in a polymer matrix, e.g. PA or NBR. It has been observed that the online conductance increases strongly with progressed clay dispersion up to a plateau, which corresponds to the maximum dispersion (intercalation/exfoliation) of nanoclay. The results received from different experimental techniques, like optical microscopy, atomic force microscopy, SAXS analysis, bound rubber measurements and tensile testing confirm this correlation. The time when the plateau is reached, is strongly influenced by the mixing conditions and material composition. Furthermore, also the technological conditions influence the exfoliation process significantly. Thus, higher shear intensity will shorten the mixing time up to reaching the plateau value of conductance. The effect of different clay types and clay concentrations will be discussed in detail.