Executive Summary:

Self Reinforced Polymer composites (SRP) are thermoplastic composites which have a matrix and reinforcement of the same polymer family. The advantages are lighter, stiffer, more impact resistant and recyclable composites. The current state of the art at the beginning of the project was PP-based flat sheets which, although having some excellent properties, are limited in their application because of the moulding limitations of flat sheets. The ESPRIT project (FP7 Contract number 214355) was created to develop SRPs beyond the existing flat-sheet formats and make flowing materials for complex 3D shapes along with novel selective heating by means of electromagnetic energy.

The key challenge for the project was to process an SRP composite by heat and pressure, allowing the matrix to soften and form without damaging the reinforcement which naturally has a very similar melt profile. A wide selection of polymers was chosen for development in the project including PP, PET, PBT, PEEK, POM, PPS, PA and more. The processing options were injection and compression moulding the former being a particular challenge due to uncontrolled shear-generated heat. The alternative heating method of selective heating was developed, to heat just the matrix and not the reinforcement by means of electromagnetic heating.

The partners successfully made the intermediate materials required. Pellets were made from commingled fibres which were then pultruded into rods and then chopped. They were also made by melt impregnation of matrix into reinforcement and again chopped into pellets. Sheet materials were made by powder impregnation of the matrix into various textiles or fibre reinforcements and also by extruding the SRP pellets into sheets. To facilitate electromagnetic heating additives such as iron powder or carbon nano tubes (CNT) were successfully compounded to an excellent distribution quality.

These intermediate materials were heated and formed. Induction energy was used to heat sheets and subsequently form them. Microwave energy was also used to heat susceptor-laden sheets to a point where they could be moulded. Pellets were injection moulded into various complex shapes proving that, with carefully controlled parameters, flowing SRP is a reality.

The ESPRIT project achieved the set objectives of an SRP with 30% weight reduction (HTPET/LPET) which can be flow-moulded. Controlled selective heating was also achieved, by induction and microwave methods, and the efficiency of conductive heating was improved by a dual-channel heating system with a unique energy battery. As with many new materials it is difficult to bring the costs down to a competitive level but a very promising example of an SRP is the HTPET/LPET combination which can sell at 7 EUR/kg compared to an equivalent PBT with glass fibre at 4 EUR/kg but when the weight reduction is taken into account (replacing glass with PET) the difference becomes quite narrow and, of course, the srPET has good recycling credentials, a growing requirement for many end users.

An array of case study parts was made to demonstrate the new technologies, ranging from automotive parts, to fans, sportswear and hand- tool parts. Some parts are relatively simple shapes but with, for example, exceptional impact properties, others have thin walls and ribs and demonstrate stiff, lightweight parts. The injection moulded parts were achieved with standard machines albeit with close attention paid to the process parameters.

The post-project development and collaboration has continued into further research proposals thrown up by the ESPRIT work and into commercial applications such as luggage, sports goods, medical apparel and consumer goods already being pursued by various partners in collaboration and individually.

Project Context and Objectives:

Background

The European plastics industry, worth an overall EUR 200 billion per annum, saw a decline in the number of plastic injection moulders of 9% from 2000 to 2006. Slowing economic growth, OEM price pressure, rising raw material costs and the relocation of manufacturing to Asia, have all combined to put pressure on injection moulders, demonstrating the need for new knowledge-based materials and process techniques to maintain the European leadership in this sector. The materials developed within this project aim to offer that competitive advantage to European moulders whilst also bringing environmental impact advantages.

The ESPRIT project was born out of previous research into Self Reinforced Polymer composites (SRPs) in projects such as Novem (a Dutch cooperative research project), Recycle and Futureplas (UK DTI funded projects) and Hyject (a German-led EC funded research project). These last two projects had elements of investigation into flowing self-reinforced plastics, and the ESPRIT project was created to further this specific knowledge. SRPs are defined as thermoplastic composites having a reinforcement and matrix (or resin) of the same polymer family, the main advantages being, compared to current non-SRP materials, excellent impact resistance, improved stiffness for a given weight and easier recyclability due to the absence of thermoset resins, glass or carbon fibres. Self-reinforced PP is 40% lighter than PP and 25% lighter than glass fibre-reinforced PP (GMT) for equal stiffness, reducing raw material, through-life energy use and associated emissions.

SRP composites have been present in the market since the 1990's but only in the form of textiles or sheets which creates severe limitations on applications possible. NetComposites initiated the ESPRIT project, gathered the European experts and created a vehicle for the development of new materials and processes to make the next generation of flowing SRP materials. By developing these flowing SRP materials and the processes to use them ESPRIT has opened more opportunities for designers and manufacturers and more opportunities to reduce the volume of plastics used.

Objectives

The aim of the ESPRIT project was to develop the next generation of lightweight, self-reinforced polymer composites together with the energy-efficient manufacturing processes needed to produce components from this family of materials and as a consequence reduce the amount of plastic used to make components. The targets were set at a 30% reduction in materials used and therefore a commensurate 30% reduction in weight.

In self-reinforced polymer composites, a polymer matrix is reinforced with high-tenacity fibres or tapes of the same polymer family, such as polypropylene-reinforced polypropylene, creating a material with typically 3-5 times the strength and stiffness of the unreinforced polymer, giving the ability to use less material for the same mechanical properties in a component. Additionally, unlike glass or carbon reinforced plastics, self-reinforced plastics are not contaminated with high levels of mineral fibres, so they have the same level of recyclability and density as the base polymer.

However, current forms of self-reinforced plastics, especially in commodity and low-cost polymers, are only available in sheet or fabric form, made from extruded or co-extruded tapes. The extruded high-tenacity tapes are 'hot-compacted' into sheets which can be subsequently re-heated and formed under highly controlled temperatures and pressures. The effect is to soften the outer layer of the tape which forms a binding matrix and therefore a composite is created. The co-extruded tapes have both the reinforcement and matrix elements present and can therefore be woven into textiles and be formed by heat and pressure in a less critical process to form thermoplastic composites.

The textile or pre-consolidated forms severely restrict the range and types of components that can be manufactured from these materials, as the parts made from these materials must have constant wall thickness and significant amounts (typically 30%) of excess waste material must be trimmed away after forming. This also limits the applications of these materials as it is not easy to integrate parts into a single component, compared to conventional injection moulding or compression moulding processes.

The concept for the ESPRIT project was therefore to develop flowing versions of self-reinforced plastics from commodity polymers – polyolefins, polyamides and polyesters – and key to this was the development of techniques that will allow the selective melting of the polymer matrix without causing adverse effects on the polymer reinforcement fibre. This necessitated the development of energy-efficient microwave and induction-heating techniques to allow selective melting of the matrix polymer. High performance, commodity polymers were used to develop self-reinforced plastics with significantly enhanced properties over existing plastic and, for the first time, the ability to be compression and injection moulded into complex shapes.

In broad terms the aims were to develop flowing self-reinforced polymer composites with a strength or stiffness at least 3 times higher than the parent polymer, develop cost-effective heating and moulding processes for flowing self-reinforced plastics and to carry out technical and commercial evaluation of the developed materials and process by manufacturing and testing 3 case study parts.

To achieve these primary objectives, the problem was broken down into a series of technical objectives, the key ones being listed below:

- Identification and development of heating promoters, such as silicon carbide, aluminium, carbon black to allow the matrix to be selectively heated by microwave or infrared energy
- Modification of the matrix polymer to reduce the melting temperature of the polymer, below the temperature at which the reinforcement phase becomes thermally unstable
- Increasing the temperature stability of the polymer phase used to create reinforcement fibres and tapes, to maximise the matrix and reinforcement melt temperature difference.
- Drawing of high-tenacity fibres from modified polyolefin, polyamide and polyester for the reinforcement of modified polyolefin, polyamide and polyester matrices
- Impregnation of the high-tenacity fibres with modified matrices, using powder, melt, commingling and bi-component filament techniques.
- Development of microwave, infrared, conventional and induction heating methods to enable selective preheating of the developed matrix polymers and their compounds
- Determination of suitable process conditions for injection and compression moulding of LFT pellets, using conduction preheating and compounding in an extruder.
- Demonstration of the developed materials, heating and forming technologies on a range of 'real life' case study parts

The overarching scientific methodology was the development techniques to allow the polymer matrix to melt and flow without causing shrinkage, melting or loss of properties in the polymer reinforcement, given that the matrix and reinforcement are two forms of the same base polymer. Two approaches were taken to this challenge:

- Differential Melt Temperatures: The difference in melt temperatures between the matrix and the reinforcement polymers would be maximised to give a practical 'process window'.

- Selective Heating: The matrix polymer would be modified with a susceptor or filled to allow it to be selectively heated, using microwave, infrared or induction heating to melt the matrix phase without affecting the reinforcement.

These materials would then be trialled by using compression and injection moulding technologies, first of all on a small scale to characterise the various combinations, additives and techniques and then on a larger scale to produce case studies to demonstrate the advancements.

There were some key challenges faced by the project partners:

- To engineer a larger 'process window' (the difference in melt temperatures between the reinforcement and matrix)
- To use standard, or close to standard, injection moulding equipment. The uncontrolled shear-generated heat in the barrel is very damaging to SRP materials.
- To achieve adequate distribution of additives in difficult polymers such as polypropylene, sufficient to achieve even heating by electromagnetic sources.
- To combine reinforcement and matrix in an efficient, controlled manner
- To produce intermediate materials (pellets or sheets) in a usable, economic way
- To control the heating of polymers when using electromagnetic methods
- To speed up cycle times and improve efficiency of conductive heating methods.
- To manufacture representative case study parts to showcase the particular advantageous properties of the new materials and processes
- To establish and sustain a collaborative approach over the three and a half year project

Partners

The ESPRIT partners were selected to give a mix of expertise in specialist research areas, for knowledge of materials or processes and for industrial activity in related areas. The partners are listed below:

- NetComposites Ltd: Coordinator and originator of the project, NetComposites, from the UK, has been developing and moulding self-reinforced plastic components for many years and is also highly experienced in the management of collaborative European projects. Responsible for developing flow compounds, compression moulding trials.

- Comfil ApS: Comfil ApS, a Danish Company, was founded in 2001 and their core business is development and production of thermoplastic reinforcement yarns. The yarns are manufactured in a commingling process on in-house developed machinery and in ESPRIT Comfil designed and produce reinforced commingled yarns and pultrusion processing for pellet manufacture.

- Fibroline France SarL: Fibroline has developed a dry impregnation process technology which replaces customarily used wet processes in the manufacture of composite and technical textiles. The role of Fibroline in this project was to develop and produce pre-pregs based on thermoplastics using powder impregnation and flat calendaring.

- Institut fuer Verbundwerkstoffe (IVW) GmbH: IVW is a non-profit research institute in Germany, exploring and advancing applications of composite materials. IVW has great experience in research projects and was involved with fundamental investigations on compression moulding, inductive heating and production or prototype parts.

- Regloplas AG: Regloplas is a privately owned company based in St.Gallen Switzerland and for over 45 years in designing and building temperature control units. Regloplas was involved in the design of temperature control and monitoring systems, and in the design and build of temperature control units specific to self-reinforced plastics.

- Fricke und Mallah Microwave Technology GmbH (F and M): F and M was founded in 1995 and is now one of the leading German suppliers of microwave ovens for industry and research. Their role in the project was the development of microwave preheating systems for the matrix materials.

- AIMPLAS – Instituto Tecnológico del Plástico: AIMPLAS, located in Valencia, Spain, is a private, non-profit Association which has offered its services to the Plastic industry sector since 1990 its main work relating to technological research and development of plastic materials, products and processes. In this project their main activities were the identification and testing of additives for the microwave and induction heating effects and the improvement of the susceptor dispersion through compounding processes. Other activities have been the simulation/technical assistance of the scale-up process and specific characterization of final specimens.

- Promolding BV: Promolding is an innovative company that transforms high-performance polymer technology into industrialized applications offering product design, material and process development and manufacturing. In ESPRIT their role was 3D-CAD & CAE design, lab-scale compounding and testing, tool design & manufacture and injection moulding trials.

- Ticona: Ticona came into the project by way of purchasing Future Advanced Composites & Technology GmbH, one of the original partners. Ticona continued the work of developing long fibre reinforced self-reinforced polymers with an in-house melt impregnation technology

- AVK eV + European Alliance for Thermoplastic Composites: AVK is the legal entity for the EATC which was formed in 2000 by leading European companies, with a concerted commitment to the technological advancement of long fibre reinforced thermoplastics over the complete product chain. EATC acted to steer the direction of the project to meet the needs of their members, and the thermoplastic composites industry as a whole.

- Polisilk SA: Polisilk is a leading extruder of polypropylene multifilament yarns for industrial applications and are also active in the extrusion of speciality polymers. The company has 200 years of experience in the textile world and 30 years' experience in the production of polypropylene yarns. In ESPRIT their task was to develop and supply monofilament and bi-component yarns for use in subsequent impregnation processes.

- PEMU Muanyagipari Zártköruen Muködo Részvénytársaság: PEMÜ Plastic Processing Co. has become one of the most important companies of the Hungarian plastic industry since its foundation 40 years ago. The company has always been very innovative regarding the processing of new raw materials and the introduction of new plastic processing technologies. Their role in this project was compounding of matrix polymers with fillers to allow microwave and induction heating, as well as extrusion and moulding of parts from the compounded materials.

Project Results:

Description of main S & T results/foregrounds

The work carried out was divided into logical Work Packages in order to plan and manage the activities and therefore the results of the project are presented in the same sub-divisions.

WP 1 Definition of Materials, Processes and Applications

- Task 1.1 - Definition of Materials, Processes and Applications
- Task 1.2 - Details of Heating Techniques and Processing Routes to be used in the Project

These two subjects are in many ways co-dependent and so were reported in tandem through the project. These tasks relate to the early laying of foundations in order to assess all the possible options and to start the project in a sensible, considered direction. The knowledge generated through these tasks was a comprehensive flow chart of the processing routes, leading to an understanding of requirements leading in turn to a list of all possible polymers which was then narrowed down to some key candidates based on a thorough analysis of properties. The matrix and fibre materials were naturally reviewed together as they can feature in the same SRP and need to be considered as a unit.

They were also been divided into 3 commonly used categories in order to target the properties required:

- High Temperature/Specialist
- Technical/Construction
- Commodity/Common polymers

The list of considered polymers analysed was; PEEK, PVDF, PPS, PEI, PC, POM, PBT, PET, PA, PE, and PP. Each one was analysed and an individual table created, with price, availability, melt temperature, possible treatments, service temperatures and potential end use. After considering the alternatives the ESPRIT consortium agreed the following would be chosen as the materials to be pursued:

- Commodity polymers: Polypropylene (PP), Tm 140°C - 175°C, Service temperature ~ 80°C, Very low cost
- Technical Polymers: Nylons (PA, including semi aromatic PAs) Large range of Tm (180°C - 283°C), Good service temperature (90°C - 160°C), Large range of grades giving a range of properties and prices
- Polyesters (PET) High Tm (255°C), High service temperature if matrix is crystalline, Low cost
- Speciality Polymers: None were selected at this point as the raw material properties were deemed are so far unsuitable for SRP aspirations.

Additives

Additives were considered at this point as well, the amount, type and quantity of additive used affects not only the accuracy and success of the heating method but also, potentially, the properties of the SRP itself. Great care must therefore be taken in selecting and testing the additives. Additives selected:

- For microwave: Silicon carbide, Aluminium, Carbon black, Polyaniline doped with aqueous acid, Carbon nanotubes, Barium Ferrite, ZnO whiskers
- For induction: Steel fibres, Magnetite, Iron particles, Iron pigments, Manganese-zinc oxide, Copper particles

- For Infrared: Polymethine, Phthalocyanine

Heating Systems

Having selected the appropriate polymers the heating systems were considered. These were broadly laid out in the DoW but more detailed consideration was given now to the Microwave, Induction, Infrared and Thermal Transfer methods.

Processing

Out of the possible processing routes various stages were considered and partners agreed basic requirements for the pre-processing stages of pultrusion, commingling, powder impregnation and speciality chopping. In addition the processing stages were considered and the concepts of investigation set out: Injection moulding and Compression moulding

- Task 1.3 Broad Outline Specifications for Case Study Components

From Task 1.1 and 1.2 decisions were made about materials and process which in turn allowed the planning of possible demonstrators. A table of desired properties was created based on the expected scope of case studies possible. Three typical mouldings were selected as being a complex injection moulding, a large high aspect compression moulding and large high aspect injection moulding. As expected, later in the project the aims moved slightly as the materials and processes were developed. Not every category was explicitly covered but many more demonstrators from varied applications were actually made

WP 2 Modification and Heating of Matrix Materials

- Task 2.1: Chemical Modification to Reduce Matrix Melt Temperature

Reducing the melt temperature of the matrix phase of an SRP is desirable in order to increase the 'processing window' of the composite, which is the difference between the melt temperature of the matrix and the point at which the reinforcement fibres begin to degrade (lose their mechanical properties). In this task AIMPLAS carried out modifications on the 3 main selected polymer groups from WP1which can be summarised as below:

  - Polyesters: Co-monomer polymerisation was carried out using CHMD (cyclohexanedimethanol) or MPDiol (methyl propanediol). Although the chemically modified polyesters showed a reduction in melting temperature they also showed a very significant loss in mechanical properties. It was decided to select the best commercially available polyester-related a DUPONT PBT. This presents good mechanical properties and low melting temperature compared with the typical PET fibres used in the industry.

  - Polyolefins: The physical modification of polypropylenes with beta nucleant additives was a promising, affordable whilst maintaining good mechanical properties. There was a strong dependence between beta crystallization and cooling process which was important because for the project objectives the most interesting thing is to have beta crystals just after the compounding process, and before the processing with the reinforcing fibres. For this reason the cooling rate of the materials, after the compounding of polymer matrix with the additives, takes critical importance. Experiments were carried out on the re-crystallisation process and when the cooling rates are too slow, the melting temperature of alpha peak increases. This is negative in relation to the project objectives. It seems that post-beta nucleation is not suitable and only controlling the compounding process (or injection process) could provide useful beta phase in the crystalline structure of the polypropylene. After the extensive investigations it was decided that the best options for a polyolefin matrix to take forward into the project would be an INEOS HDPE with high mechanical properties and low melting temperatures and a LYONDELL metallocene polypropylene that maintains the typical mechanical properties of Ziegler-Natta isotactic polypropylene and reduces melting temperature.

- Polyamide: Tests using copolyamides and blends of PA6/PA66 showed excellent low melting temperatures but again there was a very significant loss of modulus. Organoclays (montmorillonite) were also investigated. These modify the crystalline phase of the polyamide reducing the melting temperature. However, the reduction was in the order of only 5ºC which is not enough to achieve the goals required for the project although the mechanical properties would be increased using nanoclays. To go forward into the project a DSM PA6 with good mechanical properties and a conventional melting point was chosen.

- Task 2.2: Compounding of Matrix Polymer Systems with Heating Promoters

In order for the later processing tasks to have raw materials to work on it was necessary for AIMPLAS to investigate the compounding of various susceptors into the matrix polymers. A wide range of additives (13) were selected and trialled in HDPE, m-PP, PBT and PA6 with additives such as Carbon Nano Tubes (CNT), PZT, Carbon black, FE, SiC and TiO2 at levels from 0.5 to 20%.

The Carbon Nano Tubes (CNT) showed excellent susceptor qualities but poor dispersion, the challenge was to improve the dispersion to give better heating effects. The following work showed a progression from dispersion with many agglomerates, to an excellent homogeneous result. The compounding parameters were closely monitored and recorded and were then passed later in the project to partners for up-scaling in larger quantities.

- Task 2.3: Development and Evaluation of Microwave, Induction, Infrared and Conduction Heating

Microwave heating

Basic tests were carried out to ascertain the effectiveness of the additives and the compounding process. Multimode (AIMPLAS) and monomode (NetComposites) microwave (MW) systems were used and it was clear that the monomode was much more efficient but more difficult to control, so attention was paid later to novel control and implementation systems. CNTs were shown to be 10 to 20 times more efficient than carbon black and it was also decided that 2.45 GHz gave a more efficient heating effect than 5.8GHz (both systems were supplied for trials) and would be the system of choice.

Induction heating

Tests by IVW on the AIMPLAS-compounded polymers showed that iron showed the best induction heating behaviour and that carbonous semiconductor particles didn't work as induction susceptors. The effect of frequency, concentration and particle size were studied and several solutions were applied to improve the heating homogeneity.

The outcomes were passed onto the later work packages: Iron and Magnetite powder (ferromagnetic particles) are suitable as induction susceptors and the maximum particle size was established as 150 microns. The additive loading was optimum at around 5-10wt%.and the chosen frequency was around 500 Hz with 7kW of power. It was also noticed that the best coupling distance was a constant 2 mm and if the sample was rotated over the coil it improved heating homogeneity.

Infrared heating

Both dark radiator and short-wave radiators were used for testing the effectiveness of infrared (IR) heating. It was found by AIMPLAS that additives had little or no effect and therefore selective heating was not possible. The heating was also effective only on the surface. It was agreed that IR heating would not be taken forward into the rest of the project.

- Task 2.4: Preparation of Matrix Materials for Subsequent Process Stages

Working with other partners AIMPLAS provided compounded samples which were analysed for heating efficiency in MW and induction processes with a feedback loop to optimise particle size, percentage of additive and dispersion. They were then able to supply various compounds to partners.

Having established the compounding methods and materials AIMPLAS also used software to first of all create an accurate simulation of their own processing and then to upscale the parameters to larger machines at PEMU. Various parameters were analysed: Residence time, dissipated energy, specific energy, melting energy, torque and mixing efficiency. This was successful and is described later in the report.

WP 3 Development of Reinforcement Fibre Systems

- Task 3.1 Modification of Fibre Polymers to Increase Temperature Stability

A number of activities were pursued in order to improve the temperature stability and properties of the reinforcement fibres:

Manufacture and Measurement of raw materials

From WP1 fibres (HT PET, HT PA46, HT PA66, PPA, PP, LCP, LPBT, PA6 and PCT) were selected and further tested, spinning them if required. The overall goals were to:

  - Improve initial modulus,
  - Improve overall modulus,
  - Reduce shrinkage,
  - Improve tenacity and
  - Reduce filament diameter

DSC and DMTA analysis was carried out to ascertain melt temperatures and mechanical properties and the following materials were chosen for the project:

  - PP – HTPP Polisilk
  - PET – Commercial grades (Comfil supply)
  - PA – Commercial grades (Solvay Amodel PPA)
  - LCP - Commercial grades (Vectran HS)

Heat Setting and drawing

One of the potential problems with the reinforcement element of an SRP is shrinkage of the fibre during processing and fibre shrinkage can start before the melt temperature of the fibre is reached. It is therefore preferable to modify the reinforcement fibre to be as stable as possible at as higher temperature as possible

The principle of heat setting is to take a yarn which has been manufactured and drawn and to expose it to a temperature above its shrink point whilst it is under tension and then to cool it back down to ambient. This has the effect of allowing the molecules to relax and they become more resistant to shrinking when they are subsequently re-heated. What was discovered is that heat setting reduces the shrinkage effect but the penalty is reduced mechanical properties. As expected, subsequent processing has a further negative effect on mechanical properties.

Irradiating

Irradiation of fibres by gamma radiation causes ionisation in the polymer molecules which promotes cross-linking between them, which, in theory, improves temperature resistance. Trials were carried out with PP fibres, as a standard fibre and as a fibre which has been extruded with a Sartomer (Trimethylolpropane Trimethacrylate) additive. This additive is recommended as promoting successful irradiation.

A selection of polymers (18 samples) were compounded with the additive, drawn and then sent for irradiation trials by IONISOS. The conclusions were that irradiation lowers PP yarn shrinkage and Sartomer further helps reduce this shrinkage. A FDY PP yarn with 3% Sartomer would see its shrinkage reduced 95% but tenacity would be reduced 20% which suggests polymer degradation or damage occurred. Therefore it seemed that irradiation probably increases the temperature resistance of PP yarn but will also reduce its tenacity.

Additives

The additives relating to reinforcement (as opposed to those already discussed in the matrix) were developed to improve the effectiveness of irradiation (Sartomer), discussed above, to improve the physical properties of the high tenacity yarn (CNT and nanoclays) and improve the resistance to temperature (CNT). Much work was therefore put into developing the extrusion of yarns from polymers compounded with CNT. Partner Comfil tried to extrude them into yarns, initially unsuccessful due to agglomeration of the CNT but with better dispersion supplied via AIMPLAS yarns were successfully drawn.

It was concluded that CNT reduces shrinkage of the PP yarn considerably but at the penalty of decreased tenacity of the yarn, as the more CNT content, less tenacity is achieved. CNT presence also allows a higher draw ratio than with standard PP, this partly compensates the loss of tenacity mentioned above. CNT slightly increases the Modulus of the yarns at low contents. Even though CNTs have proven to reduce shrinkage considerably these reductions were not enough to consider CNTs as a solution to the shrinkage problems of reinforcement yarn. At the same time modulus is decreased and there is no other advantage associated to its use.

Nanocalys were also used an additive. The masterbatch gave no problems when processed into yarns but it was clearly seen from the results that the mechanical strength is significantly reduced by the presence of nanoclays.

- Task 3.2 Performance Evaluation of Modified Reinforcement Polymers

Different Partners and suppliers use different units and properties when describing the strength/stiffness of yarns and so a method of standardising measurements for the project was needed so a standard method of obtaining comparable yarn modulus values was made, using a simple spreadsheet. It is important to know at what temperature the onset of shrinkage occurs in each of the reinforcement fibres and it is important to know “how” the fibres shrink, gradually over a range of temperatures or suddenly at one temperature. A hot-stage microscope was used for some initial investigation by NetComposites and they recorded a video of fibre shrinking over time as the temperature increased. This was later dropped as a test but it was recognized, that it is a useful bench mark for comparative work and that it provides data on the real onset of shrinkage.

- Task 3.3 Spinning and Drawing of High Tenacity Filaments and Tapes

Polymers selected form the results of Tasks 3.1 and 3.2 were melt-spun into fibres and drawn to achieve a high orientation and tenacity. The aim of task 3.3 was to increase the mechanical performance of yarns made of PP and PET. The task was expanded slightly as there are different ways to alter the mechanical properties in order to produce suitable yarn for the benefit of the aims of the project: increase yarn tenacity, increase modulus and reduce shrinkage. The chosen routes were: Carbon Nanotubes, Nanoclays, Nucleating agents, Taslan textured yarn to increase interface with matrix, an Adstif high modulus PP, Extrusion of PP yarn with Sartomer + Cross linking

Carbon nanotubes

The conclusions were that CNT decreases the tenacity of the yarn, as more CNT content, less tenacity achieved. CNT presence allows a higher draw ratio than with standard PP, where the maximum draw ratio is around 8. In PP+CNT draw ratios up to 9 were achieved, this partly compensates for the loss of tenacity mentioned above. CNT slightly increases the Modulus of the yarns at low contents and reduces the shrinkage of the PP yarn considerably.

Nanoclays

In SRP's the matrix plays a very significant part in the overall properties of the composite and in this task the aim was to improve the properties of the matrix polymer by the addition of nanoclays so that the benefits of the low melting point could be exploited to give a wider processing window. AIMPLAS produced a PP + nanoclay masterbtach that was processed into yarns by Polisilk. The masterbatch gave no problems when processed into yarns but the mechanical strength was significantly reduced by the presence of nanoclays.

Nucleating agents

Nucleating agents change the crystalisation behaviour of polymers. Several nucleating agents were considered and finally two versions were trialled. They were extruded by Polisilk but unfortunately gave no increase in mechanical performance

PP Taslan yarn

In order to improve yarn/matrix interface from the Fibroline powder impregnation process it was thought that it could be possible to alter the physical shape of the filaments. Instead of having flat filaments waves and loops were introduced to the filaments thereby improving interfacial bond and the mechanical properties of the SRP composite and also reducing the shrinkage of the fibres at high temperatures as they are firmly held within the matrix. Once again the advantages gained in terms of fibre/matrix adhesion were negated by the loss of mechanical properties.

Adstif PP

This grade chosen has a high Modulus but Elongation at break was somewhat lower (17%) compared to standard PP which is 20-22%. The biggest difference found was shrinkage in hot air at 130ºC for 1 min. which was only 2,4% compared to 9-10% in normal PP, modulus was 3,52 GPa. Because of the poor results in mechanical strength, Adstif was abandoned as a reinforcement yarn option, but continued as a matrix option.

Extrusion of PP yarn with Sartomer

The addition of a masterbatch Sartomer and further cross-linking via ionisation could help increase the thermal and mechanical properties of certain polymers by transforming a linear network of polymer chains into a three-dimensional network by a direct linking of carbon atoms. The results were:

- Ionisation lowers PP yarn tenacity about 25% in case of FDY, regardless of the presence of Sartomer.
- Ionisation lowers PP yarn shrinkage and Sartomer further helps reduce this shrinkage.
- A FDY PP yarn with 3% Sartomer would see its shrinkage reduced 95% with 25 kGy ionisation but tenacity would be reduced 20%.
- Ionisation will probably increase the temperature resistance of PP yarn but will also reduce its tenacity.

- Task 3.4 Commingling/Bi-Component Extrusion of Reinforcement and Matrix Polymers

Bi-component extrusion was not pursued as early trials showed that a significant loss of mechanical properties was suffered during the process.

Commingling was shown to be successful as a method of combining matrix and reinforcement for subsequent pultrusion (it can also be used for weaving fabrics). This process uses a system of feed and pull-off rollers to pass yarns through a controlled air-blast, resulting in intimately mixed multi-filament fibres. A very large number of samples were created by Comfil covering PET, PP, PA, PBT and HDPE. An innovation introduced for the ESPRIT project was to include a small percentage of black 'tracer' fibre (5%) into the reinforcement yarn. This is the same yarn but pigmented. This allowed easy visual examination of the SRP composite later in the process flow.

WP 4 Development of Flow Compounds

- Task 4.1: Processing of LFT Pellets for Injection and Compression Moulding

Two routes were pursued to achieve pellets:

- Pultrusion from commingled yarns
- Melt impregnation of reinforcement yarns with molten matrix

Pultrusion

The line built by Comfil for the project performed a series of operations sequentially. It fed commingled yarns under controlled tension to a preheating station before the die which heats and melts the matrix. This makes a consolidate rods which is cooled down and chopped into pellets. This line was highly successful and included some innovative features such as a two-stage drying and heating process, with an inert-gas shielding system and chopper to make pellets which could handle the normally difficult task of chopping non-brittle fibres. It made medium-sized batches of pellets for partners throughout the project and proved flexible in its ability to take various commingled yarns, at different ratios and with different base fibres.

Conclusions were drawn for each type of combinations fibres/ matrix based on the characteristics of the rods:

- Model material HTPET / PP: A very good polymer composite material which shows very good mechanical properties for a low price. For 30% in weight of reinforcement, the average properties would be: Average tensile modulus: 4 GPa, average maximum tensile strength: 200 MPa
- Self reinforced polyolefin srPO: It was difficult to produce such srPO due to high viscosity of HDPE matrix and low melting temperature differences. This type of SRP can be interesting for their impact properties and cost. For 33% in weight of reinforcement, the average properties would be: Average tensile modulus: 2.8 GPa, average maximum tensile strength: 110 MPa
- Self reinforced polyamide srPA: Despite a lot effort to improve SRPA, mechanical properties are not good maybe due to the oxidation of polyamide and the bad spin finish of fibres. Work discontinued on this topic
- Self reinforced polyester srPET: LCP – PBT combination is designed for specialty product due to his high mechanical properties and cost. For 31% in weight of reinforcement, the average properties would be: Average tensile modulus: 36 GPa, average maximum tensile strength: 740 MPa

For HTPET – PBT combination elongation at break needs to be improved. The PBT matrix needs to be combined with anti oxidation additives to reach the best properties. For 31% in weight of reinforcement, the average properties would be: Average tensile modulus: 5.5 GPa, average maximum tensile strength: 240 MPa

HTPEN – PBT gives a bigger temperature window for moulding than PET – PBT but for the same mechanical properties and higher price. This combination is abandoned in favor of HTPET – PBT. For 31% in weight of reinforcement, the average properties would be: Average tensile modulus: 5.9 GPa, average maximum tensile strength: 260MPa

HTPET – LPET combination is the most promising one regarding price / performance ratio. For 33% in weight of reinforcement, the average properties would be: Average tensile modulus: 6.5 GPa, average maximum tensile strength: 275 MPa The main limit could be is the low thermal resistance of LPET matrix.

Melt impregnation

A series of tests were carried out on the pilot plant at Celstran with combinations of PET/PP, PA66/PA6, PA66/PA12, PA46/PA12, PET/PBT, PP/HDPE, PP/MDPE and LCP/PBT.

Conclusions can be drawn for each type of combinations fibres/ matrix:

- Model material HTPET / PP: It is a very good polymer composite material with shows good mechanical properties for a low price PP. Matrix viscosity has to be very low in order to impregnate the PET fibres. It seems that moulding temperature have more influences than impregnation parameters. For 30% in weight of reinforcement, the best properties would be: Best tensile modulus: 2, 38 GPa, Best maximum tensile strength: 55,9 MPa

- Self reinforced polyolefin srPO: It was difficult to produce such srPO due to high viscosity of mPP or HDPE matrix and low melting temperature differences. For 30% in weight of reinforcement, the best properties for a HTPP/mPP SRP would be: Best tensile modulus: 2,02 GPa, Best maximum tensile strength: 21,9 MPa

- Self reinforced polyamide srPA: A lot of trials of srPA were carried out in order to evaluate the influence of fibres, matrix, additive, process parameters without success. Whatever was the improvement, the final properties are low. For 30% in weight of reinforcement, the best properties of a PA66/PA6 SRP would be:
  Best tensile modulus: 3,28 GPa, Best maximum tensile strength : 74 MPa

- Self reinforced polyester SRPET: The LCP – PBT combination is disappointing due to the very low balance of mechanical properties vs. price. For 10% in weight of reinforcement, the best properties of a LCP/PBT SRP would be: Best tensile modulus : 3,56 GPa, Best maximum tensile strength : 49 MPa

For HTPET – PBT SRP, mixtures of different PBT were tested to optimize processing and properties. Best mixture gave an improvement of 15% of the modulus but divided by 2 the maximum strength. So results were very poor and CELESTRAN decided to stop the development on PBT. For 31% in weight of reinforcement, the best properties of a HTPET/PBT SRP would be: Best tensile modulus : 3,14 GPa, Best maximum tensile strength : 33 MPa

The conclusion of this task showed that as a first approach it was good to use model material, HTPET-PP, to validate the feasibility of LFT. Then, numerous trials of polymer combinations led to improvements of the both impregnation processes. The two companies involved in this work have produced many batches of material for the other partners with numerous variations based on the type of pellet, amount of fibres, fibre preparation and different additives in the matrix. For some of these SRP, the mechanical properties are very interesting and choices were focused on the following the most interesting combinations:

- Task 4.2: Development of Sheet Semi-Finished Materials for Compression Moulding

The route to achieve the semi-finished sheets was the Fibroline powder-impregnation process, which had not previously been used to manufacture thermoplastic composites or SRP materials. The first work done was to characterise the possible matrix and reinforcement options using the same 3 basic polymer families agreed in WP1.

The powder impregnation line passes the reinforcement (as a chopped, UD, non-woven or woven textile) on a conveyor through an electrical field which carries micronized (powdered) particles into the substrate. The resultant combination is hot-calendared between rollers to give a semi-finished SRP sheet which can subsequently be moulded. One of the advantages of this process is its flexibility to be able to combine any reinforcement with any matrix in any desired ratio. Sheets were initially made with chopped fibres, made in-situ with chopping equipment cutting continuous yarns. Non-woven needle-punch fabrics were also impregnated, these being commercial textiles bought in rather than made on the line. These were followed by uni-directional SRP semi-finished sheets made from small-scale wound samples which proved that impregnated UD was very possible.

Demonstrating the flexibility of the Fibroline process continuous non-woven sheets were also made, again with the laying of the continuous fibre being carried on the impregnation line directly. The various material and process combinations were characterised and evaluated and the UD samples showed particular promise.

Unidirectional fabrics were up-scaled in PP and produced in larger quantities to allow larger impregnation trials. The materials manufactured by the D-PREG technology developed by Fibroline show great promise and were used to make good case study parts later in the project.

- Task 4.3: Development of Microwave, Induction, Infrared and Conduction Heating Methods

Microwave

The initial version of the microwave machine, received from F&M and installed in the workshop of NetComposites was a based on a Monomode magnetron, with a maximum power of 1000 W and a single frequency of 2.45 GHz. The machine had a rotating chamber into which pellets could be loaded, the rotation being used to distribute the energy more evenly. This configuration did not work well and resulted in over-heating areas. It was noticed that the device used to fine-adjust the reflection of the MW, to maximize power usage, could also be used to change the focus of the MWin the chamber. This led to the concept of manipulating the MW inside the device and the machine was modified to try this concept.

Ultimately, after development this machine was capable of heating sheet materials with a high degree of consistency over the surface. Original thermal images show the heating effect without the reciprocating plunger and demonstrate the peaks in heating caused by the standing microwave. After developments the thermal images show a sheet using the manipulated MW with good heating homogeneity and the graphs which show the temperature profile across the sheet ate several points. For any given profile the temperature variation is 10 to 20oC which is very promising and can be improved with further refinement of the control system and additional programmability. The profiles show difference to each other because the sheet is cooling as it emerges from the machine. An alternative system supplied by F and M used a chamber with multiple antennas which was trialled and did not work in its initial format. There were some issues with MW escaping the system which were not resolved in the lifetime of the project but which will be worked on after the project finishes.

Induction

Trials were carried out on the materials generated in WP 2 and 3 using new induction heating equipment at IVW, a Hüttinger TruHeat HF 5010, 10 kW with a cooling device, running at between 100 and 500 kHz (discrete setting not continuous) and with the associated infrared pyrometer to control heat output.

Different additives were evaluated with the optimised parameters being Iron particles with size < 100 µm, frequency around 500 kHz and initial energy about 6 kW

It was proven that with the correct additives and control system induction heating could heat a polymer, hold it at temperature and then allow cooling. This shows very promising results for later applications.

Conduction heating

Conduction heating refers to using a more conventional hot water or oil system to heat tools direct, in order to control heat or to provide a complete cycle of heating and cooling. ESPRIT partner Regloplas developed a system particularly suited to tightly controlled temperature, quicker cycle times that use the energy-intensive process of heating and cooling, an unusual requirement from the use of SRP materials.

A dual circuit system was developed which continuously runs hot and cold channels from 2 separate units, one heating, one chilling. Early trials of this dual-heating system were used at Promolding for injection moulding.

Later in the project the 'energy battery concept' was introduced which allows the storage of hot or cold energy for later re-use when the cycle goes full circle, shown on right. Several iterations were trialled as there are particular problems in controlling hot and cold systems in a very dynamic way. Concurrently an advanced control system was developed to provide active, real-time feedback control based on the can-bus system. The system has been successful enough to generate immediate customer interest

- Task 4.4: Physical Analysis of Semi-Finished Materials Pre- and Post-Heating

The main objective in this task was to evaluate the performance of the materials, processes and outputs in order to see what effect additives, repeated heating and cooling, pressures and so on would have on flowing SRP materials in the form of pellets and sheets.

Performance of pellets after injection:

- Fibre content: Tested from 0 to 57% w/w and increase of tensile properties stops after 37% w; there was no improvement from 37 to 57% w/w
- Fibre length: Tested lengths from 1 to 20 mm. Better tensile modulus and strength with shorter fibres. Shorter fibres are better distributed
- Injection temperature: Parameter from matrix melting to fibre melting. Better tensile modulus and strength for lower temperature
- Ageing time: Ageing time tested from 2 to 200 hours. Cold crystallisation needs time to take place. For PET/PP and PET/PBT pellets ageing time must be = 1 week

Performance of sheets after compression:

- Fibre content: Increase of properties from 40 to 60% w/w
- Compression temperature: Better tensile modulus and strength for lower temperature
- Compression pressure: Tested pressure: 10 to 40 bars. No loss of pressure needed, No influence of pressure during hot step. Better properties with lower pressure during cold step

WP 5 Net-Shape Injection and Compression Moulding Processes

- Task 5.1: Injection Moulding using Conduction Preheating of LFT Pellets

This task involved taking the new compounds and the knowledge gained about materials and their processing limits and investigating the possibilities of injection moulding flowing SRP. The particular challenges are to overcome uncontrolled heat generated by shear in the barrel, nozzle, sprue and tool and to maintain the required processing window.

Three streams of development were identified, in order of desirability:

- Standard and adapted processes to utilise SRPs for operators who do not wish to invest in new equipment. Needs a large process window with wide difference between melting of matrix and fibres.
- New and adapted processing machinery like piston and short screw design which can be used also for SRP with a narrow processing window.
- New developed materials with heat-promoting additives are can achieve processing through heating with special type of energy like micro waves or induction.

A multitude of tests and evaluations were carried out with injection moulding machines and their elements such as:

- Side-feeding units,
- Vertical feeding units,
- Crew designs and configurations,
- Nozzle designs,
- Mould and sprue design.

Both the hardware, as well as the process parameters were examined:

- The humidity of the used materials,
- Size of pellets,
- Melt and nozzle temperature,
- Plastification time,
- Injection profile,
- Screw position control
- Injection pressures and Velocities,
- Filling time,
- Holding time under pressure,
- The cycle time of the process

A series of test specimens (tensile and impact bars, plates) were produced and mechanically tested. It was found that the best mechanical values are reached after a period of app. 2 weeks after production, allowing full crystallization of the polymer therefore a method was built up by the consortium to follow up the history of material grades (on the ESPRIT website) and the samples.

This task concluded, after a review of all possible processing variants suitable to be used for the manufacturing of SRP, with some initial recommendations:

- The used processing concept fits to SRP structure
- The fibres survive to the end of cavity, but fibre relaxation effects occur by the higher temperature level in the core layer of the 4 mm parts
- The fibre orientation is better if shear heating is low during filling
- Mechanical properties are better using a shorter cavity (only tensile bar)
- Mechanical properties are better using a reduced cavity height of 2 mm
- The outer layer is at the moment quite thick and of lower orientation
- Mechanical properties have a potential to be further increased getting higher orientations
- “Playing” with shear for orientation is limited by higher fibre relaxation or/and degradation

These were further refined during the manufacture of case studies and some final recommendations made for a 'Best Practice' document

- Task 5.2: Compression Moulding using Induction, IR & Microwave Heating of Semi-Finished Sheet

As detailed earlier, IR heating was discounted early on.

Compression Moulding using Induction

For the compression moulding trials with induction heating, Fibroline HDPE/PP sheets as well as Celstran PP/PET tapes were used. The manufactured plates with the dimension of 120 x 120 mm² had a thickness of 2 mm. These sheets were then tested and characterised (fibre integrity, impact).

The optimal results were then trialled practically, using a moving induction coil to heat a sheet and to form a shoe cap, as used in protective shoes. In contrast to a convection oven the heating time of semi-finished sheets could be reduced by 40 %. Additionally, the induction heating process indicated limits in terms of component size and efficiency: a lower temperature at the edges in comparison to the centre of the semi-finished sheets due to thermal losses to the environment was observed. The application of induction assisted particle heating was revealed to be a feasible method for the preheating of self-reinforced materials in thermoforming processes. The method was able to decrease the necessary preheating time in comparison to a convection oven. Furthermore, the impact results and the microscopy observation lead to the conclusion that the induction heating does not damage the fibrous reinforcement and decrease the material impact properties.

Microwave Heating of Semi-finished Sheets

For the microwave trials six different self-reinforced plastics were used all of them supplied by Fibroline, Each material had CNT in the matrix to increase the material microwave sensibility. These sheets were pre-consolidated in a press, to make thicker sheets suitable for the manufacture of test specimens. As described previously, the modified MW machinery was used to carry out a series of trials on the above materials with a wide range of parameters.

The conclusions for the microwave heating of semi-finished sheets case-study were:

- The woven fabric material displays much more homogenous heating due to greatly improved matrix distribution compare to the random reinforced SPR;
- All the pre-consolidated sheets still having small problems with CNT agglomerations;
- Increasing the matrix of a random reinforced material to increase the microwave sensibility doesn't give the effect that was expected (quicker heating);
- The manipulated MW significantly improves the homogeneity of heating across the material;
- The microwave technique is fast and efficient with polypropylene based sheet material heated to moulding temperature in only a few seconds using a 1KW magnetron;
- Microwave technology is scalable with the inclusion of larger capacity/additional magnetrons and wider wave guides.
- The microwave heating efficiency is between 50% and 60%.

- Task 5.3: Compression Moulding using Conduction Preheating of LFT Dough

The objective of this work package was to investigate the feasibility of self-reinforced pellets in an LFT dough process. The experimental series contained an analysis of the rheological properties of the compounds at IVW. Within the rheological study, the viscosity and appropriate dough temperatures were investigated. Subsequently, feasible parameter setups were used for the manufacturing of moulded sheets which were analysed in tensile as well as impact tests. A demonstrator was manufactured to show the good applicability of the LFT dough process for SRP pellets. Similarly, PEMÜ investigated the processing of hot dough materials by extruding and injection moulding.

For the moulding of the preheated dough, a hydraulic press, an industrial standard machine was used. Several moulds were used for the trials which included, besides the rheometer setup, a shear edge mould for a sheet (540 x 540 mm^2) and a complex shaped tool with ribs for the later manufacturing of the demonstrator part.

The rheometry tests confirmed that PP/PET and HTPET/LPET could be extruded, transported to a tool and moulded by the application of pressure. It gave an indication of the optimum (minimum) temperatures to achieve mouldings. Flat sheets were then moulded and the tested by impact and tensile methods to again verify the optimum processing parameters and to show the mechanical properties of dough-moulded SRP.

Finally a more complex shape was moulded. The hot 'dough' loaded into the tool direct from the extrusion machine and, below right, is shown the moulding with thin internal ribs. The part is a U section of approximately 50mm x 50mm cross section, the lateral and longitudinal ribs add further challenge, but the part was 100% filled with PP/PET but was only partly filled with the HTPET/LPET version.

Within this task it was demonstrated that the processing of SRP pellets in a LFT dough process chain is feasible. The PP based material system revealed good rheological properties resulting in a well processed demonstrator. The viscosity of the material allows also the filling of complex cavities. The self reinforced polyester revealed higher mechanical properties than the investigated PP-PET system but resulted also in a higher viscosity. The viscosity was measured in the range of 10000 Pas and did not allow a sufficient flow processing within complex moulds. Nevertheless, the filling of sheet-like moulds was possible. The mass temperature of self-reinforced materials has to be chosen to be a minimum to preserve the performance of the polymeric reinforcement. Nevertheless, a compromise has to be made since a lower mass temperature simultaneously causes a higher viscosity which limits the application of the respective material within complex tools.

- Task 5.4: Mecanical and Physical Characterisation of Composite Panels

In order to evaluate the level of success of the various material and moulding combinations generated by the project it was necessary to carry out tests to benchmark against current, non-reinforced or none-SRP variants and to compare results of the modified composites in order to optimise them.

During the project Promolding developed and built a new tool, having some desirable functionality regarding the SRP materials used. The following features were implemented in the mould:

  - Tensile and impact test bars can be made with variable thickness to test the influence of wall thickness on the mechanical properties due to orientation effect at the wall
  - Tensile test bars can be injected on 2 sides to test weld-line strength
  - All 4 runners can be opened or closed independently
  - All 4 gates are interchangeable parts
  - A pin gate and various thickness of the film gates are at choice to test influence of gate size on mouldability and mechanical properties

Using this tool and a variety of materials and processing conditions a number of critical effects were studied:

  - Melt flow in barrel, screw and cavity
  - General influence of plastification parameters and optimum screw speed
  - Influence of temperature on processing
  - Influence of injection speed on processing
  - Plastification under reduced back pressure
  - Influence of pellet length on feeding

The moulded parts were delivered to partners and were then mechanically tested. The results were fed back to the sample production as feedback loop for further optimisation. The conclusions were:

  - SRP materials can be processed on conventional injection moulding machines although care should be taken that the fibres should not be subjected to excess heat and shear. The following settings are advised for optimal mechanical properties:
    - Very slow feeding speed
    - Low back pressure, although some may be useful in dispersing the fibres.

    - Flat temperature profile. Advised processing temperatures:
        - PP matrix: 170-180 °C.
        - PBT matrix: 230 °C.
        - LPET matrix: 180-190 °C.
    - A slow to medium injection speed should be used.
    - A large diameter nose, sprue, runners and gates are advised.

- Task 5.5: Processing of Complex Shapes

Related to task 5.4, this task gathered the knowledge of specific processing limitations and guidelines necessary for the ultimate end users for the design of components and tools suitable for SRP injection. The areas studied, and shown in detail in Deliverable 5.5 were:

  - Injection phases in the cavity
  - Mould filling mechanism and orientation effects in SRP materials
  - Microscopic studies of model system and SRP (trace fibres)
  - Influence of gate size
  - Influence of wall thickness
  - Influence of fibre length on flowability
  - Influence of weld lines on mechanical properties
  - General processing remarks

In summary the conclusions were:

  - The processing windows for plastification and injection are significantly influenced by nozzle, gating and cavity design.
  - The performed studies showed that an exact temperature management is crucial for repeatable properties in the part.
  - A very low shear management for SRP during plastification and injection by using low screw and injection speeds prevent partial distortion effects of the SR compound.
  - Mixing or shear elements in the homogenising area at the screw end are not suitable for SRP materials
  - The backflow valve and nozzle tip design needs wide channels and no sharp edges and/or flow hindrance in order to reduce shear heating

The effects of processing variations

The analysis of different parameters that influence flow behaviour of SRP material were analysed in order to obtain a general route for an optimised part geometry made from SRP material. This work can be summarized as follows:

If the recommendations for the process and material have to be guaranteed as described in the chapters above, then:

  - Relaxation effects can occur by the higher temperature level in the core layer of parts > 4 mm wall thickness
  - Fibre orientation is higher if shear heating is low during filling
  - Mechanical properties are better using a shorter cavity
  - Mechanical properties could be enhanced using a reduced cavity height (sample thickness)
  - The outer layer has a lower orientation when using low injection speed

- Mechanical properties have a potential to be further increased getting higher orientations by fast filling
- Tailored orientation by flow effects is limited because of high fibre relaxation and/or degradation
- Sprue and runners should be wide enough to minimize shear heating (> 6mm)
- Gates should be wide enough to minimize shear heating (> 3mm)
- Fibre depleted areas could be observed in very thin sections
- Sharp edges and flow undercuts should be avoided
- Weld lines on highly loaded parts should be avoided: weld lines are relatively weak points because the fibre network is less strong

WP 6 Application to Case Studies

- Task 6.1: Detailed Part Specification

In this task it was intended to build a detailed specification for a limited number of case study parts .A good knowledge was built through the project of the advantages and limitations of the materials being developed and the partners put forward a large number of possible case study parts based on the key characteristics of impact resistance, light weight and stiffness. These are fully described in Deliverable 6.5 and whilst all have specific requirements some are more formally documented than others. A cost and property table was drawn up for injection moulding pellets and gradually filled towards the end of the project and this helped determine suitability.

It was seen the cost of ESPRIT materials is significantly higher than the comparable commercial polymers in a weight for weight comparison but it should be born in mind that less material can be used so PP/Pet and HTPET/LPET start to look more competitive. A similar exercise was carried out for the sheet materials and these materials can be seen to be more competitive and therefore be possibly closer to market. Out of the case study parts described below the Under Body Protection, Door Module, Battery Tray, Nail Gun Magazine and Shin Guard all had desired properties specified.

- Task 6.2: Complex Injection Moulded Component

Door Module

A challenging part that requires a long tool travel for the injection material and flow characteristics to form details: this part is currently made in PP-GF20 and several ESPRIT materials were trialled, varieties of srPP, srPET. For all tested materials, including the reference material, the highly detailed loudspeaker web could not be filled completely but for the main body of the part the Comfil PP-HTPET performed very well, pellets contained trace fibres which were dispersed evenly throughout the moulded part. Even the loudspeaker web, which is the most difficult part to fill, was filled almost completely.

Sunvisor

Although it was not a good example of a part that would benefit from SRP materials the detailed rib design of this tool made an interesting challenge. The materials trialled were PP-HTPET30 from Ticona and LPET-HTPET33 from Comfil, the latter of which provided a full fill of the part and improved mechanical and impact properties.

Fire Hydrant Cap

This is a thick-walled cap which needs to resist high hydrostatic pressures. Tests showed that the PP-HTPET model system was sufficient to replace the current material. The LPET-HTPET was not sufficient for replacing the current material. Due to the lower density of the PP-HTPET material, the price per part is similar to the original material. Also the maximum pressure in the hydrostatical pressure test is of the same order. However, a big advantage of the ESPRIT material is that in case of a pressure burst, it will break in a tough manner, whereas the current GF filled PA will break brittle into pieces that possibly could cause injuries

Climbing Frame Covers

Requirements for these demonstration parts are a very high impact resistance, stiffness and a high UV stability, currently in TPU. Celstran PP-HTPET30 with UV stabilizer was trialled as replacement for TPU in three different moulds for this application. The part could be moulded but for the successful use of Celstran PP-HTPET30 as a replacement for TPU the gates should be optimised and the surface should be improved, maybe by using higher mould temperatures.

Fan Blade

This demonstrator is a fan blade which is part of 9 bladed fan for distribution of heat in large buildings such as greenhouses. A high stiffness and low density material is needed to prevent bending of the asymmetric blade when spinning fast. In addition, the material should be ductile enough for having a snap fit. In general the snap fit is too brittle for most glass filled materials. ESPRIT SRP materials are ductile and have a good stiffness/weight ratio to fulfil this application. PP-HTPET30 displayed no problems with mould filling, although in very thin sections the matrix material squeezed out the fibres. A very ductile part was obtained, which hardly showed any warpage. However, the modulus was much lower compared to PP-GF40. Therefore a hybrid between HTPET and glass fibres was tested to give the best of both worlds, i.e. a high stiffness and ductility. I was shown that ESPRIT PP-HTPET can be easily blended with commercial PP-GF in order to tune the stiffness and impact of the PP-HTPET.

- Task 6.3: Large High Aspect Compression Moulded Component

Under Body Protection. Volvo

This type of component is conventionally produced by compression moulding of LFT or GMT. This part requires an excellent impact performance. Trials were performed with various woven and non-woven sheet materials, injection moulded sheet and extruded sheet. Extrusion and injection moulding of sheet material was done from Comfil pellets by PEMU. Because contact heating was used the material need to be clamped to prevent shrinkage. The stiffness of the Fibroline materials was insufficient. The Comfil material displayed better stiffness, although processing was challenging because of the weave structure.

When comparing the properties of the best practice material (Seeberlite 2 146) and ESPRIT SRP it became clear that the Seeberlite material outperformed the ESPRIT materials on basis of cost, weight, mechanical properties, acoustics, and heat resistance. Nevertheless, the ESPRIT LPET-HTPET displayed better flammability as being self-extinguishing.

Complex Y-shaped Geometry Using LFT dough

This compression moulding demonstrator mould was a U shaped profile in a Y-shape geometry. The die contained several ribbings for examining the flowability of the dough. For good quality products Comfil PP-HTPET needed to be processed at a temperature of 195-200°C, which was a higher temperature than used for optimal mechanical performance, namely 180°C. Besides the higher temperature, the processing was unproblematic with PP-HTPET. The mould could be completely filled without voids resulting in a good demonstrator surface quality. In contrast, the processing of, LPET-HTPET was more difficult. The high viscosity led to an incomplete mould filling which resulted in frozen melt flow fronts and voids within the demonstrator.

Shoe Cap

This demonstrator was selected to investigate whether SRP is suitable for compression moulding using induction heating. The material used for this demonstrator was HDPE-PP sheet with iron additive. The sheets (120x120x2 mm3) were heated by rotation above an electric magnetic coil (453kHz, 35 A, ~700 V, ~6.8 kW). By using a heating time of 7 minutes the sheet surface temperature was between 130 and 140°C. During compression moulding, some problems occurred with cooling of the edges due to convective and radiative heat losses, which have not been solved so far.

Battery Tray

This demonstrator is a compression moulded battery tray which is currently made from PP-GF 20% sheet material. Fibroline srPP sheet was trialled as a replacement, processing at 140°C using a 60 sec preheating cycle proved to be acceptable to produce a battery tray with good surface quality. In addition battery trays were prepared from SRP pellets which were first injection moulded or extruded into sheet material. Celstran PP-HTPET and Comfil srPET were trialled.

Foot Plate

PEMU have manufactured a foot plate demonstrator. This part was originally made from PP. Because the main requirement is impact resistance this is a good demonstrator part to be replaced with SRP. Celstran PP-HTPET, Comfil PP-HTPET and Comfil srPET have been used to prepare the plates. Due the large gate size (20 mm) used, no problems were observed during mould filling, even with the viscous srPET material. Also the fibres were not damaged due to the processing. Thanks to modification of the mould plates of 500 x 500 mm can be produced.

- Task 6.4: High Performance Part

Shinguard

This part has been designed and specified by Progressive Sports Technologies Ltd and is a high performance demonstrator part for compression sheet moulding, requiring lightness with excellent impact protection. A compression mould was designed and produced by Promolding to demonstrate production technologies on this product using conventional heating, microwave heating and the Regloplas cyclic heating/cooling system. Heating/cooling channels are very close to the tool surface to minimise the mass of steel that is heated. For the first trials the materials were heated using conventional heating between two plates. Materials trailed were: Extruded Comfil srPET sheets, Fibroline powder impregnated HDPE-HTPP continuous random fibre sheets, Fibroline powder impregnated srPP with CNT 2x2 twill woven sheets, Fibroline powder impregnated srPP random fibre sheets with and without CNT, Fibroline powder impregnated PP-HTPET random fibre sheets +CNT.

Compared to conventional heating the cycle time and (surface) quality of the shinguard was highly improved when the Regloplas cyclic heating/cooling system was used. The shinguards were characterized by blunt and stud impact tests. These tests simulate the amount of energy a person feels upon impact. Compared to a commercial shinguard a higher amount of resultant energy and less deflection was measured for the srPP ESPRIT shinguard indicating that the energy felt by a person upon impact for the ESPRIT shinguard was lower.

Magazine for Nailgun

This demonstration part is originally manufactured by injection moulding of TPU-LFG60. Requirements for the nailgun are low warpage, high stiffness at high temperatures, and good aesthetics. In total 5 demonstrator parts were prepared. 2 demonstrator parts were prepared from PP-HTPET, 2 from PP-HTPET + black colorant and 1 from TPU-LGF60. The ESPRIT materials were processed at 210°C. Lower processing temperatures will produce better mechanical properties but this temperature was necessary to obtain good aesthetics. The magazines were characterized by bending test and charpy impact tests. Compared to the commercial TPU-LFT60 material, the PP-HTPET displayed better strain at break but worse flexural modulus and flexural strength. The charpy impact results are not meaningful since none of the samples broke during testing. Addition of the colour masterbatch resulted in a severe reduction of flexural strength and strain.

Rod Stabilizer

The original material of this demonstrator part is 50 wt% glass filled semi-aromatic PA which comes at a cost of ~7 EUR/Kg. This demonstrator part was selected in order to investigate whether ESPRIT SRP material is suitable for gas injection, a rather complex processing operation. PP-HTPET was the material selected for these trials. Although melt blowing using PP-HTPET was possible, high pressures over 250 bar were necessary to create hollow spaces in the core since long polymer fibres tend to stick in the core layer.

- Task 6.5: Technical and Commercial Evaluation of Prototype Parts

The main fibre matrix combining techniques studied in the ESPRIT project were commingling and melt impregnation to prepare SRP pellets for injection and compression moulding and D-Preg powder impregnation to produce a dry mat of reinforcement impregnated with fine matrix powder.

The commingling process, i.e. intimate mixing of dry yarns, developed at Celstran showed little problems in producing SRP pellets and can in principle be used for any type of material which can be spun into a fibre. The melt impregnation process from Celstran – reinforcement combined with molten matrix via a heated/cooled tool, displayed a lot more problems and was only successful for PET reinforced PP matrix composites. The D-Preg Powder impregnation process of Fibroline, i.e. a dry mat of reinforcement impregnated with fine matrix powder was successful as long as the matrix can be micronized into a fine powder. This process was successful in producing real SRP, such as PP-PP and PET-PET.

Injection moulding appeared difficult but possible by carefully controlling the process parameters to minimize fibre shrinking and breakage of fibres due to excessive shear and temperature. Due to the high viscosity of certain SRP combinations (particularly those with an LPET matrix) flow problems could arise during injection moulding of complex shapes which can give rise to underfilling of the mould and fibre depleted areas in thin sections.

Compression moulding was successful for both sheet and dough. The high viscosity of some SRP material was less of a problem for compression moulding SRP materials. In addition, fibre-matrix combinations with a smaller temperature gap of their melting points could be selected.

To become effectively competitive with existing materials, the main issue at this moment is the price of the new SRP materials. This has mainly to do with the small scale production of the new materials that would have to compete with existing materials that are produced much more efficiently in enormous volumes. However, the production techniques that are developed can easily be scaled up if a larger demand would exist, so the is no fundamental problem for a more competitive position in the future.

WP 7 Dissemination

- Task 7.1: Web Site Development

The project website has a number of roles: To inform the wider public about the project, To act as a source of data for interested parties, To act as a collection point for contacts, To have a partner only intranet area (see task 8.2)

The domain name was purchased and a basic website was in place for the project Kick-off meeting, the website address being http://www.espritproject.eu. The website was maintained and updated regularly with flyers, bulletins and changes to the content as the project progressed. As a summary of the number of visitors (4052) unique visitors (2780) and other analysis over the 3.5 year project as well as a list of the nationality of the top 20 visitor countries. Interestingly there are high levels of interest from the USA and India and overall 90 countries were registered as having visited the website showing that there is a global interest in SRP technology.

The website will be maintained for the foreseeable future by NetComposites. There is a Final Technical Overview 12 page brochure on the website to download which describes all the achievements and technologies created through the project. Also available as downloads are the presentations from an Information Day held at Q13 in the project, which summarise the findings.

- Task 7.2: Exploitation Plans and Dissemination

Dissemination

The ESPRIT project has been active in publicising its activities throughout the projects life span. Presentations were made at the following events K Fair 2010, ECCM14 Budapest 2010, Kolloquium Germany 2010, ICCM 18 Korea 2011, ECCM 15 Italy 2012, FPCM 11 New Zealand 2012, Composites Recycling France 2011, JEC France 2011, Opportunities in Thermoplastic Composites UK 2010, Composites Eng Show UK 2011, Composites UK 2011, JEC Forum Paris 2012. In particular the ESPRIT-sponsored forum held at JEC was a high profile exercise with the ESPRIT name, objectives and results put before one of the worlds premier composites events.

A number of academic papers have been also been written:

  - Induction Heating of Thermoplastic Materials by Particulate Heating Promoters, T Bayerl
  - Melting of polymer-polymer composites by particulate heating promoters and electromagnetic radiation, T Bayerl
  - Selective Heating Applications for the Processing of Polymer-Polymer Materials, 15th European Conference on Composite Materials, A Benedito
  - Processing of Long-Polymer-Fibre Reinforced Thermoplastic Pellets by Compression Moulding, T Bayerl

Press releases were made 4 times during the project resulting in magazine articles in many journals and publications including European Plastics and SA plastics (South Africa). In addition a flyer was produced early in the project (in English and Spanish) and a Technical Bulletin in 2011 as well as several posters used at trade shows and one which won a small competition in the UK (Materials KTN Annual event).

Contact details were collected through the life of the project to create an informal 'End User Group' and the list was used to invite interested parties to 'Information Days' at AVK and Celstran, adjoining Quarterly project meetings. The event at Celstran attracted representatives from Toyota and Samsonite amongst others (shown on right)

IVW and AIMPLAS supplied a chapter 'Melting of polymer-polymer composites by particulate heating promoters and electromagnetic radiation' for the book 'Synthetic polymer-polymer composites', not yet published at the time of writing.

The work of ESPRIT has featured on the partner's websites, particularly NetComposites, Comfil, AIMPLAS and Promolding and EATC.

Exploitation

The purpose of the ESPRIT project is to develop materials and technologies which can be taken forward for commercial benefit of the partners, their industries and, most importantly, the European plastics industry as a whole. One of the main tools used to focus the aims of the partners was the Exploitation Strategy Seminar service offered by the EC through Cimatec. The initial seminar was held on 30.06 2009 at NetComposites for one full day, before the regular Quarter 3 General Meeting. The standard format of generating exploitable results, categorising them and analysing them for possible routes to exploitation, partnerships and post project investment was followed. This was seen by the partners as a useful and interesting exercise although at that relatively early point of the project it was difficult to be certain about the most likely routes to exploitation. Therefore follow-up sessions were held as part of the regular Quarterly meetings and a specific session in the Q11 meeting June 2011. The most useful part of the ESS was the production of the foreground/background/ownership/use table which facilitated discussions between partners about future work. The ESS was also helpful in producing the Plan for Use and Dissemination of Foreground Knowledge.

Towards the end of the project an Exploitation Sub Committee (ESC) was set up, starting at the Q 9 meeting in January 2011, to help activities in the last year of the project. This was successful in organising the 12 page Technology Overview document and the Information Day.

Following the completion of the project there are a number of possibilities for future commercialisation:

- Fibroline have an evaluation agreement with a consumer goods manufacturer using their preconsolidated sheet materials. NetComposites have also received an enquiry from a similar consumer goods manufacturing company and are working with Fibroline on exclusivity issues. Both of these enquiries are interesting as they are organisations seeking alternatives to the current growing Curv, Pure and Tegris technologies, showing that there is a significant market interest.
- Fibroline and Polisilk have a partnership for the supply of high tenacity PP reinforcement yarns
- Comfil have on-going negotiations with orthoses suppliers for fabric-based SRP applications and are actively selling SRP pellets of HTPET/LPET, HTPET/PBT and LCP/PBT.
- Regloplas are already selling dual-channel heating systems
- Regloplas are close to being able to sell the new energy battery concept.

There are also some other spin-off benefits:

- AIMPLAS have already successfully used the compounding knowledge gained to produce high quality expanded graphite and grapheme compounds for the Nanomaster project (285718)

- NetComposites have submitted a proposal for a novel selective moulding process, through to the second round of the 2013 FP7 call (FP7 310409-2), utilising conduction and induction techniques developed in ESPRIT.

Patents

NetComposites have carried out a novelty search on the scanning microwave technology to judge the possibility of applying for a patent. The search reveals that similar ideas have been patented in general terms for manipulated microwaves and also for polymers with susceptors although none actually combine the two. It has been decided not to patent the technology but to develop it further through their own trials in thermoset curing and improvements to the control system in order to try to achieve either a commercial application or a more refined, patentable process.

Some key exploitable outputs

- Compounding
  - High quality compounded polymers have been made with excellent additive distribution
  - Carried out on the most challenging of polymers – PP
  - Theoretical modelling of compounding used to predict up-scaling parameters
  - Up-scaling of the optimum compounding parameters has been carried out to a high level of success
  - The methods used in the compounding can be transferred to other nano-level additives
- Pultrusion
  - The creation of a pilot pultrusion line to manufacture SRP pellets
  - Development of a cutter for cutting SRP rods into pellets
  - The spinning out nanomodified matrix polymer as multifilament yarn
  - Production of pultruded SRP pellets with 30% weight reduction in final samples
  - Production of pultruded SRP with 30 – 200% increased modulus compared to pure polymer
  - Pultruded production of economically viable HTPET/PP and SRPET
  - Knowledge about SRP processing and properties vastly increased
- Melt Impregnation
  - Modification of a pilot line to produce SRP pellets by melt impregnation
  - Melt impregnation production of SRP pellets based on PP and PA.
  - Production of economically viable HTPET/PP by melt impregnation
  - New contacts for sharing of interests, exchange of experiences
- D Preg Powder Impregnation
  - Development of a new spreading unit for in line D Preg production of non-woven
  - Set up of a pilot line to manufacture SRP sheets by D Preg
  - Manage to produce SRP with 30% weight reduction for PP/PP sheets
  - Manage to produce economically viable PP/PP SRP sheets by D Preg
- Extrusion
  - Extrusion of sheets from SRP pellets has been demonstrated
- Conduction Heating
  - Development of high pressure valve arrangements for dual channel heating
  - A completely new energy battery concept tested
  - Integrated control systems for dual channel heating
  - Commercially ready systems on the market
- Induction Heating
  - Additive suitability fully investigated
  - Accurate heating and maintaining of said temperature successful
  - Roc Tool technology shown to work well with SRP materials.
- Microwave Heating

    - Practical knowledge of MW systems gained
    - Novel solution of manipulated MW shown to work
    - Further development potential
    - Homogenous, selective heating demonstrated
  - Injection Moulding
    - The use of SRPs in injection moulding is proven
    - All variable parameters have been ascertained
    - Tooling requirements have been largely defined
    - Materials suitable for SRP injection moulding are available
  - Compression Moulding
    - The use of SRP pellets and sheets have been shown to be successful
    - Sheet moulding in conjunction with induction heating has been proven
    - SRP pellets have been dough-moulded and retained properties

WP 8 Project Management

- Task 8.1: Project Coordination

NetComposites were conscientious in the role of coordinating the project and communicating with the European Commission through the Project Officer and the Project Technical Assistant. The philosophy of having Quarterly General Meetings proved to be instrumental in keeping momentum, focus and communication in the project. Specific management challenges faced through the project were:

  - The take-over of FACT by Ticona.
  - The transfer of work to Comfil. Partner Comfil were able and willing to take over pultrusion work and ultimately did more work than Celstran would probably have been able to do. The transfer of budget was agreed at a special meeting at Celstran and was amicably managed.
  - The bankruptcy of Structoform. Late in the project Structoform went out of business having completed a large part of their work. Partner Promolding agreed by negotiation to take over the remaining work.

The consortium was well-motivated and active on the whole and was managed by means of meetings, teleconference calls, special technical meetings, regular minutes, project plans and a continuous action list. The latter generated 228 actions and was updated at least 16 times (generally after quarterly meetings). Seven NDA documents were created for suppliers of materials or end users, which are posted on the ESPRIT website. Communication to the Project Officer, Helge Wessel, and the Project Technical Assistant, Esther Barrutia, was continuous through the project and their assistance was greatly appreciated.

- Task 8.2: Intranet Development

The intranet part of the project website was actively used as a repository for information and for the management of the many samples generated by the project. All agendas, presentations and minutes of meetings are stored in the partner-only area of the website and are therefore available to the partners for as long as the website is live. Technical information is also stored for future reference such as data sheets, internal and external reports and Non-Disclosure Agreements relating to various external interactions.

Because so many samples were generated within the project it was agreed that a Sample Management system would be introduced. This is a database of 131 samples made by partners and it shows a summary table which can be entered into to show details for every sample such as what the constituents were, what additives were used, what process parameters were used, where it was sent and much more. This has proved to be a useful reference and traceability tool.

Potential Impact:

The baseline impact expectations as laid out in the original ESPRIT Annex 1 were to make a Self-Reinforced Polymer composite (SRP) material which has the ability to flow-mould therefore giving a30% reduction in plastics use by using plastic fibres, 30% lower component weight compared to existing plastics and higher recyclability. This basic metric was achieved with HTPET-LPET which exhibited modulus increase of 25-30% and HTPET-PBT which showed 20 – 80% improvement over the base matrix polymers. Since the reinforcement adds no additional weight the improvements in mechanical properties translate to weight reductions and material savings and improve significantly the recycling possibilities as there are no glass or carbon fibres to remove or to act a contaminent in ground polymers. LCP-PBT also showed some dramatic improvements which require some further verification but have great possibilities. It should also be noted that the material used as a 'model material', HTPET/PP, also showed notable improvements over the base matrix material with an impact strength improvement of 25 to 30%. Although not a true SRP this material was used by partners as an early trial for processing techniques as it has a bigger 'processing window' (the difference between matrix and reinforcement melt points) .

The concept of SRP composite materials by its very nature results in the need to process them in a tightly controlled window of temperatures between the matrix melt or softening point and the point at which the reinforcement fibres start to degrade. Therefore the most challenging process used to convert the pellets into product is injection moulding which necessitates, in standard machinery, polymers being forced at pressure through a heated barrel and screw, small apertures used to control the back pressure and small apertures in the tool. The ESPRIT project, in order to maximise commercial interest, concentrated on process control rather than new or modified machinery. Moulders will be reluctant to buy new machines or carry out extensive modifications in order to use a new material, whatever the benefits. The result is that with little or no modification to an injection machine SRP pellets can be processed, but extreme care must be taken with the parameters used and these are now available via ESPRIT partners and have been widely disseminated.

The potential impact is that injection moulders can shift to lighter SRP materials with minimal disruption. The main barrier is that parts will need to be re-tooled or, more likely, new parts can be designed with thinner walls and bigger gates to capitalise on the SRP technology. There will therefore be a significant lag as industry shifts to the new concepts.

The associated technology of dough moulding of SRPs should have a short route to commerciality when pellets are available in quantities. IVW were able to take the SRP pellets and send them through an extrusion machine to make a hot dough which was then compression moulded into a relatively complex, ribbed part. With a true direct deposition set-up a very efficient method of moulding SRPs could be envisaged. This would consist of something very much like current Direct-LFT technologies where a tool, press and extrusion machine would be positioned or moved such that extrudate would be dropped into tool straight from the nozzle and immediately compression moulded. The principal is proven, but requires a little further development for which there are no known plans but IVW could pursue this with further projects or Ticona and Comfil can communicate with potential customers, or in fact PEMU could develop it further, given demand.

The other significant impact will come from sheet materials developed in ESPRIT. By using a technique not previously investigated for the manufacture of SRP materials, namely powder impregnation by electrostatic means (D Preg). The technique allows for the use of any porous substrate be it woven, chopped, continuous or uni-directional fibres. This process offers a much-needed and more flexible alternative to the current hot compacted and co-extruded options. These current materials have aligned themselves as high-end, high-performance materials with a commensurate price tag which does not help the expansion into commodity products. Intermediate sheets, especially in PP/PP combinations, using the techniques developed in ESPRIT will, within a year or two, be competing for similar applications (luggage, sports goods, automotive) and opening the European market for more SRP usage. Partners Fibroline and Polisilk are continuing work started in ESPRIT as their own new partnership. An alternative sheet was made by partner PEMU by extruding the SRP pellets into continuous sheets. These were subsequently tested and found to be suitable for, as an example, a battery tray currently made with PP/glass materials. This is a low-cost method to make sheets materials and could be a serious competition for the very widely used GMT (Glass Mat Thermoplastics).

Further back down the production chain the project has provided a number of innovations. Comfil developed a commingling and pultrusion process which can make economical SRP pellets. The line is small-scale and will require further investment to achieve volume manufacture, but Comfil were actively marketing the srPET products before the project finished. AIMPLAS dramatically improved the dispersion of CNTs in PP and other polymers by carefully controlling the machinery and process parameters. This high quality dispersion facilitates selective heating by microwave or induction and spinning of fibres with CNT additives included, not previously possible. With materials compounded by AIMPLAS (or PEMU), Comfil are able to spin fibres with CNT additives leading to the possibility of commingled yarns with microwave or induction susceptors for selective heating. It also opens possibilities of conductive polymers and mechanical improvements. The up-scaling work carried out by AIMPLAS and PEMU was critical to take the high quality compounding from lab to factory and also to thoroughly validate the software analysis provided by the LUDOVIC programme facilitating the up-scaling to many different industrial compounding machines. AIMPLAS have already successfully used the compounding knowledge gained to produce high quality expanded graphite and grapheme compounds for the Nanomaster project (285718) which aims to develop techniques for mass-production and use of graphene-based materials.

Further down the technology readiness scale are the selective heating methods using microwave and induction heating. IVW investigated induction susceptors, selected the best and with AIMPLAS' assistance manufactured induction heatable sheets. The heating machinery was likewise optimised and controlled induction heating demonstrated. This was particularly impressive as to heat, hold and cool a sheet material in a controlled profile is not easy with any technique, let alone a new one. This requires further development and a proposal (FutureFlex) has been submitted with NetComposites and AIMPLAS to further refine this technology as part of a new variable consolidation concept.

NetComposites, using machinery supplied by F and M, developed a novel heating method using CNT additives in matrix polymers heated by microwaves (MW). The large problem of 'hotspots' (areas where the polymer heats, softens and therefore heats up even more, leading to thermal runaway) was overcome by the high quality additive dispersion from AIMPLAS and the development of a method to manipulate microwaves to give an even energy distribution. The manipulated MW concept works but needs further investment and development. Patent routes were investigated by NetComposites but there were a number of competing patents, not necessarily polymer-centred, which suggested more targeted development will be required to have a technology which can live by its novel approach or by being patented in a more specific way. For example the control system/method can be further developed.

A technology which can be used in several of the aforementioned processing methods is the Regloplas dual channel heating system. They have developed their current machines alongside new valve and control systems to make a dual-channel heating system with an energy battery which shunts hot or cold liquids into a holding reservoir thereby increasing energy efficiency. This system was developed throughout the project, tested by partners and is now in its early production phase with several commercial customers taking systems for their current production. These refined machines are attractive to many thermoplastic moulders who need to control tool temperatures to achieve surface finishes with conventional process but also have potential to give users of SRP materials the desired quick cycle times to heat, hold and cool a tool. The technology is already accepted into the marketplace and NetComposites have included Regloplas in a further development project proposal, FutureFlex, which aims to use the system for a novel variable consolidation process.

In summary, it can be seen that there a number of strands of technology arising from ESPRIT which are in the marketplace or will be there very shortly. Some of the developments need further work to achieve commercial readiness and plans are in hand to take them further. Overall the developments have the potential to reduce material usage, make lighter or impact-resistant products or open new market possibilities, all of which is beneficial to the European plastics industry.

Through the lifetime of the project a considerable effort has been made to engage with the world outside Research Projects, documented in full elsewhere in this report. Exhibitions and seminars have been attended, presentations made, papers written, press releases made, exploitation strategy seminars have been held, websites created and so on. For the ESPRIT project a final summary called 'Technology Overview' was created as a 12 page brochure which resides on the ESPRIT website as a downloadable document and, of course, is held by all ESPRIT partners for their own use. All of this effort is targeted towards successful exploitation.

The partners in the project now have a network of contacts which, if needed, can assist them in commercialising the ESPRIT outputs and a number of the innovations from the project are already in process of becoming commercial realities.

List of Websites:

http://www.espritproject.eu

<u>Contact:</u>

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