



**PROJECT NO: FP6-516258-1**

**CONTRACT NO: COLL-CT-2005-516258**

## **DYNAMIC**

# **The Closed Loop, Active Control of Reaction Dynamics, Through Dielectric Monitoring, Enabling Greater Competitiveness of The European SME Plastics Processing Community**

## **42 Month Activity Report – Final**

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Lead Contractor: Composites UK Ltd (CUKL)	
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SME Contractor: Integrated Aerospace Sciences Corporation (INASCO)	
SME Contractor: Isojet Equipments (Isojet)	
SME Contractor: Astromal Sp. zoo (Astromal)	
SME Contractor: Technika Plastika SA (Techplas)	
SME Contractor: Excel Composites	
SME Contractor: Nanoforce Technology (Nano)	
RTD Performer: Pera Innovation Ltd (Pera)	
RTD Performer: Universite Claude Bernard Lyon 1 (UCBL)	
RTD Performer: National Technical University of Athens (NTUA)	

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## CONTENTS

<b>EXECUTIVE SUMMARY .....</b>	<b>3</b>
<b>PROJECT OBJECTIVES .....</b>	<b>4</b>
1.1. Overview of General Project Objective.....	4
1.1.1. Technological Objectives.....	4
1.1.2. Secondary Goals of the Project: .....	4
1.1.3. Resulting Economic Targets to Improve Competitiveness .....	6
1.1.4. Resulting Societal and Policy Impact Targets .....	6
1.1.5. Enabling Innovation-Related Objectives .....	6
<b>2. WORK PACKAGE PROGRESS REVIEW .....</b>	<b>7</b>
2.1. Work Package objectives .....	7
2.2. Overview of Work Package Technical Progress.....	7
2.2.1. Work Package 1 – Enhance Scientific and Technological Understanding .....	7
2.2.2. Work Package 2 – Material Models.....	13
2.2.3. Work Package 3 – Development of Process Monitoring.....	14
2.2.4. Work Package 4 – Development of Process Control.....	17
2.2.5. Work package 5 - Integration .....	24
2.2.6. Work package 6 – Pilot Plant Set Up .....	25
2.3. Deviation from the Plan and Corrective Actions .....	30
2.4. Work Package Deliverables Update.....	31
2.5. Work Package Milestones Update .....	37
<b>3.0 CONSORTIUM MANAGEMENT .....</b>	<b>38</b>
3.1 Consortium Status Overview .....	38
3.2 Project Timetable & Status .....	39
3.3 Meetings and Communication.....	41
<b>4.0 DISSEMINATION ACTIVITIES .....</b>	<b>42</b>
4.1 Management of Knowledge and IPR.....	42
4.2 Plans for Using & Disseminating Knowledge .....	42
4.3 Exploitation & Dissemination Activities Undertaken.....	43
<b>5 OTHER ISSUES .....</b>	<b>46</b>
5.2 Conclusions.....	46

## EXECUTIVE SUMMARY

This report covers the work carried out during the entire project, namely periods 1, 2 and 3. The main body of this report is a precise overview of the work and results undertaken by the consortium. Deliverable reports cover each of the work tasks in more detail.

All the technical elements have been completed successfully culminating in the production of a Dielectric monitoring system consisting of **a)** six fully fabricated prototype Dielectric sensors for use up to 300deg Celsius, **b)** Dielectric monitoring system for multiple sensors with optimal monitoring strategy, **c)** fully assembled heating and cooling system.

The project was extended in order that the consortium could overcome difficulties with sensor fabrication which have proven to be the most difficult scientific and technical barriers to resolve. These related to the plating and polishing regimes for the sensor. Trials of the Dynamic system have been carried out by the consortium in three locations using Injection moulding and Pultrusion equipment.

The consortium worked efficiently in order that the three main separate elements of the project be integrated in to a working system capable of being retro-fitted into production plant and equipment. In order that thorough scientific and technical advances could be achieved, two materials were chosen for use with the Dynamic system though the sensor is capable of monitoring more materials than those trialled.

Materials modelling and characterisation was undertaken by UCBL. This included thermal and rheological characterisation and dielectric mapping between the material properties and the dielectric signal.

The research undertaken by UCBL enabled NTUA to develop Process Monitoring Techniques achieved through process modelling, development of control strategies and algorithm's. As NTUA developed the monitoring techniques close links were maintained with UCBL in order to verify accuracy of the data. The high quality of the work undertaken in this area resulted in a set of control parameters for the real time dielectric monitoring of thermoplastic processing and as a result trials with the control system proved the accuracy of this data. NTUA developed a control system capable of monitoring the dielectric response.

The work done by UCBL and NTUA was further supported by Inasco who took responsibility for integration of the software implementation and interface with the dielectric sensor signals to create the dielectric monitoring and control system. This package of work meant that Inasco maintained close links with each of the consortium members in order to build effective systems for use in trials and pilot plant studies.

On completion of the system and pilot plant trials, studies were undertaken at end-user plants covering Injection Moulding and Pultrusion techniques. The trials proved that the Dynamic system is fully capable for integration into existing systems. The Lead-Partner, CUKL have disseminated the Dynamic project throughout the full 36 Month period using various media forms and through stands at major industry exhibitions. EurExcel have complimented these activities by delivering some further dissemination and more specifically training events for interested parties.

In summary, the technical work is considered to be successful and of value to all of the consortium and the thermoplastic processing industry as a whole, the SME consortium members are thus keen to exploit the technology further.

# PROJECT OBJECTIVES

## 1.1. Overview of General Project Objective

The main project aim is to overcome the slow cycle times within the thermoplastic processes by initiating, controlling and partially achieving the polymerisation reaction within the injection barrel instead of in the mould after injection.

### 1.1.1. Technological Objectives:

Produce a prototype material-state process control system able to:

- Control the temperature in a barrel of 80mm diameter and 1000mm long, containing 5kg of material to an accuracy of control +/- 2 deg C /m and affect a +5 deg C or -5 deg C change in volume within 5 seconds.
- Achieve 60% of the polymerisation process in a multiple charge barrel (typically accommodating 3 to 5 shots for the mould) within 5 minutes, enabling a 60% reduction in cycle time.
- Be installed onto a standard RIM machine for a cost of less than €20,000.
- Generate typical manufacturing cost savings of 30% on components made of Nylon material and typically 0.1M volume.

Overcome the following technical obstacles and current technology limitations related to:

- The limitations of the current interdigital dielectric sensors developed for thermoset processes, in regard to their ability to detect permittivity and dielectric loss accurately over a large material areas without distortion at temperatures and pressures greater than those found in thermoset processing and specifically at temperature up to 350°C and 20 bar, by developing a dielectric, interdigital electrode, sensor fabricated using ceramic material and utilising a novel high temperature micro welding method of signal connection, that will allow it to withstand the higher temperatures and pressures experienced in thermoplastic processing.
- The limited amount of data available relating to the polymerisation reaction progress in micro-scale within thermoplastic materials, which has prevented the creation of materials state models to be able to infer degree of polymerisation and crystallinity from permittivity and dielectric loss, by exploiting the recent advances in modulated calorimetry and on-line DSC.

### 1.1.2. Secondary Goals of the Project: Economic and Societal Impacts and Innovation Related Objectives to Enable Exploitation

In order to achieve the societal and economic objectives, an enabling set of innovation related objectives has been defined:

- *To protect the IPR*, The results will be compiled into a protectable form for a patent application, carrying out patent searches to assess the viability of a patent application in relation to the achieved results. In particular, at this stage a number of potentially patentable aspects of the project is foreseen:
  - Durable dielectric high temperature sensor
  - Development of an on-line calorimeter for the evaluation of the thermoplastic material properties
  - Intelligent guidance and control of the manufacturing process through the use of a model to infer polymerisation and crystallinity from dielectric permittivity and dielectric constant.
  
- *To enable absorption of the results by the participants*. Each participating SME comes from a specific technological background to form a link in the supply chain. The RTD performers will provide a case study to facilitate specific knowledge transfer relevant to the SME's, and therefore to enable the rapid application and embedding of the technology within the SME's core business, so they gain rapid technological and commercial benefit. The following specific case studies will be created as a result of the project;
  - Rapid manufacture of complex and technically challenging un-reinforced parts
  - Rapid manufacture of complex reinforced moulding' with varying wall thickness

The provision of the individual case studies will be followed by two full project consortium events and one-off individual (SME) company knowledge transfer event and secondments from each SME partner within one of the R&D performers' research team for a period of one week.

- *Dissemination of the knowledge beyond the consortium*. The potential industrial user communities, in particular the SME's in the polymer composites sector, will receive the benefits and principles of the new technology. The three R&D performers will facilitate these activities through two dissemination events held at each of their premises, the three SME Core group proposer's undertaking the pilot plant trials will host two demonstration events also. The national IAG's, (CUKL) will hold a dissemination event and the national AIG member organisations of the European IAG. EurExcel will hold their own event to further disseminate the knowledge to a wide as population as possible. For each of the case studies above a non-confidential summary case study document will be created. This will be used to contact around 5,000 firms across the EU. Presentations will be given at 4 major international conferences with attendances greater than 500 and attendance will be arranged to poster sessions at 6 other conferences using the non-confidential case studies. Two key aerospace conferences/events will be targeted to promote the key benefits of the new RIM processing technology, and it's potential to enable the use of a wider range (than the two case study materials) in applications in aerospace that are normally associated with more expensive and longer cycle time manufacturing techniques than the *Rapid RIM* process.
  
- *Validation of the Technology*. Each of the identified case study areas represents a specific industry sector. Around 10 leading companies from each of the sectors will be identified and targeted. The results of work will be compiled to enable these 20 companies to assess in detail the performance of the control, monitoring and injection system developed and also to validate the end user benefits and potential cost savings using this technology.

### **1.1.3. Resulting Economic Targets to Improve Competitiveness**

We have undertaken an extensive economic study of the impact our technology will have on the polymer composites sector as a whole and we have established a number of economic targets;

- We estimate that we can displace €100m p.a. of imports safeguarding 800 jobs
- We estimate that the domestic sales in Europe alone will be worth €104m, creating 300 jobs and safeguarding 540 jobs
- We estimate that we will sell manufacturing systems to the value of €4m p.a. creating 40 jobs
- We estimate that the cost savings in the manufacturing of polymer parts using our new technology may generate savings of €42m.

### **1.1.4. Resulting Societal and Policy Impact Targets**

- *Workplace Health and Safety*; The increased use of thermoplastics rather than thermoset resin systems improves the workplace for our citizens for a number of reasons;
  - No hardeners or catalysts are required to cure the resin, a majority of these are regarded as harmful if proper care is not taken, this will reduce dermatitis and the risk to occupational asthma
  - No loose glass fibres which cause irritations and no contact with wet resins such as epoxies or polyesters which can cause sensitisation in the workers leading to skin disorders and occupational asthma
  - Reductions in styrene emissions as the process is thermoplastic based and styrene is not required.

### **1.1.5. Enabling Innovation-Related Objectives**

The dissemination of the results amongst the SME's, represented in the projects by their industrial associations/groupings is an important element we have planned significant dissemination activities from the very start of the project and have included special training and demonstration ("take-up") actions.

## 2. WORK PACKAGE PROGRESS REVIEW

### 2.1. Work Package objectives

The specific work package objectives for the project are summarised in the table below. The project was split into three phases:

**Phase 1** - Development of new scientific knowledge and technology.

**Phase 2** – Integration and pilot plant.

**Phase 3** – Dissemination and training.

WP No.	Work-package Title	Lead Contractor Short Name	Person Months	Start Month	End Month	Deliverable No.
1	Hardware development	Pera	40.0	1	12	1 - 5
2	Materials Model	UCBL	24.8	1	12	6 - 9
3	Development of Process Monitoring	NTUA	12.8	1	12	10 - 14
4	Development of Process Control	NTUA	29.2	1	15	15 - 19
5	Integration	Inasco	22.5	12	18	20 - 22
6	Pilot Plant Set Up	TechPlas	33.1	18	30	23 -26
7	Innovation and Dissemination	CUKL	22.9	24	42	27 - 31
8	Training	EurExcel	28.7	24	42	32 -33
9	Consortium Management	CUKL	6.5	1	42	
10	Project Management	CUKL	8.7	1	42	

Table 1: Work package plan

### 2.2. Overview of Work Package Technical Progress

#### 2.2.1. Work Package 1 – Enhance Scientific and Technological Understanding

## Task 1.1 Specification for the Hardware

Task Leader: Inasco

### Objectives:

Define the physical, electrical, mechanical and cost characteristics of the dielectric sensor and heating / cooling system taking into account the fact that the sensor and heating / cooling system will be mounted to moulds and processing tools.

### Progress: This task is Complete

The specification of the hardware has been completed and uploaded to the EC.

The specification was derived from two aspects, firstly, a definition of the physical envelope as perceived by the end users and secondly, a definition of signal limitations.

The above aspects were defined for the sensor, test rig and end user equipment.

## Task 1.2 Sensor Design – Materials and Geometry

Task Leader: Inasco

### Objectives:

Definition of the geometrical and operational characteristics of the dielectric sensor in terms of dimensions, materials and grid layout.

### Progress: This task is complete

The design encompasses the material selection of the sensor substrate based upon the processing parameters as well as ease of machining, electrical response of the sensor using a capacitance estimator tool to determine the geometrical aspects of the sensor grid.

The sensor design was optimised for reliable electrical connections to the monitoring equipment and also in respect to the sensor housing for adaptation to process tooling.

### Final sensor design:

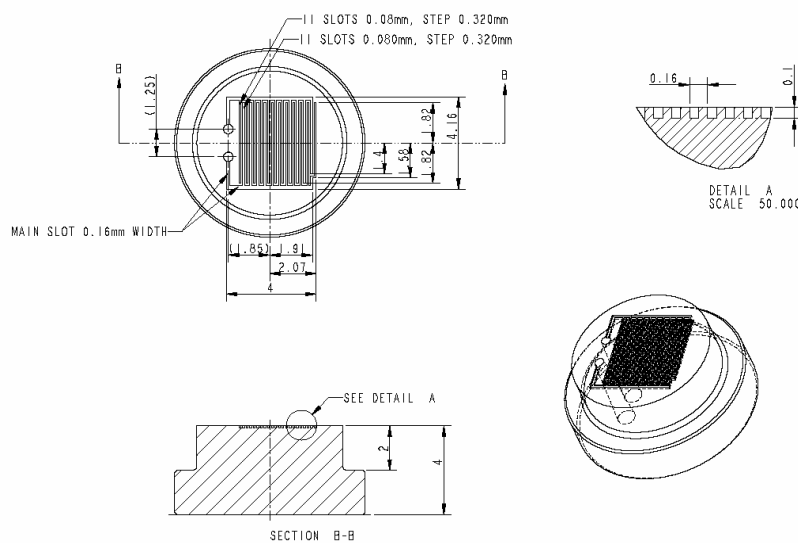


Figure 1: Final sensor design



### **Task 1.3 Heater / Cooler Design**

**Task Leader: Pera**

**Objectives:**

Design of the Heating / Cooling System

**Progress: This task is Complete.**

It was originally intended to use heater mats but this design had to be modified following calculations that such a design could not match the specifications for the heating and cooling regimes required for the target materials. A new design was adopted which utilises cartridge heaters and water cooled channels.

A test rig has been successfully designed and implemented for the project.

### **Task 1.4 Hardware Fabrication**

**Task Leader: Pera**

**Objectives:**

Prepare prototype dielectric sensors with a fabrication method selected on the basis of design issues and economical aspects. Samples with simple grid will be initially manufactured. Based on the feedback from the testing of these simple geometry sensors, the final prototype dielectric sensor will be produced.

**Progress: This task is Complete.**

Several prototype sensors are available to the consortium. Reproducibility of the sensors proved to be highly technically challenging so Nanoforce were recruited into the consortium to overcome this issue, and develop increased wear resistance and thermal limits in sensor systems. NiP Alloy manufacturing methods have been improved by LZH, a subcontractor to Inasco.

### **Task 1.5 Hardware Testing**

**Task Leader: Pera**

**Objectives:**

Test sensor samples on standard liquids and representative materials under simulated process environment. Obtain early indication of sensor performance to fine tune the sensor and obtain verification of the dielectric response.

**Progress: This task is Complete.**

A lab scale test bench or 'test rig' was built for the purpose of conducting this study. The test rig encompassed not only the sensor but all of the Dynamic control systems and trials were undertaken to fully assess the capability of the sensor. These results can be seen in Deliverables D5 and D7 which is a combined report.

The dielectric sensor is proven to be fully capable.

## **Task 1.6 Development of Sensor Mounting**

**Task Leader: Pera**

### **Objectives:**

Design and fabrication of sensor mounts for adaptation to moulds and process tooling. Provision for robust mounting and easy replacement of the sensor element within the sensor mounting with consideration of cost optimisation.

### **Progress: This task is Complete.**

Sensor mounts have been designed, fabricated and successfully trialled during Task 1.5 (Hardware testing). The mounts are fabricated and supplied with each sensor as an assembled item.

## **Task 1.7 Assessment of Current Specification & F.A.S.T Compatibility**

**Task Leader: Nanoforce**

### **Objectives:**

To confirm that F.A.S.T technology, materials and processes is suitable for the production of a Dielectric Sensor.

### **Progress: This task is Complete.**

An assessment of meta-stable conductive ceramic phases (2D) using rapid sintering techniques has been made. It is based on a multilayered ceramic structure consisting of two types of laminates, (Al<sub>2</sub>O<sub>3</sub> and TiN). These base materials are then stacked one on top of the other in an alternating array as shown thus:

## Task 1.8 Multilayer Sensor Grid Creation

**Task Leader: Nanoforce**

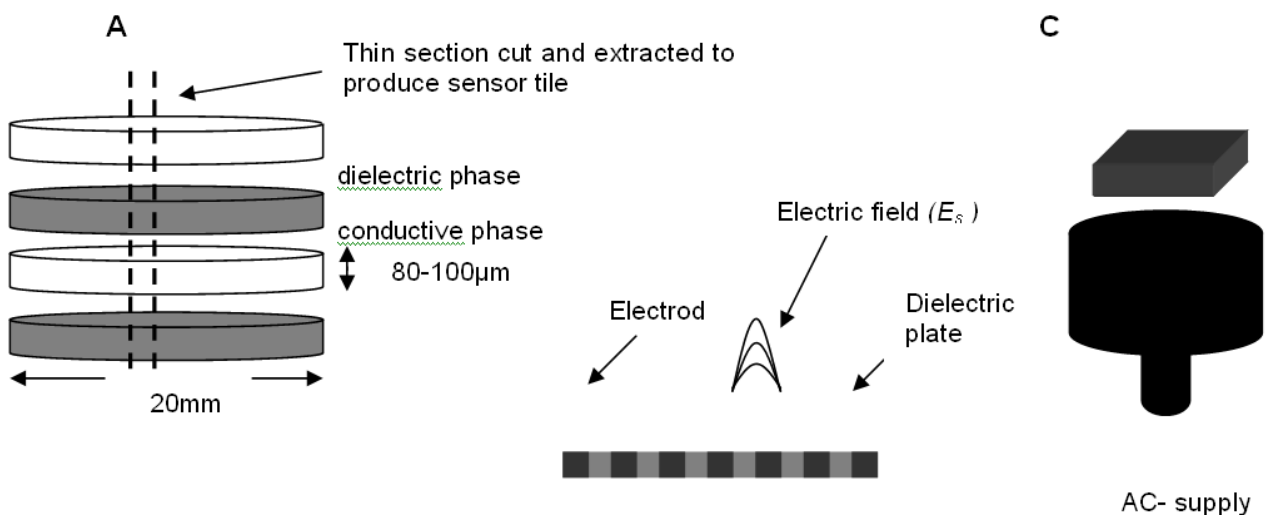
### **Objectives:**

Produce a multilayer grid pattern design suited to F.A.S.T technology and Dynamic sensor design to facilitate completion of Work packages 3 & 4.

### **Progress: This task is Complete.**

Nanoforce researched various methods of multilayer grid design and produced a design utilising a powder sintering method. This method uses a 4 stage process and is envisaged that once fully proven production costs will be significantly reduced over current state of the art processes.

A picture of a multilayer grid produced by this technique is shown in Task 1.10.



**Figure 2: Multilayer sensor grid concept for F.A.S.T.**

## Task 1.9 Tooling and Construction

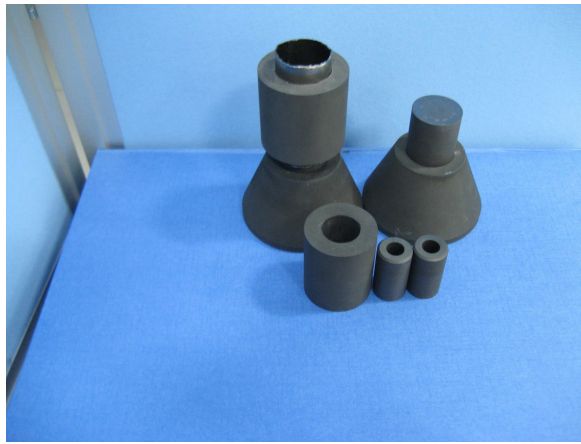
**Task Leader: Nanoforce**

### **Objectives:**

Develop construction methods for unstructured ceramic pucks using F.A.S.T methodologies and to benchmark / optimise apparatus and production methods to produce / incorporate 2D multilayer ceramic grids and 3D puck bodies in order to facilitate completion of Work Packages 3 & 4.

### **Progress: This task is complete.**

An assessment of die design was undertaken between Nanoforce and Pera. CAD drawings were produced and die sets were manufactured by Pera.



**Figure 3: 30mm Die Set for F.A.S.T.**

### **Task 1.10 Design Optimisation**

**Task Leader: Nanoforce**

**Objectives:**

To optimise sensors that use rapid sintering and functional ceramic studies

**Progress: This task is Complete.**

An all ceramic fully sintered capacitive grid (non interdigitated) was produced using a F.A.S.T methodologies (Field Assisted Sintering Technique). An example of the grid is shown thus:

**All ceramic, fully sintered capacitive grid**



Consists of multiple layers:

- a) Conductive TiN
- b) Dielectric alumina

**Figure 4: All Ceramic Multilayer grid**

### **Task 1.11 Production Assessment**

**Task Leader: Nanoforce**

**Objectives:**

To make an assessment for scaling up experimental methods for production of 100, 1000 and 10,000 sensors per year.

**Progress: This task is complete.**

A full assessment has been undertaken of likely production costs. It is noted that F.A.S.T technology has considerable potential for reducing costs over current state of the art techniques. This is detailed in Deliverable 1.11

## 2.2.2. Work Package 2 – Material Models

### Task 2.1 Material Selection

**Task Leader: Astromal**

**Objectives:**

Studies of reactive systems with different reactivities and polymers, with various time, temperature and pressure cycles.

**Progress: This task is Complete.**

Materials have been selected: CBT160 (one-component pellets) and NyRIM.

### Task 2.2 Main Material Characterisation (Thermal/Rheological)

**Task Leader: UCBL**

**Objectives:**

Knowledge of the reaction kinetics and build-up of the physical and mechanical properties.

**Progress: This task is complete.**

The materials (CBT – a Cyclic polyester based material and NyRIM, a caprolactam based material that polymerises to form Nylon 6) have been analysed anisothermally and isothermally by the use of optical microscopy and TGA to identify the influence of water. Isothermal characterisations were used to obtain molecular weight estimations as well as characterisations such as DSC, rheological and dielectric measurements and torque measurements. This is detailed in deliverable 10

### Task 2.3 Dielectric Mapping (Assignment between material properties and dielectric signal)

**Task Leader: UCBL**

**Objectives:**

Dielectric studies to assign the target material properties and control the process cycle. In-situ analysis of the dielectric signal for bulk or dilute processing (conductivity, relaxation time, permittivity). Influence of the formulation and fillers on this response. Multi-detection to improve the intelligence of the manufacturing process.

**Progress: This task is complete.**

Dielectric signal has been recorded using the mounted sensor and Micromet equipment. In addition tests have been undertaken with the PXI. The characterisation of the obtained materials (c-PBT) and PA6) has also been performed by thermal and mechanical analysis in order to correlate the dielectric sensing investigations with the material properties. This work is detailed in Deliverable 11

### **Task 2.4: Health monitoring:**

**Task Leader: UCBL**

**Objectives:**

Dielectric studies to assess the capability of Dielectric systems to analyse and identify changes in materials over time: raw materials stored under less than ideal conditions can alter their material properties significantly, the objective of this task is to assess if dielectric signals can be used to identify these material changes over a 3 month period

**Progress: This task is Complete.**

For samples polymerized at lower temperatures (~190 °C), the mechanical properties are similar to those obtained without aging. Samples polymerized at higher temperatures are more ductile after aging, this can be related to some r-crystallization . chain rearrangement occurring at 200 °C. Dielectric signals can determine changes in polymerisation/cure time as a result of ageing effects. Water absorption can also be inferred from sensor signals (as shown in deliverable 12).

### 2.2.3. Work Package 3 – Development of Process Monitoring

Task 3.1: Signal Analysis, Pressure Effects, Signal Processing

**Task Leader: NTUA**

**Objectives:**

Analysis of dielectric signal treatment in order to adapt to the specific conditions for the thermoplastic processing routes.

**Progress: This task is complete.**

Each of the main components of the system has been developed separately, the hardware, the control software and the sensors and the interdependencies of the monitoring operation has been considered. Correlation of dielectric signals ( $Z''_{MAX}$ ) to material properties has been considered as detailed in deliverable 13

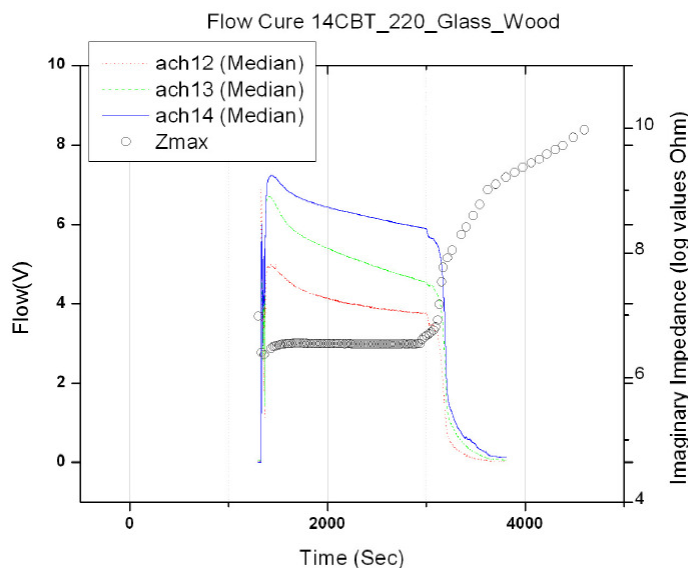


Figure 8. Correspondence of the logarithm of imaginary impedance max (circles at the right axis) and the voltage of the three flow sensors during the cure of a typical in-situ CBT resin with glass fibres.

**Figure 5: Correspondence between Z"MAX and Voltage during cure cycle of reinforced CBT**

**Task 3.2: Monitoring Strategy**

**Task Leader: Inasco**

**Objectives:**

Determination of monitoring strategy in terms of objectives, tasks, succession, interactions and allowable limits (alarms). Customisation of monitoring strategy for NyRIM and thermoplastic RTM. Upgrade of the monitoring system with the monitoring strategy for the processing routes.

**Progress: This task is complete.**

A detailed analysis of the dielectric signal has been undertaken in order to adapt to the specific conditions for thermoplastic processing routes. The work covers signal analysis methodology for optimisation of the speed and accuracy of the dielectric response and measurement of flow and

cure. Analysis is also undertaken of the pressure and compaction effects on dielectric properties of thermoplastic polymers with the inclusion of reinforcing fibres on the dielectric signal.

### Monitoring Strategies and Control Perspective

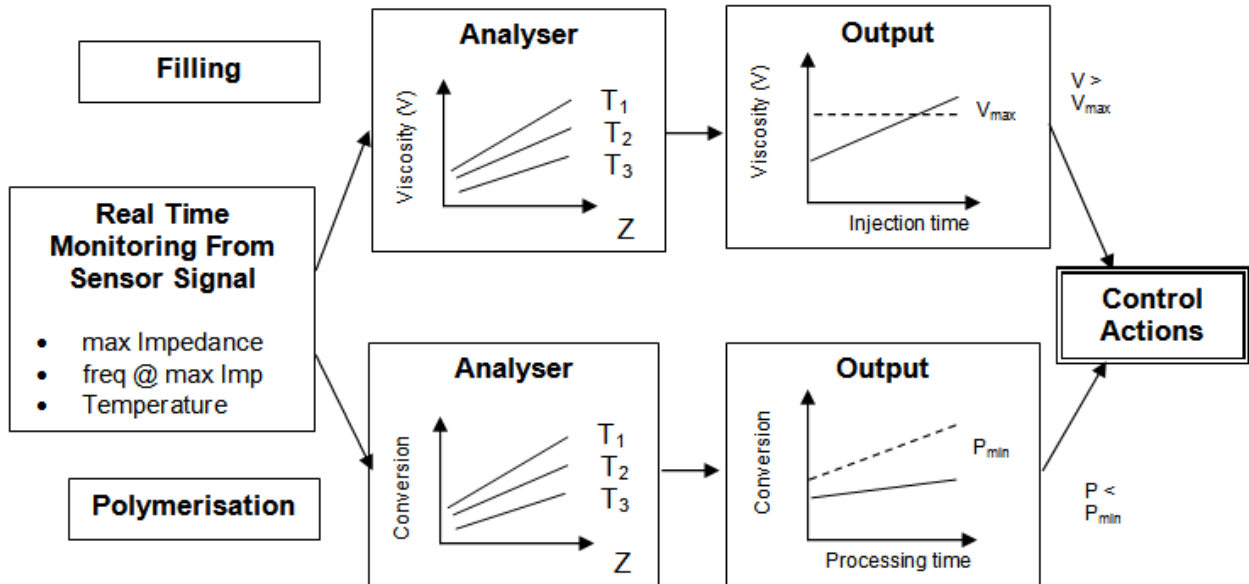


Figure 6: Monitoring Strategies and Control Perspective

This work is detailed in deliverables 13, 14 and 15

### Task 3.3: Data Collection and Data Processing

Task Leader: ISOJET

#### Objectives:

Definition, design and implementation of data analysis routine for accurate derivation of material state and optimal performance of monitoring tasks.

#### Progress: This task is complete.

The main aspects covered by this work are *a)* the design of the experimental set-up facility, *b)* the description of the methodology to obtain measurements using dielectric monitoring and *b)* the methodology towards establishing a material state index for reactive thermoplastics.

### Task 3.4: Prototype multiplexer and integration to dielectric monitoring system

Task Leader: ISOJET

#### **Objectives:**

Definition, design and implementation of data analysis routine for accurate derivation of material state and optimal performance of monitoring tasks.

#### **Progress: This task is complete.**

Work has been successfully completed in developing the multiplexing capability in monitoring the thermoplastic reactive moulding. Measurement capability has been established over multiple sensing points. This work is detailed in deliverable 16.



Figure 7: National Instruments PXI and Dynamic system user interface

### Task 3.5: Guidelines for selecting dielectric measurements parameters

Task Leader: NTUA

#### **Objectives:**

Definition of measurement parameters and their relationship to measurement accuracy for real-time dielectric monitoring of thermoplastics processing.

#### **Progress: This task is complete.**

The standardisation of dielectric measurements for thermoplastic reactive moulding have been established by designing an acceptable frequency range that will move during the reactive processing. This ensures high quality and standardised data is attained in the required time. The selection criteria for signal monitoring have been detailed in deliverable 17



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the interpolation product will be:

$$x(\xi_i) = a_1^0 \cdot x(\xi_1) + a_2^0 \cdot x(\xi_n) = (1 - \xi_i) \cdot x(\xi = 0) + \xi_i \cdot x(\xi = 1) \quad (1)$$

where  $x(\xi=0) = x_0$  and  $x(\xi=1) = x_n$ .

This will return a linear distribution, of n frequencies i.e  $\log(x(\xi_i))$ . The system will then generate those frequencies for the next scan.

#### Flow diagram

The steps of the monitor strategy algorithm are:

- 1) Update values  $f_n$ ,  $f(n-1)$ ,  $f(n-2)$  and  $s$ .
- 2) Use the norm expression (e) for  $f_p$  and  $f_n$ . □
- 3) If  $s > 2$  compute the new  $f_p$  by using backward differences and update the value  $f_p$ .
- 4) If  $(e) < 1$  then  $f_{next} = f_p$ , else  $f_{next} = f_n$ . □

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Confidential

- 5) Construct the k cases.
- 6) Find the first case where  $f_{next} > tri$ .
- 7) Do a linear interpolation for the case  $i$  □

Figure 8: Dielectric signal selection criteria

## 2.2.4. Work Package 4 – Development of Process Control

### Task 4.1: Process Variability

Task Leader: Pera

#### Objectives:

To study and define the processing window and margins of pilot processes and materials.

#### Progress: This task is complete.

The consortium have carried out a study of process variability in order to determine the processing variables of the chosen materials and pilot processes. This has been detailed in Deliverable 18

## Task 4.2: Control Strategies

Task Leader: NTUA

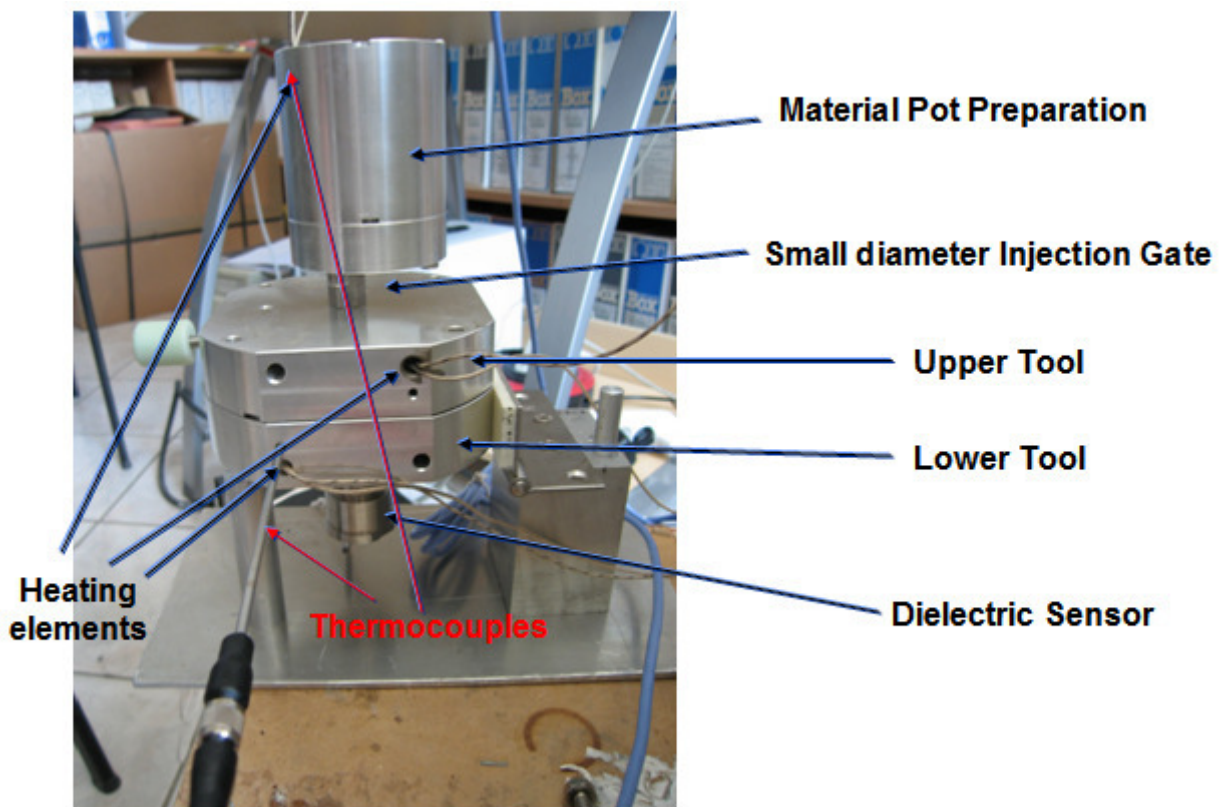
### Objectives:

To study and select the most appropriate control strategies according to process and material requirements.

### Progress: This task is complete.

Work has been undertaken to select the control strategies and criteria. This was achieved by developing process simulators capable of providing information regarding the behaviour of reactive thermoplastic materials during processing as well as what the information the dielectric sensors deliver during the cycle. Numerous trials have been undertaken to determine control scenarios for temperature and moisture etc, as detailed in deliverable 19.

### Advanced Simulator:



### Task 4.3: Process Modelling

Task Leader: NTUA

#### Objectives:

To develop a fast and accurate process simulation tool.

#### Progress: This task is complete.

The process simulation has been developed with three primary equations as detailed in Deliverable 20

The 7 parameter polymerisation model

2. a 7-parameters autocatalytic model:

$$\frac{d\beta}{dt} = P_3 e^{\left(\frac{P_1}{R(T+273)}\right)} (1-\beta)^{P_5} + P_4 e^{\left(\frac{P_2}{R(T+273)}\right)} \beta^{P_6} (1-\beta)^{P_7} \quad [3]$$

Figure 9: 7 parameter CBT polymerisation model

$$P_1 = -10618.669020$$

$$P_2 = -10240.378834$$

$$P_3 = 634271096.226533$$

$$P_4 = 450613325.359270$$

$$P_5 = 0.961426$$

$$P_6 = 0.383520$$

$$P_7 = 0.877519$$

with a modelling error of 2.5%.

a five parameter viscosity model

$$\eta = P_1 \exp(P_2 T + (P_3 + P_4 T)\beta) \quad [7]$$

Where from the parameter identification tool the following parameters were identified

$$P_1 = 0.000494$$

$$P_2 = 0.012355$$

$$P_3 = 13.882390$$

$$P_4 = 0.004221$$

$$P_5 = 0.052435$$

Its good accuracy can be seen in fig. 10 with respect to data taken from the literature [10].

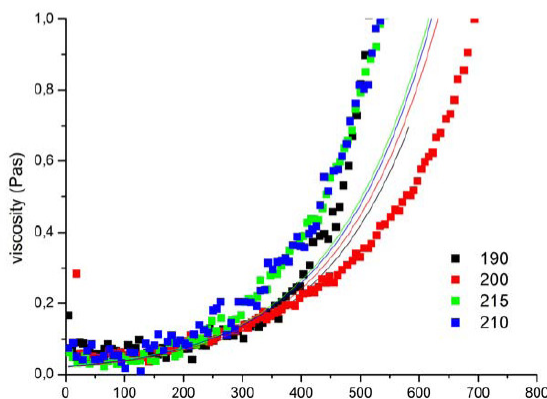


Figure 10: 5 parameter CBT viscosity model

And a crystallisation model based on the polymerisation model (as both polymerisation and crystallisation happen simultaneously in the cPBT system)

However, because in the present study we are dealing with a crystallization process that is related more to the polymerisation process and not on the cooling of the polymer, we are going to use a similar formula to the one used to model the polymerisation process and more specifically the 7-parameters autocatalytic model:

$$\frac{d\chi}{dt} = P_3 e^{\left(\frac{P_1}{R(T+273)}\right)} (\chi_{\max} - \chi)^{P_5} + P_4 e^{\left(\frac{P_2}{R(T+273)}\right)} \chi^{P_6} (\chi_{\max} - \chi)^{P_7} \quad [8]$$

Where  $\chi_{\max}$  is the maximum level of crystallinity reached during the polymerisation process but not during cooling.

#### Figure 11: 7 Parameter CBT crystallisation model

$$P_1 = -10618.669020$$

$$P_2 = -10240.378834$$

$$P_3 = 634271096.226533$$

$$P_4 = 450613325.359270$$

$$P_5 = 0.961426$$

$$P_6 = 0.383520$$

$$P_7 = 0.877519$$

#### Task 4.4: Numerical optimisation of the pilot processes and materials

Task Leader: NTUA

##### Objectives:

Optimisation of the process model.

##### Progress: This task is complete.

The development of algorithms and the corresponding tools towards the process modelling of the in-situ polymerisation of reactive thermoplastic materials.

Neural networks and genetic-based optimisation tools have been used to identify the model parameters, two of which are the main process variables that directly affect the process namely viscosity and polymer fraction. Crystallisation is also considered. These three process variables have been modelled with good accuracy for CBT (case 5 detailed in deliverable 21) for which the principle was proven while the whole procedure can be applied to similar reactive materials such as AP A6 (NyRim) and PA12. Furthermore processing time has also been optimised.

## Results

In order to test and check the performance of the developed optimisation algorithms several representative cases have been designed as indicated in Table 1 below.

Case	1 <sup>1</sup>	2	3	4	5 <sup>2</sup>
w(Tmax)	5	5	5	5	5
w(a)	100	100	100	100	100
w(t)	0.01	0.01	0.01	0.1	0.1
a max (%)	98	98	100	100	100
Tmax (°C)	215	220	220	220	220
dTmax (°C/min)	10	10	10	10	5
dTmin (°C/min)	-20	-20	-20	-20	-20

<sup>1</sup> first stage is dynamic from 180 to 195°C in 5 minutes

<sup>2</sup> first stage is isothermal at 190°C for 3 minutes

Table 1. Representative test cases with corresponding weighting functions, maximum temperature and target polymerisation fraction as well as maximum allowed heating and cooling rates.

In all cases the initial and the final temperatures were kept constant as they refer more to the minimum melting temperature (the first) and the maximum allowable demoulding time (final temperature). Starting from case 1 we can see in fig. 2 that as the first stage is kept constant the rest cycle is still faster than the initial cycle. However if we don't maintain the first stage and allow for a fully optimal regulation of the profile based only in the same constraints as case 1 we can observe in fig. 2 for the case 2 profile that the first two stages have actually become one heating stage at the maximum allowed heating rate so this cycle is considerably faster than cycle 1. However if the part is large it is questionable whether or not the filling time is adequate.

	Case 1		Case 2		Case 3		Case 4		Case 5	
(t <sub>1</sub> , T <sub>1</sub> )	0	180	0	180	0	180	0	180	0	190
(t <sub>2</sub> , T <sub>2</sub> )	5	195	0,38	183,5	1,06	189,5	1,44	193,9	3	190
(t <sub>3</sub> , T <sub>3</sub> )	7,19	214	3,99	219,2	4,46	219,3	4,09	219,6	9,02	219,9
(t <sub>4</sub> , T <sub>4</sub> )	9,83	213,6	7,16	210,6	10,73	218,8	9,62	216,7	11,94	217,0
(t <sub>5</sub> , T <sub>5</sub> )	13,02	150	10,36	150	14,34	150	13,01	150	15,32	150

Table 2. The optimal temperature profiles for each case.

### Figure 12: Algorithm optimisation

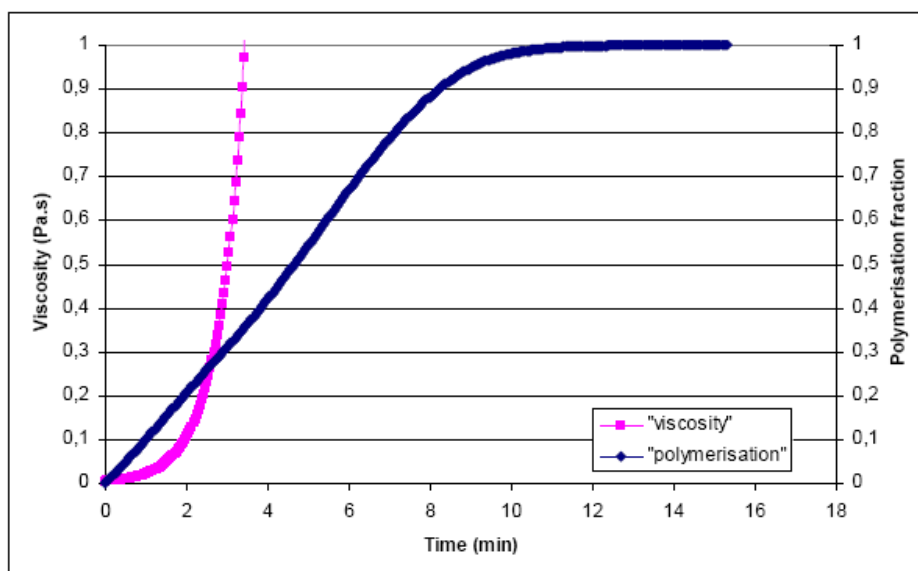


Figure 10. Viscosity and polymerisation evolution of case 5.

It is interesting also to consider the required computational time which is shown in fig. 11 for all test cases. Although the reduction of the initial objective function is not the same for all cases the convergence to their final value is quite similar and takes place in less than 100 CPU seconds which means that the real-time optimisation of the process, if required, is feasible.

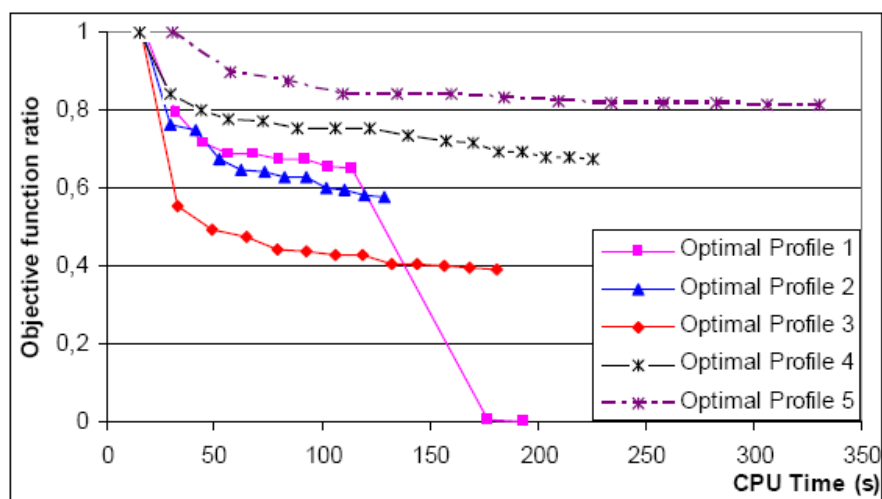


Figure 11. Scaled objective function convergence for each test case with respect to CPU time.

### Figure 13: Algorithm optimisation

## Task 4.5: Control realisation of the proposed strategy in lab scale

Task Leader: Pera

### Objectives:

Lab scale bench marking of the completed Dynamic system

### Progress: This task is complete.

Lab-scale bench testing has been completed by Pera, Exel Composites and UCBL using the chosen materials (Cyclics CBT100 and Caprolactam AP). A two stage approach was adopted, firstly advanced polymerisation prior to injection into the mould tool and secondly, material state monitoring in the mould tool. The resultant deliverable report (Deliverable 22) details the process control achieved using the Dynamic sensor, material state models and process control interface.

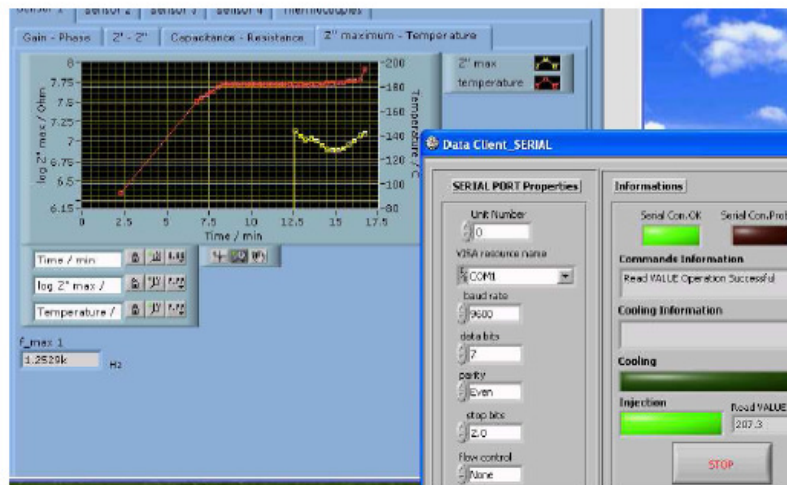


Figure 48: Process control software - Injection

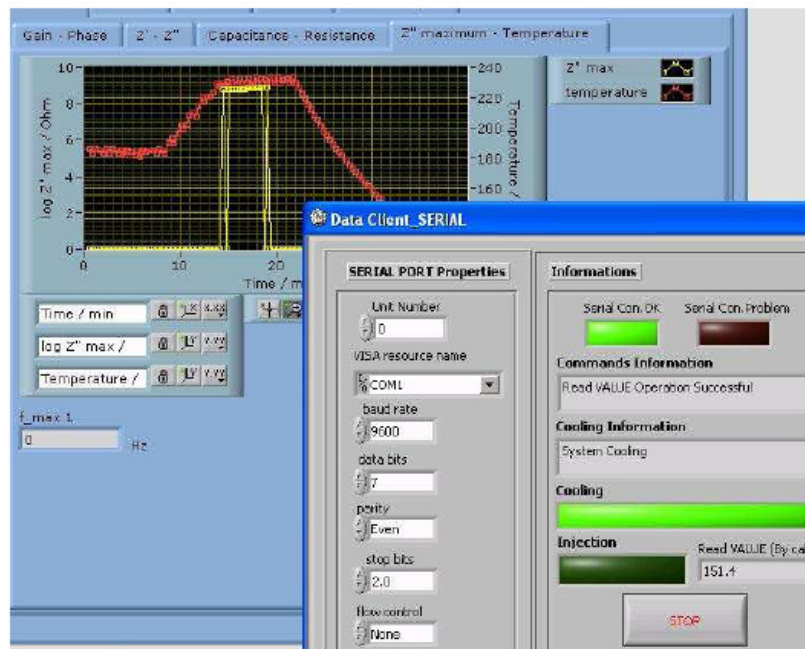


Figure 49: Process control software - Cooling

Figure 14: real time process control on test bench equipment

## 2.2.5. Work package 5 - Integration

### Task 5.1: Control test Specification

Task Leader: NTUA

#### Objectives:

To specify the parameters for the integration, implementation and evaluation of the material state model and process control system into the dielectric monitor.

#### Progress: This task is complete.

Parameters for the control testing specification have been fully defined by NTUA, Exel Composites, Inascoa and Pera during the course of their work on the Dynamic project. This is detailed in D23

### 3.0 Lab Scale Bench Testing Specifications

#### 3.1 Real-Time controllable parameters

Parameter	Characteristic	Value (Min, Mean & Max)			Notes	Test
Operating Temperature	Mould	100°C	193°C	230°C	100°C is the de-mould temperature	Measure
	Injector Head & Static mixer	100°C	193°C	230°C	100°C is the de-mould temperature	Measure
	Pipe work	150°C	173°C	230°C	150 °C is the minimum temperature at which CBT can be expected to flow; 173°C is the temperature to achieve minimum Viscosity.	Measure
Heating rate	Mould	--	--	0.12°C.s <sup>-1</sup>	Heating rate to achieve 193°C to 230°C in 5 minutes	Measure
Cooling rate	Mould	--	--	0.43 °C.s <sup>-1</sup>	Cooling rate to achieve 230°C to 100°C in 5 mins	Measure
Moulding Temperature profile	Mould Injection	173°C	193°C	230°C	230°C moulding temperature indicates pre injection polymerisation heating regime: This may not prove feasible for the proposed system design	Measure
	In Mould Polymerisation	193°C	210°C	230°C]	--	Dynamic Sensor reading

Figure 15: Bench test specification

### Task 5.2: Integration Of Monitoring System To The Materials Model

Task Leader: Pera

#### Objectives:

To integrate and validate the material state model into the dielectric monitoring system.

#### Progress: This task is complete.

This work is detailed in Deliverables 22, 24 and 25



## **Task 5.3 Interfacing monitoring system to process control**

**Task Leader: Pera**

### **Objectives:**

To implement and validate the process control algorithm into the dielectric monitoring system

### **Progress: This task is complete.**

This work is detailed in Deliverables 22, 24 and 25

## **2.2.6. Work package 6 – Pilot Plant Set Up**

### **Task 6.1 Performance requirement Specification**

**Task Leader: Fforce**

### **Objectives:**

To specify the validation strategy and the methodology for performance evaluation of the control system.

### **Progress: This task is complete.**

Performance has been identified as efficient production of parts thus the specification considers *a)* high quality parts, *b)* production cycle and *c)* acceptable scrap percentages. Consideration has been given to the Injections system, thermoplastic RTM and pultrusion processes, as detailed in Deliverable 26

#### **Injection system**

- ✓ A dielectric monitoring system with a dielectric sensor at the barrel should provide a good indication of the status of the material before injection. In this way it will be possible to avoid injections without having resins at the appropriate liquid state and/or viscosity.
- ✓ Also it should be possible to identify the “aging” of the material inside the barrel. The aging of the oligomers refers mainly to the pre-mixed resins and will occur from the extended heating of the oligomers inside the barrel.

#### **Thermoplastic RTM**

- ✓ Resin arrival indication: for RTM it is very important to have a reliable indication of the resin arrival.
- ✓ Progress of polymerization: having an in-situ reactive resin it is important to monitor the progress of the polymerization and to be in the position either to shorten a cycle when the polymer is “done” or to extend a cycle until it is “done”. (control task)
- ✓ Moisture tracing: Even a very small fraction of moisture in the resin may ruin the production. For this reason timely tracing of the moisture is required. From the control a rescue scenario is also required.
- ✓ Premature built-up of crystallization: The significant built-up of crystallization during the initial phase will result in brittle parts which is an unwanted result. So it is important to have an indication of this premature crystallinity built-up in order to allow the process control to overcome this problem.

#### **Thermoplastic pultrusion**

- ✓ Progress of polymerization: having an in-situ reactive resin it is important to monitor the progress of the polymerization and to be in the position either to speed-up a cycle when the polymer is “done” or to extend a cycle until it is “done”. (control task)

### **Figure 16: End user specification**

## Task 6.2 System Installation (Sensor, Monitoring & Control)

Task Leader: Pera

### Objectives:

To install the materials state control system into the pilot plants.

### Progress: This task is complete.

The system has been installed at Pera, Exel Composites and Isojet. Astromal undertook a study on behalf of the consortium of NyRim. Pilot plant trials have been carried out at Technicka Plastica and exel composites as detailed in deliverables 27, 28 and 29.

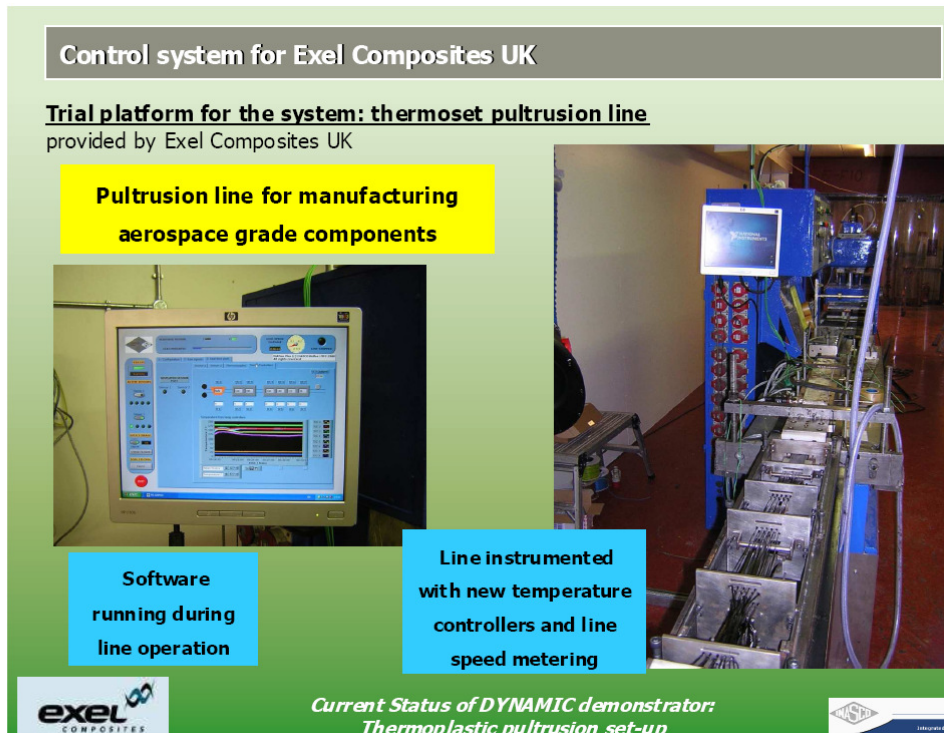


Figure 18: Exel/Dynamic pilot plant

Figure 17: Exel pilot plant

## Task 6.3 Trials

Task Leader: Astromal

### Objectives:

To manufacture components utilising the state based system on the pilot plants.

### Progress: This task is complete.

Deliverables D28 consists of video footage of the trials undertaken on the Exel composites pilot plant. D27 & 29 details the pilot plant operations at Both Technicka Plastica and Exel composites.



Figure 23: RTM at Tech-Plas

In case 7 the quality of the part was good although some bubbles were obvious mainly due to the feeding and a dry spot was observed at the end of the part (fig. below).



Figure 24: RTM at Tech-Plas case 7

Figure 18: Technicka Plastica pilot plant

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In case 7 a process cycle with cooling was tested. The control started the heating again as expected and gave the command to start cooling after conversion rate reached 99% (fig. below).

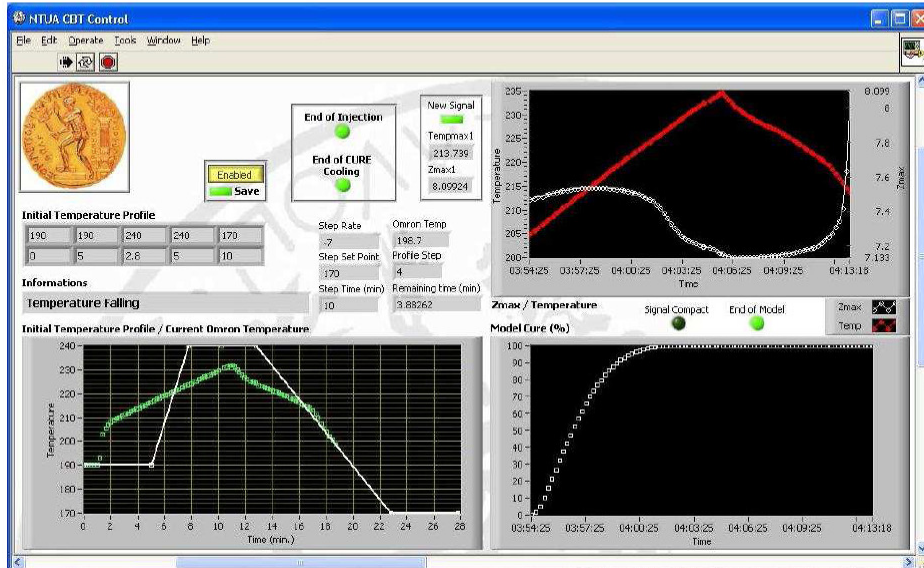


Figure 25 Case 7 process monitoring and control

Figure 19: Technicka Plastica real time process control

## Task 6.4 Analysis Of Results And Evaluation

Task Leader: NTUA

### Objectives:

To evaluate the performance of the material-state control system

### Progress: This task is complete.

Deliverable 29 details the Pilot plant evaluation, and deliverables 24 and 25 assess the performance in the lab scale.

test processing times (minutes)							
sample ID	injection	ramp start	hold start	hold end	De mould	total time	ramp start to hold end time
Athens Control							
907-136a-T1	0	5	14	17	25	25	12
907-136a-T2	0	8	11.5	16	24	24	8
907-136a-T2a	0	4	6.25	12.5	20.5	20.5	8.5
mean	0	5.7	10.6	15.2	23.2	23.2	9.5
sd	0	2.1	4.0	2.4	2.4	2.4	2.2
sd%	#DIV/0!	36.7%	37.4%	15.6%	10.2%	10.2%	22.9%
NP Profile							
907-136-T1	0	3	13	18	26	26	15
907-136-T2	0	3	13	18	26	26	15
907-136-T3	0	3	13	18	26	26	15
short Profile							
907-137a-T1	0	3	7	8	16	16	5
907-137a-T2	0	3	7	8	16	16	5
907-137a-T3	0	3	7	8	16	16	5
Glass reinforced							
Athens Control							
907-137-T1	0	2.5	7.5	10.5	19	19	8
907-137-t2	0	3	9	11	23	23	8
907-137-t4	0	3	8	11.5	17	17	8.5
mean	0	2.8	8.2	11.0	19.7	19.7	8.2
sd	0	0.3	0.8	0.5	3.1	3.1	0.3
sd%	#DIV/0!	10.2%	9.4%	4.5%	15.5%	15.5%	3.5%
NP Profile							
907-136b-T1	0	3	13	18	26	26	15
907-136b-T2	0	3	13	18	26	26	15
907-136b-T3	0	3	13	18	26	26	15

Table 7: Processing times of bench test PBT

Figure 20: Bench test processing time results

**2.2.1. Tech-plas pilot plant materials characterisation**

Three point bend testing was carried out to ISO 178 / BS2782-3 standards

Coupon length was 34 mm and ramp rate was 5mm per minute

The average results over 15 samples (5 samples for each test plaque) is shown

CBT 160 reinforced with continuous woven glass (Tech-Plas Pilot plant trials)

sample ID		width	Depth	Load at Yield (kN)	Displacement at Yield (mm)	Strain at Yield	Stress at Yield (Mpa)	Modulus (FLEX) (GPa)
907-137b-T5		10.31	2.24	0.491	4.2	4.36	510.98	13.31
	SD	0.07	0.01	0.02	0.3	0.3	22.25	0.69
907-137b-T6		10.31	2.18	0.512	4.91	4.96	562.9	13.27
	SD	0.14	0.06	0.025	0.33	0.34	47.17	1.3
907-137a-T3		10.35	2.22	0.492	4.61	4.37	523.03	12.53
	SD	0.14	0.03	0.038	0.32	0.33	37.82	0.58
Mean		10.32	2.21	0.50	4.57	4.56	532.30	13.04
	sd	0.02	0.03	0.01	0.36	0.34	27.17	0.44
	sd%	0.2%	1.4%	2.4%	7.8%	7.5%	5.1%	3.4%

**Conclusions and perspectives**

Overall the trials showed that these reactive thermoplastic matrices are very powerful and much

**Figure 21: Technicka Plastica pilot plant mechanical property results**

### 2.3. Deviation from the Plan and Corrective Actions

The project was seriously delayed by the time taken to resolve the issues associated with the plating and polishing regimes of the sensor, this affected final fabrication and delivery of sensors for testing and pilot plant trials. The consortium reviewed progress at the quarterly management meetings and at the M27 Management meeting a complete review of the project plan was undertaken and deadlines set for delivery by partners for each critical deliverable. This resulted in a modified timing plan being submitted to the E.C which was subsequently reviewed at each meeting. By undertaking this action important dissemination and training activities could be scheduled well in advance and in the knowledge that key consortium members would be available to support the events. The project extension allowed the consortium to deliver undertake D36 (presentation at a major plastics industry conference) at JEC which is considered to be the premier event of its type in Europe.

The Description of Work (DoW) was amended following changes to the consortium during the second reporting period. These changes led to a Contract Amendment (**No.3**) being submitted and accepted by the EC.

During the third reporting period the project was extended from 36 to 42 months to allow for all the technical work to be completed. Consequently, a Contract Amendment (**No.5**) was requested and accepted by the EC.

WP No.	Work package title	Deviations from Plan	Corrective Action
1	Hardware development	Inclusion of Nanoforce Technologies to develop an alternative design of sensor.	N/A
2	Materials Model	None	N/A
3	Development of Process Monitoring	None	N/A
4	Development of Process Control	None	N/A
5	Integration	None	N/A
6	Pilot Plant Set-Up	None	N/A
7	Innovation and Dissemination	None	N/A
8	Training	AHPI withdrew from consortium, Eurexcel took responsibility for all WP 8	N/A
9	Consortium Management	None	N/A
10	Project Management	None	N/A

## 2.4. Work Package Deliverables Update

The specific objectives for the 18-month period of 1 October 2005 – 31 March 2007 of the project are summarised in the table below.

Del. No.	Deliverable Name	WP No	Lead Participant	Estimated Persons Month	Nature	Dissemination Level	Delivery Date	Progress Towards Achieving Objectives	% Complete	Date Submitted to the EC
D1	Dielectric sensor and heating/cooling system specifications	1.1	Inasco	4.5	R	CO	1	Complete	100%	Dec 2006
D2	Dielectric sensor design	1.2	Inasco	2.0	O	CO	7	Complete	100%	Dec 2006
D3	Heating/cooling system design	1.3	Exel	2.2	P	CO	9	Complete, combined with D5	100%	Dec 2006
D4	Prototype of dielectric sensor	1.4	Pera	1.0	R	CO	37	Complete	100%	Jun 2009
D5	Prototype of heating/cooling system	1.4	Pera	1.0	R	CO	11	Complete, combined with D3	100%	Dec 2006
D6	Test Results on dielectric sensor	1.5	Pera	1.0	R	CO	38	None		Jun 2009
D7	Test Results on heating/cooling system	1.5	Isojet	2.2	R	CP	12	Complete	100%	Mar 2009
D8	Prototype of sensor mounting	1.6	Pera	3.4	R	CP	12	Complete	100%	Nov 2006

Del. No.	Deliverable Name	WP No	Lead Participant	Estimated Persons Month	Nature	Dissemination Level	Delivery Date	Progress Towards Achieving Objectives	% Complete	Date Submitted to the EC
D1.7	Fabrication of an all-ceramic sensor tile for Dielectric monitoring of thermoplastic composites	1.7	Isojet	1.19	R	CO	24	Complete	100%	Mar 2009
D1.8	Multilayer sensor grid prototype	1.8	Pera	3.3	P	CO	27	Complete	100%	Jun 2009
D1.9	Tooling & Construction	1.9	Pera	2.3	O	CO	27	Complete	100%	Jun 2009
D1.10	Optimisation of ceramic sensor design	1.10	Pera	4.1	R P	CO	30	Complete	100%	Jun 2009
D1.11	Production assessment	1.11	Pera	1.65	R	CO	31	Complete	100%	Jun 2009
D9	Selected materials for detailed study	2.1	Astromal	4.7	O	CO	1	Complete, CBT160 and selected NyRIM	100%	N/a
D10	Thermal and rheological characterisation of the selected materials	2.2	UCBL	10.0	R	CO	12	Complete	100%	Dec 2006
D11	Correspondence between dielectric signal and material properties	2.3	UCBL	6.7	R	CO	12	Complete	100%	May 2007



Del. No.	Deliverable Name	WP No	Lead Participant	Estimated Persons Month	Nature	Dissemination Level	Delivery Date	Progress Towards Achieving Objectives	% Complete	Date Submitted to the EC
D12	Health monitoring	2.4	UCBL	1.7	O	CO	15	Complete	100%	Mar 2009
D13	Signal processing methodology in acquiring dielectric response from sensors	3.1	NTUA	2.3	R	CO	2	Complete	100%	Aug 2006
D14	Monitoring strategy in thermoplastic composites processing	3.2	Inasco	3.8	R	CO	7	Complete	100%	Dec 2006
D15	Data collection and analysis methodology in deriving material state	3.3	Isojet	2.3	O	CO	9	Complete	100%	Nov 2006
D16	Prototype multiplexer and integration to dielectric monitoring system	3.4	Inasco	1.7	P	CO	12	Complete	100%	Dec 2006
D17	Guidelines for selecting dielectric measurement parameters	3.5	NTUA	1.3	R	CO	12	Complete	100%	Dec 2006
D18	Report on the expected variability for the	4.1	Pera	4.2	R	CO	3	Complete	100%	May 2006

Del. No.	Deliverable Name	WP No	Lead Participant	Estimated Persons Month	Nature	Dissemination Level	Delivery Date	Progress Towards Achieving Objectives	% Complete	Date Submitted to the EC
	pilot processes and materials									
D19	Report on the appropriate control strategies and selection criteria	4.2	NTUA	6.2	R	CO	5	Complete	100%	Dec 2006
D20	Modelling of the pilot processes and materials	4.3	NTUA	8.5	R	CO	9	Complete	100%	May 2007
D21	Numerical optimisation of the pilot processes and materials	4.4	NTUA	7.2	O	CO	15	Complete	100%	Oct 2007
D22	Control realisation of the proposed strategy in lab scale	4.5	Pera	4.3	R	CO	15	Complete	100%	June 2009
D23	Test Specification	5.1	NTUA	6.0	R	CO	13	Complete	100%	Jun 2009
D24	Test Results of Laboratory Trials	5.2	Pera	9.1	R	CO	38	Complete	100%	Jun 2009
D25	Performance Evaluation Report	5.3	Pera	6.8	R	CO	39	Complete	100%	Jun 2009
D26	Performance Specification	6.1	Exel	8.5	R	CO	19	Complete	100%	Oct 2007
D27	Installation of Process Control Equipment on the Pilot Plants	6.2	Pera	10.1	O	CO	40	Complete	100%	Jun 2009

<b>Del. No.</b>	<b>Deliverable Name</b>	<b>WP No</b>	<b>Lead Participant</b>	<b>Estimated Persons Month</b>	<b>Nature</b>	<b>Dissemination Level</b>	<b>Delivery Date</b>	<b>Progress Towards Achieving Objectives</b>	<b>% Complete</b>	<b>Date Submitted to the EC</b>
D28	Demonstration of Process Control the Pilot Plants	6.3	Astromal	15	D	CO	40	Complete	100%	Jun 2009
D29	Pilot Plant Evaluation Report	6.4	NTUA	2	R	CO	41	Complete	100%	Jun 2009
D30	Demonstration of NyRIM manufacture using material-state process control	7.1	CUKL	3.5	D	CO	40	Complete	100%	Jun 2009
D31	Training Programme for IAGs and SMEs	7.2	CUKL	5.9	O	CO	37	Complete	100%	June 2009
D32	Multi Media Web Site	7.3	CUKL	2.0	O	PU	38	Complete	100%	June 2009
D33	Technology seminar by IAGs to associate members	7.4	CUKL	5.4	O	PU	38	Complete	100%	June 2009
D34	Demonstration event of material-state process controller	7.5	CUKL	3.6	D	PU	40	Complete	100%	June 2009
D35	Training seminar by IAGs to associate members	8.1	Eurexcel	14.4	O	PU	41	Complete	100%	June 2009
D36	Presentation at major plastics	8.2	Eurexcel	14.3	O	PU	42	Complete	100%	June 2009

<b>Del. No.</b>	<b>Deliverable Name</b>	<b>WP No</b>	<b>Lead Participant</b>	<b>Estimated Persons Month</b>	<b>Nature</b>	<b>Dissemination Level</b>	<b>Delivery Date</b>	<b>Progress Towards Achieving Objectives</b>	<b>% Complete</b>	<b>Date Submitted to the EC</b>
	industry conference									
D37	Publish a brief project presentation	10.1	CUKL		O	PU	3	Complete	100%	Mar 2006
D38	Plan for using and disseminating knowledge	9.1	CUKL	1.9	R	CO	36	Complete	100%	Jun 2009
D39	6 monthly reports	10.1	CUKL	2.2	R	CO	6 monthly	Complete	100%	Jun 2009
D40	18 Month Mid term review	10.1	CUKL	2.2	O	CO	19	Complete	100%	Jun 2009
D41	Final Project meeting	10.1	CUKL	2.2	O	CO	42	Complete	100%	Jun 2009
D42	Final project presentation	10.1	CUKL	2.2	O	PU	42	Complete	100	Jun 2009

## 2.5. Work Package Milestones Update

Work-package No	Milestone Deliverable	Month	Update	Partner
WP1	Fabricated prototype dielectric sensor	Month 9	Complete	Pera
WP1	Assembled and tested dielectric sensor mounting	Month 12	Complete	Pera
WP1	Assembled and tested heating/cooling system	Month 12	Complete	Pera
WP2	Materials characterised (thermal behaviour mapped and viscosity models derived)	Month 12	Complete	UCBL
WP2	Dielectric signal mapped to suitable material state index	Month 12	Complete	UCBL
WP3	Dielectric monitoring system for multiple sensors and optimal monitoring strategy	Month 12	Complete	Inasco
WP4	Demonstration of Process Control	Month 15	Complete	NTUA
WP5	Laboratory-scale demonstration of integrated process control	Month 18	Complete	Pera
WP6	Manufactured NyRIM components using the material-state control system	Month 24	Complete	Astromal
WP7	Project presentation	Month 36	Complete	EurExcel
WP8	Course feedback from IAG seminars to associate members	Month 36	Complete	Eurexcel

## 3.0 CONSORTIUM MANAGEMENT

### 3.1 Consortium Status Overview

The Dynamic consortium has remained very focused on ensuring all the deliverables have been met and have worked very closely in order to achieve this. Working party meetings have taken place between partners as and when necessary in order to resolve issues and undertake trials. There was a period of high activity between Inasco, NTUA and Pera Innovation on integration of the software systems with the test equipment which resulted in an internet link being established for the feedback of data from Pera to Inasco and NTUA, this meant that real-time monitoring of the trials could take place and software amendments made where necessary immediately.

Dissemination activities were given real focus in the last twelve months of the project, the IAG's set-up a number of events which were supported by representatives from Pera, UCBL, NTUA and Inasco who all delivered presentations in their specific area's of research.

The consortium experienced a number of partner changes which ultimately strengthened the project:

- AHPI withdrew from the project following no input and a lack of confidence from the partners that they would deliver their allotted work. Instead of seeking a new partner, it was agreed by the consortium that EurExcel would pick-up their workload and associated budget.
- Nanoforce Technologies joined the consortium after 18 months. This was done to facilitate the development of a dielectric sensor from novel sintering techniques as, at this time, the consortium were experiencing technical difficulties with the development of a ceramic sensor.

### 3.2 Project Timetable & Status



Figure 22: Original Gantt chart (Dynamic 2005)

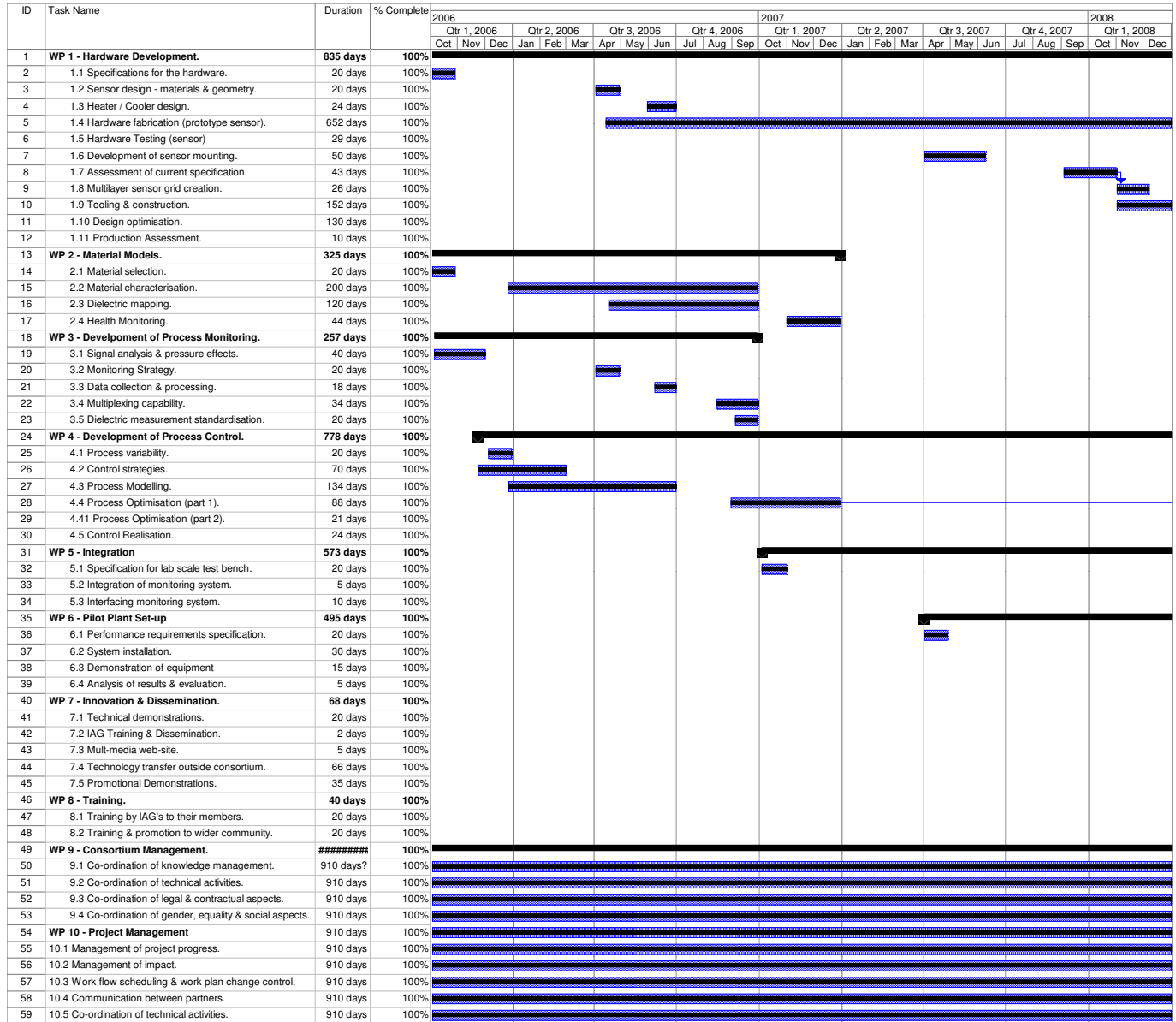


Figure 23: Final Gantt chart (Dynamic September 2008)



### 3.3 Meetings and Communication

There have been twenty three main project review meetings during the course of the project. These have combined technical, management and exploitation issues and are shown thus:

Details of project review meetings

	Date	Type of meeting	Location
1	04.11.05	Kick-Off Meeting	Pera, Melton Mowbray, UK
2	26.11.05 27.11.05	Management Meeting Technical Meeting	UCBL, Lyon, France
3	22.05.06 23.05.06	Management Meeting Technical Meeting	Inasco, Athens, Greece
4	27.09.06 28.09.06	Management Meeting Technical Meeting	Astromal, Poland
5	12.12.06 13.12.06	Management Meeting Technical Meeting	UCBL, Lyon, France
6	18.04.07 19.04.07	Management Meeting Technical Meeting	Pera, Melton Mowbray, UK
7	18.07.07 19.07.07	Management Meeting Technical Meeting	UCBL, Lyon, France
8	04.10.07 05.10.07	Management Meeting Technical Meeting	NTUA, Athens, Greece
9	13.02.08 14.02.08	Management Meeting Technical Meeting	Exel Composites, Runcorn, UK
10	22.05.05 23.05.08	Management Meeting Technical Meeting	Pera, Melton Mowbray, UK
11	15.09.08 16.09.08	Management Meeting Technical Meeting	NWCC, Manchester, UK
12	18.11.08	Management Meeting Technical Meeting	Nanoforce Technologies, QMUL, London, UK
13	23.03.09	Final Project Meeting	Paris, France

All of the technical and management meetings during the course of the project have been well attended and knowledge-transfer has been extensive throughout as described in the meeting minutes.

At each meeting each participant gave a presentation of the work undertaken during the last three month period. Each three month meeting was split into two days, a Management meeting on the first day and a Technical meeting on the second day, this ensured that all issues surrounding the

project were raised and discussed with a clear action plan agreed in order to address any issues. During each event a partner dinner was organised which helped to build strong relationships among the partners.

Many meetings took place between partners as and when required to discuss specific area's of work, these often included set-up and trials of equipment and software. For example, Inasco and NTUA spent several day's at Pera helping to install the software and PXI equipment onto the test rig and optimise the system.

## **4.0 DISSEMINATION ACTIVITIES**

### **4.1 Management of Knowledge and IPR**

A Consortium Agreement has been produced that defines the exploitation framework. Specific roles have been assigned namely that of Exploitation Manager and IPR Manager. Exploitation and IPR protection has been discussed throughout the course of the project during the 3-month Management meetings. Composites UK Ltd are responsible for protecting the technology.

### **4.2 Plans for Using & Disseminating Knowledge**

#### **Routes to Exploitation**

An Exploitation plan has been produced which can be seen in D38 (Plan for using and disseminating knowledge). The SME's in the consortium will form the main route to exploit the technology, Inasco will be responsible for building and selling the Dynamic system and have particular expertise in this area through other collaborative E.C projects such as Condicomp and Comprone, both of which are concerned with dielectric monitoring. The technology will be licensed to provide an income stream for the other SME's in the project (Astromal, Exel Composites and Isojet). Isojet have the opportunity to retrofit the Dynamic system for their customer base in their day to day business activities of bespoke systems development.

#### **Validation of the Technology**

The Dynamic system has been validated through trials at Pera, Exel Composites and Technika Plastica. These validation trials have covered Pultrusion and Injection Moulding processes and the results show that the technology can be used for real-time in-situ monitoring of reactive thermoplastics and that the system is capable of being retrofitted to a production line with the minimum of disruption. As the technology is validated and there is a comprehensive set of data available from such trials it is possible to accurately predict cost savings and scrap reductions to potential customers.

## Dissemination Method

The Dynamic project has been disseminated throughout the life of the project, mostly through major trade shows such as JEC. At JEC 2009, the technology was displayed by Composites UK and a presentation given at one of the show's forums

Dissemination of the technology will carry on after the end of the project mainly through Inasco, CUKL, NTUA and Pera all of which are heavily involved development of thermoplastic materials and processing. Pera have already been approached by two companies interested in getting access to the technology and developing it further.

### 4.3 Exploitation & Dissemination Activities Undertaken

The major opportunity to disseminate the Dynamic technology has been at the JEC Exhibition in Paris each year which is the major trade exhibition and conference for the industry in Europe. In addition, all partners have taken every opportunity to disseminate the technology at events attended by or organised by themselves. UCBL and NTUA have been very pro-active in this type of activity. CUKL and Pera have set-up web-sites that show the Dynamic technology and these have been referred to in several publications. Towards the end of the project CUKL and Eurexcel hosted specific dissemination events and invited an audience from their respective membership databases. Inasco, Isojet and Pera have discussed the technology with their clients and project partners where applicable.

The efforts of the consortium as a whole to disseminate the Dynamic technology has resulted in the project being known by most companies who operate in the field throughout Europe.

Dissemination activities undertaken throughout the life of the project are detailed herein:

2006

#### Publications

**Boiteux G., Seytre G., Stevenson I.** - In situ monitoring of structure evolution by dielectric spectroscopy

*Short Course Nanofun-Poly on Relaxation Phenomena, Poznan, Pologne 1-2 September 2006*

2007

#### Publications

**Hakme C., Stevenson I., Maazouz A., Cassagnau P., Boiteux G., Seytre G.** 2007. In situ monitoring of cyclic butylene terephthalate polymerisation by dielectric sensing. *Journal of Non-Crystalline Solids*, 353, 4362-4365.

### International - Invited Conferences

**Boiteux G., Hakme C., Stevenson I., Maazouz A., Seytre G.** 2007. *In-situ* Monitoring of Structure Evolution by Dielectric Spectroscopy, *NATO Workshop "Meeting the Challenges of the 21st Century : Novel applications of Broadband Dielectric Spectroscopy"*, 22-26 July, Suzdal, Russia

International Communications

Oral Communications

**Dargeres N., Hakme C., Boiteux G., Melis F., Bardash L., Botta L., Seytre G.** 2007. Conductive polymer composites based on cyclic butylene terephthalate oligomers and carbon fibers, *European Polymer Congress, EPF- 2007, 2 – 6 July 2007, Portoroz, Slovenia*

**Boiteux G.** 2007. In – situ Monitoring of built-up of polymers based materials by Dielectric Spectroscopy, *TOK : Marie Curie "DIELPOL" 2006-2009, 15 November 2007, Lodz, DPM, TUL, Lodz, Pologne*

**McCarthy N., Powell J.** 2007. Sustainability Live 2007 (Environmental Technologies And Environmental Services 2007), 1 – 3 May 2007, National Exhibition Centre, Birmingham, UK

Posters

**Bardash L., Rybak A., Dargere N., Hakme C., Boiteux G., Melis F., Seytre G.** 2007. Nouveaux Composites Polymères Conducteurs sur la base des Oligomères de Butylène Téréphthalate Cycliques Chargés de Micro- ou Nanoparticules, *MNPC'07, Matériaux et nanostructures □-conjugués, 17-21 Septembre 2007, Grau du Roy, France*

**Bardash L., Boiteux G., Seytre G., Hakme C., Fainleib A.** 2007. Nanostructured polymer nanocomposites based on polybutylene terephthalate and carbon nanotubes. XI Ukrainian conference on macromolecular compounds, 1-5 October 2007 , Dnipropetrovsk, Ukraine.

2008

Publication

**Bardash L., Boiteux G., Seytre G., Hakme C., Dargere N., Rybak A., Melis F.** 2008. Novel conductive polymer composites based on poly(butylene terephthalate) filled with carbon fibers, *e-Polymers, 155, 1-6*

Invited Conferences

**Boiteux G., Hakme. C., Stevenson I., Seytre G., Maazouz A.** - Dielectric sensing in reactive thermoplastic composites processing

COMPROME, 6fp, Villeurbanne, France, 23 . 01. 2008

International Communications

Oral Communications

**Bardash L., Boiteux G., Rybak A., Seytre G., Hakme C.** 2008. Conductive Polymer Nanocomposites Based On Cyclic Oligomers Filled With Multiwalled Carbon Nanotubes *Short Course Nanofun-Poly, 13-15 April 2008, Terni, Italy*

**Boiteux G.** In-situ monitoring of built-up of polymers based materials by Dielectric Spectroscopy

*NWCC Dissemination Event, Manchester, 16 Septembre 2008*

**Boiteux G., Rybak A., Cavetier L., Melis F., Cassagnau P., Seytre G., Bardash L, Fainleib A., Iurchenko M., Levchenko V., Mamunya Y.E., Ulanski J.** 2008. Broadband

Dielectric Spectroscopy applications to the studies of Conductive Polymer Composites (CPC) as Smart Materials for Temperature and Solvent Vapour Sensing  
TOK : Marie Curie "DIELPOL" 2006-2009, 7 November 2008, DPM, TUL, Lodz, Pologne

**Boiteux G.**, *In Mould Monitoring of Reactive Polymer Processes*  
Dissemination Event, Scientific Foundation for Thermoplastics, London, 19 November 2008

**Maistros G.**, In-mould monitoring of reactive polymer processes: Industry challenges and innovative solutions in Sensors and Measurement Science: Knowledge and Innovation Transfer Event: Chemistry Innovation, The Sensors KTN and the Centre for Process Analytics and Control Technology, London, 20 November 2008

#### Posters

**Bardash L., Boiteux G., Seytre G., Hakme C., Alcouffe P., Fainleib A.** - Novel conductive polymer composites based butylene terephthalate oligomers filled with micro- or nanoparticles, *POLYCHAR 16 World Forum for Advanced Materials*, 17-21 February, 2008, Lucknow, Inde

**Bardash L., Boiteux G., Rybak A., Seytre G., Fainleib A.** 2008. Conductive Polymer Nanocomposites Based On Poly(Butyleneterephthalate) And Multi-Walled Carbon Nanotubes : *4th International Symposium on Nanostructured and functional polymer-based materials and composites, NANOFUNPOLY-*, 16-18 April 2008, Rome, Italy

**Bardash L., Boiteux G., Seytre G., Fainleib A.** 2008. Electrical Properties of Polymer Nanocomposites based oPoly(butylene terephthalate) from Cyclic Butylene Terephthalate and MWCNT, *5<sup>th</sup> International Conference of Broadband Dielectric Spectroscopy (10<sup>th</sup> DRP and 5<sup>th</sup> IDS)*, 26-29 August 2008, Lyon, France

**Pantelelis N., Hakme C., Stevenson I., Boiteux G., Seytre G.** 2008. In-situ monitoring, conductivity, cyclic butylene terephthalate (CBT) oligomers , crystallization, polymerization, reactive thermoplastic, *5<sup>th</sup> International Conference of Broadband Dielectric Spectroscopy (10<sup>th</sup> DRP and 5<sup>th</sup> IDS)*, 26-29 August 2008, Lyon, France

#### National Communications

##### Posters

**Bardash L., Rybak A., Dargère N., Hakme C., Boiteux G., Melis F., Seytre G.** 2008 Nouveaux Composites Polymères Conducteurs sur la base d'Oligomères de Butylène Téréphthalate Cycliques chargés de Micro-ou Nanoparticules. *Journée de polyméristes lyonnais 2008*, 01 February 2008, Villeurbanne, France

**Bardash L., Boiteux G., Seytre G., Fainleib A.**, Synthesis and characterization of nanocomposites from cyclic butylene terephthalate oligomers and carbon nanotubes, *VI Open Ukrainian Conference of Young Scientists on Macromolecular Compounds "VMS-2008"*, September 30 – October 3, 2008, Kyiv, Ukraine, *Book of abstracts*, p. 172.

**Bardash L., Boiteux G., Seytre G., Fainleib A.** 2008. **Electrical Properties of Polymer Nanocomposites** based on

Poly(butylene terephthalate) from Cyclic Butylene Terephthalate and MWCNT, *Colloque Annuel du Groupe Français des Polymères (GFP)*, 25-27 Novembre 2008, Lyon, France

2009

International Communications

Oral Communications

**Boiteux G. In Mould Monitoring of Reactive Polymer Processes  
Dissemination Event, Scientific Foundation for Thermoset, Paris, 31 March 2009**

**Boiteux G, In Mould Monitoring of Reactive Polymer Processes  
Dissemination Event Scientific Foundation for Thermoplastics, Paris, 31 March 2009**

**Bardash L., Boiteux G., Seytre G., Fainleib A., Cassagnau P.** 2009. The altering of high performance properties of poly(butylenes terephthalate) by introducing of mwcnts, *POLYCHAR 17 World Forum for Advanced Materials*, 19-24 April 2009, Rouen, France  
Posters

**Bardash L., Fainleib A., Boiteux G., Seytre G., Grigoryeva O.** 2009. Novel High-Temperature Resistant Polycyanurate / Mwcnts Nanocomposites, *5<sup>th</sup> International Symposium on "Nanostructured and functional polymer-based materials and composites"*, (NANOFUNPOLY), 15-17 Avril 2009, Paris, France

**Bardash L., Korskanov V. Fainleib A., Boiteux G., Levchenko V., Mamunya Y.** 2009. **Effect of small additives of carbon nanotubes on thermal and electric conductivities of novel nanocomposites based on polycyanurate network**, *7th International Conference Eurofillers "From macro to nanofillers for structural and functional polymer materials"*, June 21st to June 25th, 2009, Alessandria, Italy

## **PUBLICATION**

JEC SHORT PUBLICATION

## **5 OTHER ISSUES**

### **5.2 Conclusions**

The project is considered to have been a technical success with the successful production of a dielectric in-situ monitoring system of reactive thermoplastics consisting of **a)** dielectric sensor, **b)** PXI hardware and **c)** software system. A great deal of data has been gathered concerning polymerisation progress in thermoplastics (cPBT) and the material characterisation of same.

The selection of case study materials was somewhat pre-determined through the needs of the end-users in the consortium and from minimal amount of materials used within this technology, NyRIM proved to be a highly difficult material to work with and progress with this material was slow, however, CBT was a good choice and extensive progress was made with this material.

AHPI were not able to deliver their facets of the dissemination and training, however, the project benefited from the fact that Eurexcel were willing and capable of taking on their tasks and delivering them in full.

The duration of the project (36 months) was correct planning, if it were not for the technical challenges associated with the plating and polishing regimes of the sensor then it would not have required the extension to 42 months. The consortium appreciated the support of the E.C in allowing it to analyse the correct time required for the project extension. To this end, the project benefited from ensuring issues were communicated to the E.C in good time so that solutions could be found that were acceptable to the sponsor.

The inclusion of Nanoforce into the consortium was of benefit to the project and much has been learn't about alternative methods of production which will prove to be of significant benefit to the SME's and RTD's involved.

On the whole the right partners were chosen for the project, the IAG's and RTD's were very pro-active along with Inasco and Fibreforce who were very pro-active organisations. Isojet were limited to what they could do within the project owing to the nature of their business and lack of available plant in which to undertake trials.