



*DT-NMBP-01-2018*

*Open Innovation Test Beds for Lightweight, nano-enabled multifunctional composite materials and components (IA)*

## OASIS

**Open Access Single entry point for scale-up of Innovative Smart lightweight composite materials and components**

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Duration: 44 months

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## = Deliverable: D1.6 =

**Catalogue of Up-graded performances of PLs for nano-intermediate products**

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RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	



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## Executive Summary

This deliverable (DEL D1.6) details the upgrading and upscaling of five pilot lines related to the production of nano-intermediate materials:

- **PL4: BUCKYPAPER** (Self-supporting continuous sheets of entangled MWCNTs “Buckypapers”), owned by TECNALIA
- **PL5: CNT DOPED VEILS** (Lightweight and thermoplastic nonwovens doped with CNTs), owned by TMBK Partners
- **PL6: R2R** (CNT treated prepregs), owned by ADAMANT Composites
- **PL7: PICTIC** (Sheet to sheet Printed devices), owned by CEA
- **PL8: SIMPNANO** (Nanoreinforced metallic alloy ingots), owned by TECNALIA

The deliverable describes:

- The purpose of each pilot line and the needs for the upgrading and/or upscaling carried out.
- An overview of the upgrade/upscale for each pilot line.
- Summary of the capabilities of each pilot line as well as what each offer in terms of product(s) and processes.

The delay in the submission of this deliverable was due to the COVID-19 crisis, which stopped the development activities for most of the PLs for several months.

## Table of Contents

1. Content of the Deliverable.....	5
2. Objectives of the upgrade/upscale of the PLs for intermediate products with nanoscale features .....	5
3. Upgrade of the Pilot lines for the production of intermediate products with nanoscale features .....	7
4. Conclusions .....	24
5. Degree of Progress .....	24
6. Dissemination Level.....	24
7. Appendix .....	25

## 1. Content of the Deliverable

This deliverable (DEL D1.6) focuses on the upgrade of the five Pilot Lines (PLs), whose operations relate to the production of nanointermediate products: BUCKYPAPER by Tecnalia, CNT DOPED VEILS by TMBK Partners, R2R by ADAMANT Composites, PICTIC by CEA and SIMPNANO by Tecnalia.

A description of each of the five pilot lines provides an overview of the upgrade/upscale work carried out, as well as details of the capability of each of the facilities within the OASIS Open Innovation Test Bed (OASIS OITB).

## 2. Objectives of the upgrade/upscale of the PLs for intermediate products with nanoscale features

The vision of OASIS is an ecosystem of 12 nanotechnology manufacturing pilot lines (PLs), operating under a common and demanding umbrella of Sustainable Production (OASIS framework). It aims at ensuring a competitive, high quality, safe and environmentally friendly production, of nanomaterials, nano-intermediates and nano-enabled products in compliance with the applicable regulations.

One major task of WP1 aims at upgrading the 12 PLs integrated within the OASIS OITB to meet industrial needs:

- Upgrade of the 3 pilot lines for *nanoscale structures* (nanomaterials) in unprocessed form with intrinsic functionalities (**DEL 1.5**)
- Upgrade of the 5 pilot lines for *intermediate products* with nanoscale features (**DEL 1.6**)
- Upgrade of the 4 PLs for *nano-enabled products* (**DEL 1.7**)

This deliverable (DEL D1.6) details the upgrading and upscaling of the five pilot lines related to the production of nano-intermediate products:

- **PL4: BUCKYPAPER** (Self-supporting continuous sheets of entangled MWCNTs “Buckypapers”), owned by TECNALIA
- **PL5: CNT DOPED VEILS** (Lightweight and thermoplastic nonwovens doped with CNTs), owned by TMBK Partners
- **PL6: R2R** (CNT treated preregs), owned by ADAMANT Composites
- **PL7: PICTIC** (Sheet to sheet Printed devices), owned by CEA
- **PL8: SIMPNANO** (Nanoreinforced metallic alloy ingots), owned by TECNALIA

The KPIs for the upgrade/upscale of the intermediate products with nanoscale features are presented in Table 1.


Similarly, the upgrade of the pilot lines related to both nanoscale structures and nano-enabled products are presented in DELs D1.5 and D1.7 respectively.

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Nanointermediates	PL#4. Buckypapers. Self-supporting sheets comprised of entangled CNT. <u>Functionalities:</u> Mechanical and electrical properties.	
	<u>Current production rate</u> 10 m <sup>2</sup> /week	Enhanced capacity through filter/ support upgrade. New washing and packaging modules. Control & register of dispersion content/ quality; NEP properties (thickness, EC); process defects detection/prevention
	<u>OASIS target</u> 50 m <sup>2</sup> /week	
	PL#5. CNT veils. Lightweight and thermoplastic nonwovens doped with CNTs. <u>Functionalities:</u> High flexibility and lightweight. Electrical and mechanical properties	
	<u>Current production rate</u> 250 m <sup>2</sup> /week	Control of veils thickness, areal weight and surface defects
	<u>OASIS target</u> 1000 m <sup>2</sup> /week	
	PL#6. FXPIy <sup>TM</sup> CNT treated preregs. <u>Functionalities:</u> Mechanical, Electrical and Thermal Properties	
	<u>Current production rate</u> 500 m <sup>2</sup> /day. Roll up to 600 mm width	Inline quality control and monitoring of key processing step (eg. online dispersion mapping using machine vision, prepreg thickness, tackiness control). Inline monitoring of environmental conditions and H&S. Inline Traceability
	<u>OASIS target</u> 1000 m <sup>2</sup> /day	
	PL#7 PICTIC Sheet to sheet Printed devices PL. Smart printed sensors and actuators. <u>Functionalities:</u> Smart sensing.	
	<u>Current production rate</u> Sheet: 320*380 cm <sup>2</sup> Production yield >70% (depending on the device size & resolution)	Control & mapping of the defects (scratches, cracks, contamination, aggregates... [1-100µm] size range) on the successive printed layers by in-line camera,
	<u>OASIS target</u> Production yield > 95%, for a device compliant with de design rules	
	PL#8 SIMPnano Metallic (Al) alloys with dispersed nanoreinforcements. <u>Functionalities:</u> Mechanical, thermal and wear resistance properties.	
	<u>Current production rate</u> 3kg/day lab furnace	Automatic nanoparticles dosing and dispersion Real-time chemical composition of elements, accuracy <1%
	<u>OASIS target</u> 100kg/day industrial furnace	

Table 1: KPIs for the upgrade of PLs for intermediate products with nanoscale features

### 3. Upgrade of the Pilot lines for the production of intermediate products with nanoscale features

	<b>Datasheet for pilot line: BUCKYPAPERS</b>
<b>OITB Member</b>	Tecnalia
<b>Name of Pilot line</b>	BUCKYPAPER – MWCNT based continuous sheets
<b>Number of the Pilot Line</b>	<b>PL4</b>
<b>TRL of pilot line</b>	TRL 6

#### Description of the pilot line:

The BUCKYPAPER pilot line operated by Tecnalia is located in San Sebastian in the Basque Region of Spain. The pilot line integrates filtration, washing and drying/package modules (Figure 1) for the manufacture of continuous flexible sheets based on multi-walled carbon nanotubes (MWCNTs). These sheets, also known as “buckypapers”, are randomly oriented self-supporting MWCNT structures, that are lightweight, flexible, highly conductive and offer multifunctionality for a wide range of industrial sectors.

The process uses dynamic vacuum filtration of aqueous based MWCNTs dispersions to produce sheets up to 100m long and 300mm wide (Figure 2). Depending on the required material characteristics, buckypapers can be manufactured in the range of 20-60 gsm. The plant currently has a production capacity of 25m<sup>2</sup>/week (which is being increased to 50m<sup>2</sup>/week by 2021).

It is important to note that whilst the current product range is based on MWCNTs, the pilot line is also capable of filtering other materials (nano, macro) as long as the material is capable of being dispersed in water.

#### Integrated modular design

The pilot line has a modular design – all modules can be fully integrated as a continuous process line or can simultaneously be used independently from each other. This enables process flexibility (e.g. filtration and washing modules are interchangeable for their principal functions).

Additional water-based treatments of the filtered buckypaper sheet can also be carried out on either module (e.g. introduction of property enhancing elements, such as metallic oxides). This flexibility in module functionality helps increase line output, as well as the ability to offer a wider product range from the same pilot line.

#### Images



**Figure 1:** Buckypaper pilot line showing filter membrane prior to filtration; Buckypaper pilot line in operation.



**Figure 2:** Continuous MWCNT sheet section after filtration and washing

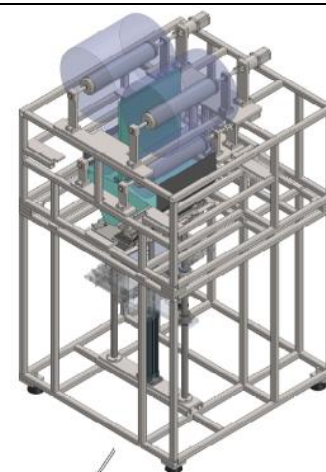


The line is composed of the following modules:

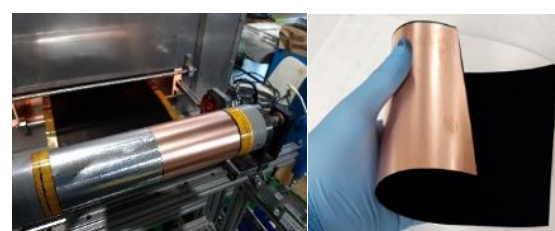
- Dispersion (sonication and mechanical stirring)
- Filtration (interchangeable)
- Washing (interchangeable)
- Drying – (inc. protective films, metallic foils etc), including sheet winding

Drying module design enables the introduction of additional material layers during processing. These can include:

- Metallic foils (copper, aluminium) for current collector functionality in supercapacitor electrode applications
- Polymeric veils – low density thin films for electrical isolation, composite impact toughening applications



**Figure 3:** Schematic of the new washing module with support membrane rollers



**Figure 4:** Multifunctional laminates – copper and aluminium electrode integration

### **Features of the Pilot Line**


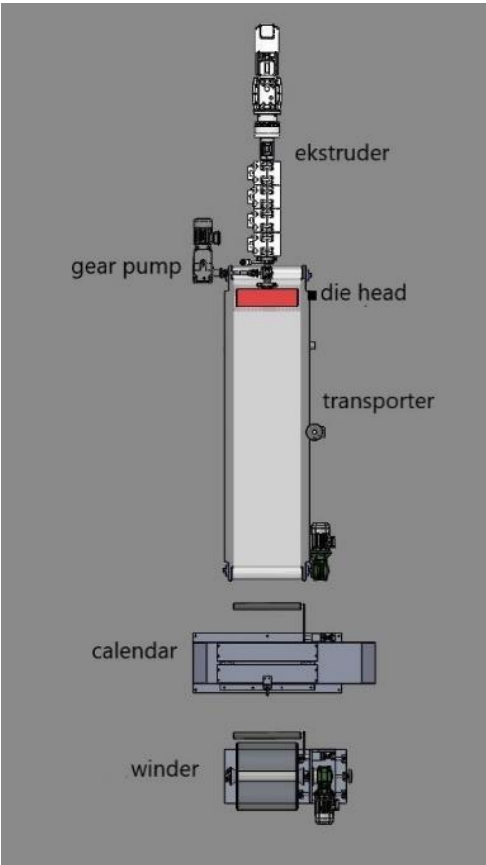

Input material	MWCNT liquid MB dispersion (aqueous) (also other water dispersible nanomaterials)
Output/Yield material	Current production capacity of 25m <sup>2</sup> /week (increasing to 50m <sup>2</sup> /week by 2021).
Production time	1 meter per hour
Energy consumption	2 kWh/hour
Name of the Process	Buckypaper pilot plant
Keywords of the process (max.5)	Dynamic vacuum filtration, IR drying chamber, multi-laminate integration, continuous process, ultrasonic dispersion
Keywords of the product (max.5)	Buckypaper, continuous MWCNT sheets, multifunctional, high electrical conductivity, nanomaterial
Technological offer	<ul style="list-style-type: none"> <li>• Development of innovative and multifunctional continuous MWCNT sheets using a scalable dynamic vacuum filtration process</li> <li>• Tailorable properties according to industrial application requirements including high electrical conductivity, large specific surface areas, low density, high flexibility, material compatibility (metals, polymers and redox)</li> <li>• Optimization of final product through variations of process parameters.</li> <li>• In-line incorporation of reinforcing material layers (polymeric veils for mechanical enhancement, metallic foils for electrode manufacture)</li> </ul>



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	<ul style="list-style-type: none"> <li>Small scale demonstrator for low volume dispersion and dynamic filtering trials (prior to full scale manufacturing on pilot line)</li> </ul>
Operating mode(s)	Automatic; semi-automatic; manual. Continuous, step, static.
Language(s)	English, Spanish, Basque

<b>Upgraded Technology</b>	
Parameters of the process	<ol style="list-style-type: none"> <li>1. Beckhoff software for process control, data acquisition and supervision</li> <li>2. New washing module (contaminants removal and BP property enhancement)</li> <li>3. Increased production capacity - supportive transport belt for filter membrane</li> <li>4. Filter vacuums up to -1 bar.</li> <li>5. Online QC sensors (pH, electrical conductivity, vacuum level).</li> </ol>
KPI achievement	<ol style="list-style-type: none"> <li>1. Continuous line for filtered CNT sheet production with increased output from 10 m<sup>2</sup>/week to 25 m<sup>2</sup> /week (50m<sup>2</sup>/week by 2021).</li> <li>2. Re-use of waste waters for manufacture of future CNT dispersions (5-10%)</li> <li>3. Addition of multifunctional material layers during buckypaper manufacture (flexible polymeric membranes, metallic foils)</li> <li>4. Monitoring and registering of quality-related parameters during processing via in-process sensors (pH, electrical conductivity, vacuum).</li> </ol>

	<b>Datasheet for pilot lines: DOPED VEILS</b>	
<b>OITB Member</b>	TMBK Partners	
<b>Name of Pilot line</b>	CNT doped veils	
<b>Number of the Pilot Line</b>	<b>PL5</b>	
<b>TRL of pilot line</b>	TRL 6	
<p><b><u>Description of the pilot line:</u></b></p> <p><u>Functional veils (non-woven fabric) produced in the one stage process.</u></p> <p>Fully automatic and continuous melt blown extrusion line. Design of this line is the result of TMBK's experience in the process of melt spinning a wide range of polymers and thermoplastic nanocomposites. In a one-stage process TMBK can produce a non-woven structure in the form of veils from the raw materials (neat or nano-doped) that are difficult to process by melt blowing. Depending on the raw material, we can produce nonwovens in the range of 10-100 gsm with a linear speed of 30-300 m/h.</p> <p><u>Modular design and flexibility.</u></p> <p>The line has been designed to minimize degradation of the raw materials as well as to increase output consistency and stability. A configurable die head allows for adapting to specific material requirements. The spin pack is easy to replace, therefore the length, diameter and shape of the nozzles can be varied.</p> <p>The line is composed of the following modules:</p> <ol style="list-style-type: none"> <li>1. Transportation &amp; Drying</li> <li>2. Plasticizing</li> <li>3. Forming &amp; Blowing</li> <li>4. Veil collection</li> <li>5. Calendaring &amp; winding</li> </ol> <p>The dryer adjusts the process airflow and its antistress function prevents over-drying. The height of the receiver can be adjusted widely and the non-woven veils can be pressed on the two-roll calendar to obtain the desired structure.</p> <p>An automated process minimizes the amount of plastic waste as well as the human contact with the veils during the production procedure.</p> <p>The quality of produced veils is controlled.</p>		<p><b><u>Pictures</u></b></p>  <p><b>Figure 5:.</b> Top view layout of the CNT-doped non-woven veils pilot line (blowers and dryer not included)</p>  <p><b>Figure 6:</b> Final product: non-woven veil on a reel.</p>

<b>Features of the Pilot Line</b>	
Input material	Polymer pellet/ Nanocomposite pellet, 20-25kg / bath, automatically refilled
Output/Yield material	Up to 1 kg/h, 200 m <sup>2</sup> /day (for 20 gsm veil)
Production time	line can run continuously 24/7
Energy consumption	15-20 kW h
Name of the Process	Meltblown
Keywords of the process (max.5)	Meltblown, extrusion, veil (non-woven fabric) formation, carbon nanotube filled polymers
Keywords of the product (max.5)	Thermoplastic nanocomposite, electrically conductive, CNT doped veils, nano filled nonwovens
Technological offer	<ul style="list-style-type: none"> <li>• Development of innovative and functional meltblown (nanofilled) non-woven veils for improved electrical conductivity and mechanical properties of composite structures.</li> <li>• Optimization of process parameters.</li> <li>• Production trials. Testing of the new raw materials and evaluation of processability by meltblown technique.</li> <li>• Preparation of the material compositions for meltblowing: homogenization and dilution from masterbatches on a twin-screw extruder based pelletizing line.</li> <li>• Filler/nanofiller dispersion and distribution improvements.</li> </ul>
Operating mode	Automatic
Languages	English, Polish

<b>Upgraded Technology</b>	
Parameters of the process	<p><u>Various possibilities for optimization</u> The possibilities for setting the process parameters at the line.</p> <ol style="list-style-type: none"> <li>1. Scada computer system as a user interface allowing for: <ul style="list-style-type: none"> <li>▪ line control from one point</li> <li>▪ setting main machine operating parameters: <ul style="list-style-type: none"> <li>○ extrusion temperature profile</li> <li>○ hot air temperature</li> <li>○ hot air speed</li> <li>○ gear pump rpm</li> <li>○ melt pressure</li> <li>○ receiver/calendar/winder rpm</li> <li>○ calendar pressure</li> <li>○ receiver suction blower pressure</li> </ul> </li> <li>▪ observation and visualization of machine operating parameters over time (temperature, pressure)</li> <li>▪ collecting historical information about the completed work cycles</li> <li>▪ creating a database of recipes (parameter sets)</li> </ul> </li> <li>2. Other externally adjustable process parameters: <ul style="list-style-type: none"> <li>▪ Die-collector-distance</li> <li>▪ Airgap/setback</li> <li>▪ Drying time</li> <li>▪ Drying temperature</li> </ul> </li> </ol>

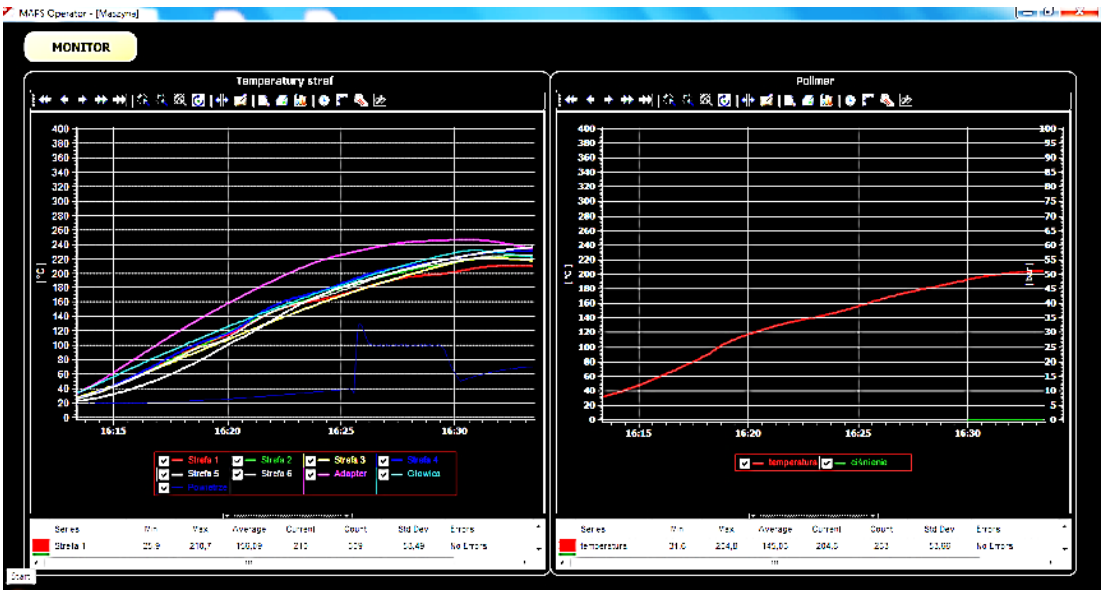


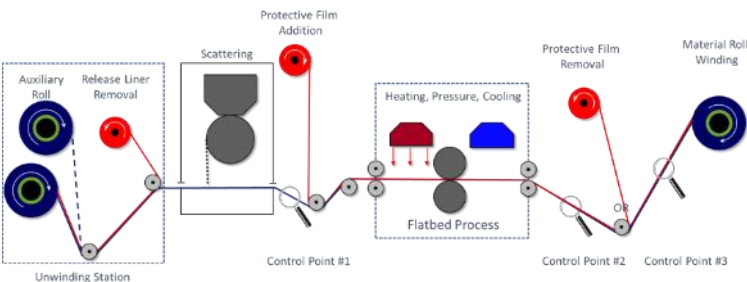


Figure 7: Process parameters visualization window.

KPI achievement

1. Continuous meltblown line for CNT-doped veil production.
2. Output of 200 sqm of 20gsm veils/day (previously: 50 sqm/day).
3. Polymer waste reduced to app. 20% (from 100% previously).
4. The veils are supplied in the form of a continuous, 0,3m wide non-woven fabric on a reel which makes it very practical for end user applications and easy to transport (previously: rectangular sheets 0,6 x 1,8m).
5. No need for interleaving paper (previously: interleaving paper between every sheet of veils was necessary).
6. Reduction of manual operations and human contact with the product during the manufacturing process (previously: the rectangular sheets had to be manually taken from the line).
7. Quality-related parameters monitored during process (previously: no monitoring).

	<b>Datasheet for pilot line: R2R</b>
<b>OITB Member</b>	Adamant Composites
<b>Name of Pilot line</b>	R2R (Roll-to-Roll) pilot line for the production of functionalized preregs
<b>Number of the Pilot Line</b>	<b>PL 6</b>
<b>TRL of pilot line</b>	TRL 8-9
<p><b>Description of the pilot line:</b></p> <p>To address Aerospace's ever-increasing demands for enhancements in the properties of the materials they use, Adamant Composites has proposed a method to enhance prepreg materials. These materials already exhibit extraordinary specific properties but based on this method, Adamant takes advantage of the unmatched electrical, thermal and impact properties of various nanomaterials by integrating them into prepreg materials via a novel manufacturing process. The effort to industrialize this process led to the implementation of the R2R pilot line using the production of functionalized preregs [Figure 8].</p> <p>Following the manufacturing process used in the R2R pilot line [Figure 9], a commercial prepreg in the form of a roll is positioned at the "Unwinding Station" (the beginning of the line). It is then fed through the R2R process to retrieve a roll of functionalized prepreg at the "Winding Station" (the end of the line). The "Unwinding Station" can handle up to two rolls, allowing for a semi-continuous production of several batches.</p> <p>The dopants have the form of a micro-powder, which safely encapsulate the nano-dopants. The dopants are deposited by a specific amount on the prepreg substrate using a scattering process. An in-house machine has been developed and built to serve the specific needs of the scattering process.</p>	<p><b>Pictures</b></p>  <p><b>Figure 8:</b> Adamant's R2R pilot line for the production of functionalized preregs (start of OASIS project).</p>  <p><b>Figure 9:</b> Schematic of the manufacturing process for the R2R Pilot Line.</p> <p>The process is a PLC based automated process and the <b>basic specifications</b> of the R2R pilot line are the following:</p> <ul style="list-style-type: none"> <li>• Roll width up to 600 mm</li> <li>• Roll outer diameter up to 600 mm</li> <li>• Roll weight max. 100 kg</li> <li>• Forward speed range: 0.2 – 9 m/min</li> <li>• Production rate up to 1000 sqm/day</li> <li>• All prepreg product formats and types as long as some dry fabrics can be processed</li> </ul>

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The micro-powder is firmly integrated into the prepreg with the help of the temperature and the pressure of the “Flatbed Laminator Process”. The final product is then inspected and wound either including (or excluding) an extra protective film.	
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<b>Features of the Pilot Line</b>	
Input material	a) All prepreg product formats and types as long as some dry fabrics up to 600mm wide. b) Up to 5-10 kg of micro-powder masterbatches that include nanomaterials.
Output/Yield material	Functionalized prepreg with enhanced Electrical, Thermal, Impact or other Tailored Properties.
Production time	Production rate can reach 1000 sqm/day.
Energy consumption	To be determined. Power is ~ 25kW.
Name of the Process	Prepreg nano-enabling.
Keywords of the process (max.5)	Roll-to-Roll, Nano-enabling, scattering, nano-modifying, functionalization.
Keywords of the product (max.5)	Prepreg, nano-enabled, textiles, nanocomposites, FXply™.
Technological offer	Now, composites can have targeted functionalities such as toughness, electrical conductivity etc. apart from the established performance of the prepreg material (e.g. strength, stiffness). The following functional formulations are available: <ul style="list-style-type: none"> <li>• FXply-EL: for improved electrical conductivity.</li> <li>• FXply-TF: for improved toughness.</li> <li>• FXply-TH: for improved thermal conductivity.</li> </ul>
Operating mode	Semi-Automatic, Automatic.
Language	English, Greek.

<b>Upgraded Technology</b>	
Parameters of the process	<p><b><u>Stage 1 Improvements</u></b></p> <ol style="list-style-type: none"> <li>1. Full automation &amp; PLC control of the line towards increasing the robustness &amp; the quality while decreasing the operational costs. <ul style="list-style-type: none"> <li>• New central PLC</li> <li>• Computer based control and monitoring of the production. Production statistics and database creation.</li> <li>• Encoder: exact meters measuring, pulses provided for control reasons.</li> <li>• Size 3 Inverters with advanced communication &amp; control abilities.</li> <li>• Automated tensioning system improving the manufacturing process and the end-product.</li> <li>• Automated liner removal in two positions.</li> <li>• Alignment of the line for good process flowability. (Stage 2)</li> <li>• Various Sensors to facilitate control and process monitoring. <ul style="list-style-type: none"> <li>○ Water tank temperature sensor</li> </ul> </li> </ul> </li> </ol>

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- Flowmeter
- 2 light sensors for detecting the presence & the absence of material
- Increased number of emergency buttons increasing safety.
- Improvements at the scattering and winding station.
- Automated barcode ordering.




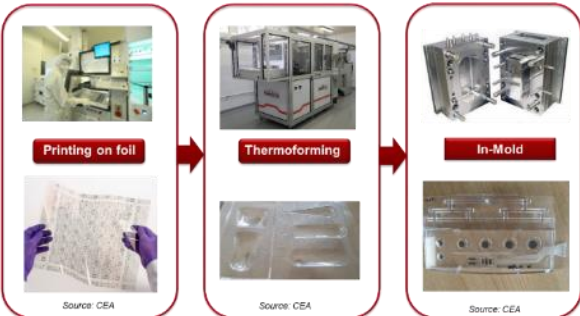
Figure 10: Stage 1 improvements for the R2R Pilot Line.


### Stage 2 Improvements

2. Quality Control Module based on Machine Vision increasing automation and the robustness in terms of quality.
  - Dedicated Platform.
  - Polarized Camera & Dedicated Lighting System.
  - Dedicated Software tuned to this manufacturing process.
3. Instead of an ISO8 room, the line is positioned at a dedicated space with industrial levels of cleanliness, where the conditions are monitored constantly leading to fully controlled manufacturing process.
  - Industrial Floor.
  - Space Separators.
  - Digital Thermometer and Hydrometer in communication with the line's operating system.
4. Further H&S instrumentation to be integrated and Improvements of the Scatterer towards increased levels of H&S (On Progress)



	 <p data-bbox="715 857 1260 884"><b>Figure 11:</b> Stage 2 improvements for the R2R Pilot Line.</p>
KPI achievement	<ul style="list-style-type: none"><li>• 50% Reduction of min personnel needed from 4 persons to 2 persons.</li><li>• 50% Reduction of time to initiate, handle and finish a production: 2h to 1h.</li><li>• 100% Automated Quality Control Process.</li><li>• 20% Increase of Manufacturing Robustness.</li><li>• 20% Increase of Product quality.</li></ul>

	<b>Datasheet for pilot line: PICTIC</b>
<b>OITB Member</b>	CEA
<b>Name of Pilot line</b>	PICTIC
<b>Number of the Pilot Line</b>	<b>PL 7</b>
<b>TRL of pilot line</b>	TRL 5
<p><b><u>Description of the pilot line:</u></b></p> <p>The CEA PICTIC pilot line is located in Grenoble, France. The platform is 600m<sup>2</sup> clean room class 10000 (Figure 12) working on Sheet-to-Sheet mode and is compatible with rigid and flexible substrate up to 320mm x 380mm (Figure 13). It develops printed electronic components which can be processed and integrated on the nanocomposite substrate. Accurate sensors (as temperature and hygrometry sensors) are matured by CEA in order to monitor crucial data all along the packaging. It includes a complete set of industrial coating, printing equipment and characterisation tools such as slot die, screen printer, inkjet, gravure printer, flexography tools, profilometer, and ellipsometer.</p> <p><b>Specific substrates are used in PICTIC platform as described below:</b></p> <p><b>Format:</b> 320 x 380 cm<sup>2</sup></p> <p><b>Substrates:</b> plastic (PEN, PC, PI, TPU), paper, metal foil, glass</p> <p><b>Inks:</b> conductive metal inks (Ag, C), conductive polymer inks (PEDOT...), dielectric formulations, organic semiconductor ink, EAP inks (PVDF-based)</p> <p>Integration of printed sensors in composite parts needs some critical steps as described in Figure 14. First, we used printing technologies on foils in order to elaborate specific components as temperature sensors, strain gauges...etc... In a second step, thermoforming is performed on printed foils. In a last step, printed sensors are over-moulded inside composite.</p>	<p><b><u>Picture</u></b></p>  <p><b>Figure 12:</b> PICTIC pilot line</p>  <p><b>Figure 13:</b> Printed electronic components on 320x380mm flexible substrate</p>  <p><b>Figure 14:</b> Integration of devices in composite by thermoforming and in-mold process</p>

<b>Features of the Pilot Line</b>	
Input material	Printed polymer sheets
Output/Yield material	One foil per minute (without clamping step)
Production time	-
Energy consumption	To be determined
Name of the Process	Thermoforming
Keywords of the process (max.5)	Thermoforming, melting point temperature, high pressure forming, IR camera temperature control
Keywords of the product (max.5)	Printed sheet, control of elongation rate
Technological offer	<p>Thermoforming equipment Niebling SAMK has been installed and started on PICTIC platform in order to improve the reproducibility of thermoforming steps (Figure 15).</p>  <p><b>Figure 15:</b> Thermoforming equipment, Niebling SAMK400, in PICTIC platform</p> <p>This specific up grading work is focused in this report on thermoforming technology. This technology is a key-process for printed sensors integration in composites components as described. Thermoforming process is mainly used to form printed sensors before their insertion in composite by over moulding process. Therefore, thermoforming process needs to follow two main rules:</p> <ul style="list-style-type: none"> <li>• To form the printed sheet with accurate dimensions, following the mold dimensions, in order to position it with good accuracy in the injection mould before over-moulding step</li> <li>• To preserve functionalities of printed components: thermoforming induces thermal and mechanical stresses on printed foils during forming.</li> </ul> <p>Thermoforming describes the process of heating a thermoplastic sheet (preferentially amorphous TP) to its softening point, stretching it over (positive form in the mold) or into (negative form in the mold) a single-sided mold, and holding it in place while it cools and solidifies into the desired shape. The softened sheet conforms to the shape of the mold and is held in place until it cools.</p> <p>Two thermoforming technologies are used in industry for stretching and holding sheets: vacuum and high pressure. Vacuum technology consists in applying depression under the sheet (between sheet and mold) to suck it around the mold. High pressure technology uses compressed air at high pressure directly applied over the sheet forcing it to press the mold.</p>

Thermoforming process takes place in three stages as described in Figure 16.

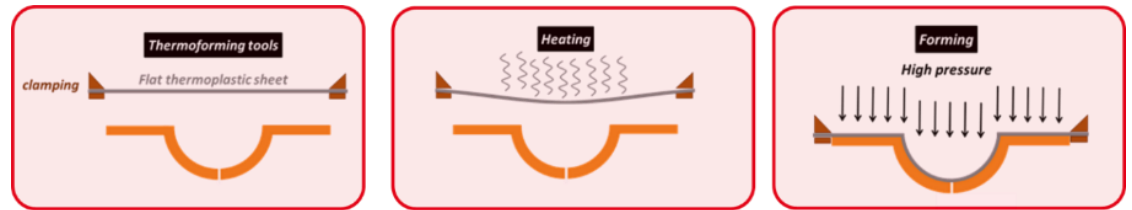


Figure 16: Main steps during thermoforming process

These three steps could generate deviations concerning forming dimension and functionalities of printed components on formed sheets:

- Clamping needs high accuracy of positioning in order to align component layout and mould CAO and also need clean foils in order to avoid dusts during forming steps;
- Heating has to be homogeneous and well controlled in order to achieve softening temperature whatever the position on the foil;
- Forming step is critical concerning dimensions on formed foil by controlling the application of high-pressure air: pressure, air temperature, mold temperature should be well managed in order to insure high reproducibility of formed foils and good behaviour of printed components on foils.

Operating mode	Automatic; semi-automatic and manual.
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Language	English; French
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### Upgraded Technology

Parameters of the process	<p>Upgrade of thermoforming technology has been performed during this task in order to improve this process reproducibility and control key parameters:</p> <ul style="list-style-type: none"> <li>• Temperature measurement on foil just after heating step and just before forming step</li> <li>• Use of high-pressure air to form sheets</li> <li>• Control of each parameter with human-machine interface with the possibility to use recipes for each kind of printed electronic sheet</li> </ul>
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KPI achievement	<p>Upgrade of thermoforming technology implemented in PICTIC platform presents some advantages for printed electronic process flow:</p> <ul style="list-style-type: none"> <li>• Human-machine interface gives the possibility to well control of process parameter through recipes.</li> <li>• Temperature of foils and pressure applied during forming step are, as an example, controlled with very good accuracy. This better control of key parameter leads to better reproducibility than standard forming technologies.</li> </ul>
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The main advantages are listed below:

- Very precise control of foils **temperature** with infrared camera temperature mapping for each foil see figure 2D temperature mapping
- Possibility to **localize** heating on foils (as example to avoid heating components)
- **High pressure forming** to insure a good conformability with mould (CAO)
- **High production rate of forming** (<30s cycle with 250µm thick PC foil)

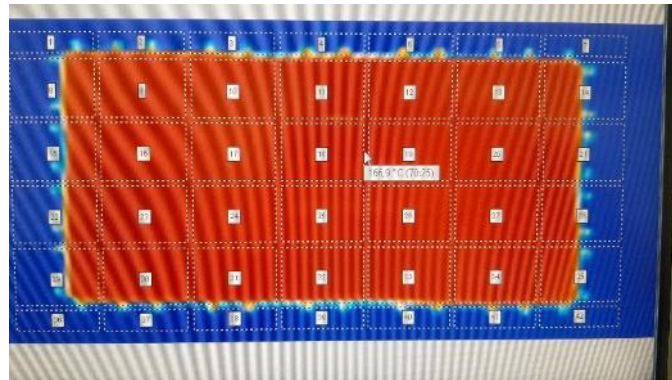



Figure 17: Example of 2D temperature mapping



	<b>Datasheet for pilot line: SIMPNANO</b>
<b>OITB Member</b>	Tecnalia
<b>Name of Pilot line</b>	SIMPANO. Nanoreinforced metallic alloy ingots
<b>Number of the Pilot Line</b>	<b>PL 8</b>
<b>TRL of pilot line</b>	TRL6

### Description of the pilot line

PL8 is the unit set up for production of nanoreinforced aluminium alloy ingots through the stir casting process. It is based on the upscaling and upgrading of the previous system where small batches of up to 3Kgs of nanoreinforced alloys were produced.

PL#8 is composed of five main elements:

1. A new electrical furnace with a capacity of 100 Kgs. for melting aluminium alloys has been upgraded to enable the production of nanoreinforced aluminium ingots through the stir casting process. (Figure 18). Furthermore, a new fume extraction system has been built that is located just over the crucible.
2. The mechanical stirrer unit where the elements in contact with the aluminium melt (graphite axis and impeller) have been redesigned to fit in the new furnace (Figure 19).
3. The ultrasound module, used to improve the dispersion of the nanoparticulates in the melt, has been upgraded to make it compatible with the 100 Kg. furnace. In the upgraded unit the ultrasound probe has been protected with a metallic structure that makes it possible to introduce the probe into the melt, thereby preventing the heating of the sonicator (Figure 20).
4. A new dosing system that can operate in a semiautomatic regime to feed the electrical furnace with pellets of nanoreinforced aluminium powders. These powders can be heated up to the required temperature (100°C) before their incorporation into the vortex created by the stirrer.
5. The ball milling unit. The design of the cylinders has been changed to improve safety aspects and airtightness of the closure mechanism. Furthermore, the system has also been upscaled fourfold to increase the production of the nanoreinforcements (aluminium powder). The unit is now capable of producing 800 g batches.

### Picture



**Figure 18:** Electrical furnace (100 Kgs.)



**Figure 19:** Mechanical stirring unit

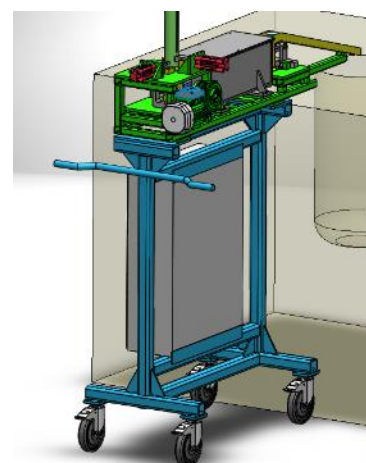


**Figure 20:** Ultrasound unit

The previous production unit consisted of a small electrical furnace with a production rate capacity of 3 kg/batch. Ultrasound waves and mechanical stirring were applied to achieve the required dispersion of nanoparticulates that were added manually.

The upgraded system is based on the same concept where nanoparticulates are introduced in the vortex created by a graphite impeller coupled to a mechanical stirrer. Ultrasound waves may be subsequently applied to disperse them within the melt but with a rate of production of up to 50-60 kg/batch.

In order to achieve this, an industrial electrical furnace is used as a melting unit in the pilot. The system has been upgraded with a controlled mechanical stirring unit, ultrasonic vibration unit and a new dosing and feeding system.



**Figure 21:** Drawing of the dosing and feeding module

### **Features of the Pilot Line**

Input material	Commercial Aluminium alloy ingots and Nanoparticulate/pure Al powder pellets
Output/Yield material	Nanoreinforced aluminium alloy ingots (0.1-0.2 wt.% of nanoreinforcements)
Production time	5-6 hours per batch of 50-60 Kgs.
Energy consumption	12 kW/h
Name of the Process	Stir casting process for the production of nanoreinforced aluminium alloy ingots
Keywords of the process (max.5)	Stir casting, dispersion, ultrasonic, furnace
Keywords of the product (max.5)	Nano particulates, aluminium, alloys, ingots
Technological offer	Development and production of nanoreinforced aluminium alloy ingots. Optimization of dispersion of nanoparticulates within the aluminium melt. Safe handling of nanoparticulates
Operating mode	Manual or semiautomatic
Language	English, Spanish, Basque.



<b>Upgraded Technology</b>	
Parameters of the process	<ol style="list-style-type: none"> <li>1. New dosing and feeding system for the drying and incorporation of pellets containing nanoparticulates.</li> <li>2. New graphite impeller system adapted to the new furnace dimensions.</li> <li>3. New design of cylinders for the ball milling process providing improved airtightness.</li> <li>4. New tilting furnace with capacity of production of 50-60 kg per batch.</li> </ol>
KPI achievement	<ol style="list-style-type: none"> <li>1. Production unit for the fabrication of nanoreinforced aluminium ingots in <b>batches of up to 50-60 kg.</b></li> <li>2. Production of nanoreinforced ingots with up to <b>0.1-0.2 wt.%</b> of nanoparticulates with good dispersion and repeatability thanks to the installation of the stirring unit and ultrasound system.</li> <li>3. Dosing system assuring the drying and controlled dosing of the pellets before their incorporation into the aluminium melt.</li> </ol>

## 4. Conclusions

These pilot lines are part of an ecosystem of 12 nanotechnology manufacturing pilot lines, providing nanomaterials, nano-intermediates, nano-enabled products and associated services for the development and commercialization of lightweight multifunctional products based on aluminium and polymer composites.

The up-graded performances for five pilot lines for the production of intermediate products with nanoscale features have successfully been performed for the following installations: PL4: BUCKYPAPER, PL5: CNT DOPED VEILS, PL6: R2R, PL7: PICTIC and PL8: SIMPNANO.

The pilot lines are fully operational and providing materials to six show case projects within OASIS. The PLs are also ready to start the development of the demonstrator cases within the OASIS Open Call, which will provide free access to this unique ecosystem to all successful applicants. This document can also be used as a public catalogue of technical information from the PLs for the OASIS Single Entry Point (SEP).

Additionally, this public catalogue of technical information reflects a state of the PLs from the OASIS project for dissemination purposes (WP7).

## 5. Degree of Progress

This deliverable is 100% complete

## 6. Dissemination Level

This deliverable is public.

## 7. Appendix

The presentation of the upgrade/upscale of the PLs for the manufacturing of the nano-enabled products is attached in that section.



### PL4. SELF-SUPPORTING CONTINUOUS SHEETS OF ENTANGLED MWCNTs “BUCKYPAPERS”.



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#### TECHNOLOGICAL OFFER

- Manufacture of continuous CNT sheets using a dynamic vacuum filtration process
- Properties tailored to industrial application requirements
- Scalable production

#### COMPETITIVE PRODUCTS

- High electrical conductivity
- Large specific surface areas
- Low density
- High flexibility
- Material compatibility (metals, polymers and redox)

#### APPLICATIONS





#### Features of the Pilot Line




#### UPGRADED TECHNOLOGY

- Beckhoff software for process control, data acquisition and supervision
- New washing module (contaminants removal and BP property enhancement)
- Increased production capacity - supportive transport belt for filter membrane
- Filter vacuums up to -1 bar.
- Online QC sensors (pH, electrical conductivity, vacuum level).






Open access single entry point for scale-up of innovative Smart lightweight composite materials and components  
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## PL5. CNT DOPED VEILS

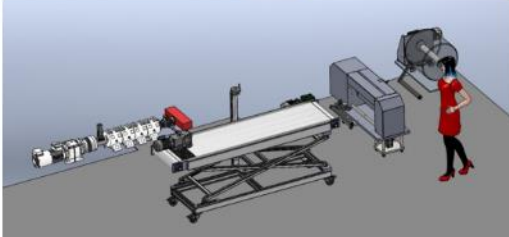


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### TECHNOLOGICAL OFFER

The Pilot Line can be used to:

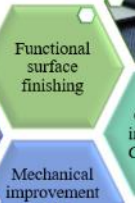
- Development of innovative and functional meltblown veils
- Various possibilities for the optimization
- Production trials with „difficult to process” polymers/nanocomposites




### APPLICATIONS



Functional surface finishing



Mechanical improvement of the CFRP/GFRP

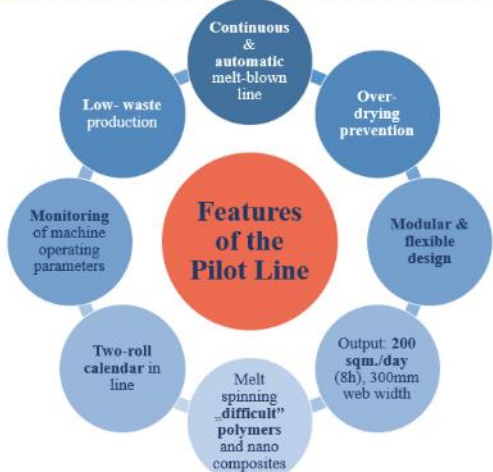


Conductive interlayers in CFRP/GFRP

### COMPETITIVE PRODUCTS


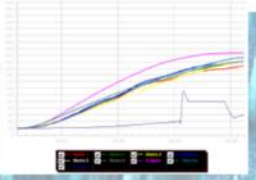
- Advanced nonwovens
- Light-weight structures
- Reduced electrical resistivity
- Compatibility with end user resins
- Wide range of areal weight
- Easy & safe way to implement nanofillers to final product

### Features of the Pilot Line



### UPGRADED TECHNOLOGY

- High output consistency – improved proces stability
- Reduced moisture absorption
- Limited polymer degradation, upgraded die geometry
- Continuous production with significantly less waste and a reduction in manual handling of the nonwoven fabric
- On-line quality control system



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**PL6. R2R PILOT LINE FOR THE PRODUCTION OF FUNCTIONALIZED PREPREGS**



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**TECHNOLOGICAL OFFER**

FXply™ Prepreg Technology enables conventional prepregs to deliver additional functionalities such as:

- Toughness
- Electrical Conductivity
- Thermal Conductivity

**COMPETITIVE PRODUCTS**

- Ultralight materials.
- Composite materials with enhanced properties.
- Tailored composite products.

**APPLICATIONS**





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**UPGRADED PILOT LINE**

- Full automated and traceable production with increased robustness & quality while having decreased operational costs.
- Improved Quality Control via Camera Visual Inspection (Machine Vision).
- Fully controlled manufacturing process through dedicated production space with industrial levels of cleanliness, where the conditions are monitored constantly.
- Improved H&S in terms of H&S instrumentation & Scatterer enhancements.








## PL7. PICTIC PLATFORM





### UPGRADED TECHNOLOGY

Integration of devices in composites by thermoforming and over-moulding process

  
**Printing on foil**  
Source: CEA

  
**Thermoforming**  
Source: CEA

  
**Over-Moulding**  
Source: CEA

↓ Thermoforming upgrading

  
Loading stage – 320x380mm foils

  
Forming stage → high pressure

  
Cleaning ramp

  
Heating stage (on both sides)

  
IR camera temperature control

### TECHNOLOGICAL OFFER

The Pilot Line can be used to:

- Develop innovative sensors
- Optimize semi conductive organic devices
- Integrate printed electronics in devices

### COMPETITIVE PRODUCTS

- Structural electronics
- Electroactive sensors and actuators
- Flexible Hybrid electronics
- Integrated sensors in components



CEA KEY ENABLING TECHNOLOGIES

Electro-active Sensors & Actuators → **CEA Printed Electronics** → Semi-Conducting Organic Devices

Structural Electronics

Flexible Hybrid Electronics – Plastonics – Stretchable Electronics

Plastic-Paper-Glass-Steel



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- Human-machine interface : control unit with recipes. Process parameter and reproducibility,
- Very precise control of foils **temperature** with infrared camera temperature mapping for each foil: Possibility to **localize** heating on foils (as example to avoid heating components)
- High pressure forming** to ensure good mould conformability (CAO)
- High production rate of forming** (<30s cycle with 250µm thick PC foil)



**Inspiring Business**

**PL8. SIMPNANO.ALUMINIUM ALLOYS REINFORCED WITH DISPERSED NANOREINFORCEMENTS**



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**TECHNOLOGICAL OFFER**

The Pilot Line can be used to:

- Develop innovative materials
- Optimize dispersion of nanoreinforcements.
- Production of large batches in a controlled and repeatable conditions.

**COMPETITIVE PRODUCTS**

- Light metallic materials.
- Good performance/cost ratio.
- Capability of production of a large range of aluminium alloys and nanoreinforcements.

**APPLICATIONS**







**Features of the Pilot Line**

**UPGRADED TECHNOLOGY**

- Semiautomatic nanoparticulate drying and dosing system.
- Mechanical stirring and ultrasonic modules to increase dispersion of nanoparticulates
- Safe handling of nanopowders and melt aluminium.



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