PROJECT FINAL REPORT

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[1] Usually the contact person of the coordinator as specified in Art.1 of the grant agreement.
[2] The home page of the website should contain the generic European flag and the FP7 logo which are available in electronic format at the Europa website (logo of the European flag: http://europa.eu/abc/symbols/emblem/index_en.htm;
logo of the 7th FP: http://ec.europa.eu/research/fp7/index_en.cfm?pg=logos).
The area of activity of the project should also be mentioned.

Content

1 Executive Summary

Europe identified within its innovation priorities the necessity to scale up the most promising material production technology to pilot production level in order to progress towards industrial manufacturing using cutting edge materials in novel new processes. Manufacturing facilities for the production of advance and nano structured materials by high energy ball milling (HEBM) suffers from low productivity and high cost which is a key barrier for its application in the wider commercial market sectors. PilotManu addressed this manufacturing gap by scaling up and building a new pilot production line for the production of nanostructured and advance materials, based on the consortium's IPR and expertise. PilotManu cuts across many technology areas including Nanotechnology Advanced Manufacturing Systems and Advanced Materials and has helped to transform the European manufacturing sector.

THE OPPORTUNITY:
PilotManu demonstrated the beneficial use of novel nanostructured materials to create new business opportunities for the consortium's small and medium enterprises (SMEs) which represents the entire supply chain in bringing new products and technologies to market. The business opportunities encompassed the production of pilot volumes of cost-effective nanostructured materials used in three product lines: coatings obtained by thermal spraying, polymer and metal composites for rapid prototyping and additive micro manufacturing components, and abrasive tools by sintering. The new pilot plant developed by PilotManu enabled increased productivity while lowering production costs which was key to allow the adoption of these materials into new innovative applications.

THE PILOT LINE:
PilotManu produced nanostructured powders based on the HEBM technology. The lead partner MBN has developed proprietary knowledge over the years based on a small scale production facility. This facility has now been optimized by a new design and scaled-up to enable pilot scale volume production with fine and homogeneous chemical distribution of elements and "ultrafine" crystalline structure. The nanoscale features of these powders allow significant improvement of material performance such as physical-chemical-mechanical properties compared against bulk scale materials. The new improved pilot line reduces the power consumption by 50% and scales up the production process by a factor of ten.

THE NANOSTRUCTURED POWDERS:
The nanostructured powders enable new properties to be harnessed in a variety of applications compared to larger micron scale similar composition powders allowing the supply of more affordable material for industry consumption. This has enabled the production of a wide range of novel systems from polymer nanocomposites, to ceramic metal composites and nanostructured metal alloys.

THE PRODUCT LINES:
The scale-up of the machine will be validated by the adoption of the materials in 3 main industrial product-lines: conventional and advanced sintering, coatings by cold gas spraying (CGS) and high velocity air fuel (HVAF) and additive manufacturing by direct metal laser sintering (DLS) and selective laser sintering (SLS). The product lines have been selected to demonstrate the versatility of the HEBM powder manufacturing technology at industrial level.
Novel metallic composite powders prepared by HEBM are used into a new series of stone working and cutting tools. The improvement of current hot pressing and the development of the free-sintering technique allows to reduce the production costs in terms of manufacturing time and energy consumed, achieving in parallel an improvement of the cutting performance and the tool life. Advanced sintering technique as Spark Plasma Sintering (SPS) allows to produce nanostructured components used in hot conditions with outstanding properties compared to current solutions. Besides this, the absence of toxic elements as cobalt in these products represents a further added commercial value.
About second product line (coatings), the aim is to validate CGS and HVAF technology to produce nanostructured coatings from powder produced by HEBM. CGS is used to deposit coatings on industrial components in automotive sector with outstanding mechanical properties and high deposition efficiency and surface quality. HVAF spraying has been investigated for WC-Co-Cr powders to achieve coatings with high wear and corrosion resistance comparable to current commercial solutions sprayed by HVOF. Regarding product line of additive manufacturing the newly developed mechanically alloyed powder prepared by HEBM are used in micro components fabrication by direct laser sintering. The aim is to define the laser sintering parameters for novel materials and to validate this technique for powders resulted from HEBM process. The SLS will be investigated with the objected to process innovative polypropylene based powders for rapid manufacturing of customer goods components, expanding the range of application for SLS to new cost effective plastic material.

Economic assessment of the business cases developed in the product lines have been performed, through the determination of expected benefits of the products/technology selected by the partners. The competitive landscape and the expected route to market for the three product lines have been carefully evaluated and, for each product/technology business plans for the next years have been outlined by the industrial partners. It has been estimated that PilotManu HEBM powder production plant will allow MBN a pilot production starting from 2018 and, foreseeing the installation of two new PilotManu production plants, to increase the annual production after five years. This will generate an estimated annual turnover of 10 million euros resulted from the selling of the manufactured powders. Furthermore the products and technologies developed by the other industrial SMEs during PilotManu project will allow to generate a substantial turnover growth that, added to MBN's turnover, is expected to reach after 5-7years up to annual 35-40 million.

2 Project Context and Main Objectives

PilotManu project aims to upscale the current High Energy Ball Milling (HEBM) facilities into a powder manufacturing pilot line by further developing the existing MBN proprietary results related to mechanical alloying technology and to innovative powder materials for different applications. The up-scaled machine will be exploited to produce powder materials suitable for sintering, spraying technologies and additive manufacturing.
Current HEBM technology facility produces powders with enhanced properties for several applications but it presents some limits in terms of cost effectiveness and productivity for commercial exploitation: these limitations affect the industrial market by slowing the request of innovative materials with enhanced properties.
PilotManu project will extend the state of HEBM technology thanks to the scale-up of the current production in order to:

- Produce innovative and cost-effective powder nanostructured materials;
- Increase the HEBM productivity of such innovative materials by ten times;
- Integrate materials produced by HEBM in transformation process in three product lines: materials for sintering of diamond grinding tools, materials for cold and thermal spraying and materials for additive manufacturing

PROJECT OBJECTIVES:
The project objectives are as follows:

Objective 1: Design of pilot scale material production technology
The target is the realization of a High Energy Ball Milling (HEBM) pilot scale machine with average batch productivity of ten times compared to current HEBM facilities with a reduced energy consumption of 50%: the pilot machine is based on a design selected after the evaluation of the effectiveness of two different concepts.
The overall safe design includes also all the technological aspects involved in powder processing machine according the state of the art EC standard and regulation for mechanical and electronic parts and to assure an integrated safety management of the pilot machine.

Objective 2: Definition of business cases for the use of IPR protected materials
The project started from the definitions of materials developed by HEBM technology for all the different manufacturing processes to demonstrate the platform productivity. Three materials classes linked to the HEBM technology will demonstrate the platform productivity through selected end user applications in the three product lines: abrasive tools, coatings by thermal spraying and additive manufacturing materials.
Other business cases, not necessarily linked, could be exploit the results of the pilot line in strategic market sectors for the consortium such as high temperature materials for fossil energy and energy harvesting materials.

Objective 3: Scale up to a pilot HEBM plant
The target is the realization of pilot scale manufacturing plant that includes the realization of the HEBM PilotManu machine and the ancillary devices.
The realization of the HEBM machine is based on the output coming from the design and modelling concept activity by choosing pure elastic dumping or combined elastic and compensation dynamics principle with the objective to upscale the production batch by 10 times. The realization of the HEBM plant includes:
- Realization of high volume milling vial for production able to work in controlled atmosphere with liquid cooling systems and fittings.
- Foundation, basement and machine vibration dumping;
- Employment of lightweight structures for the frame assembly and components of the machine, elastic and dynamic damping system;
- Electric systems, powder supplies and controls;
- Safe powder management system for pneumatic powder feeding and unloading;
- The integration in the of the powder processing treatments in the production route - classifying and rounding- able to select and refine powders on the suitable shape and size for the selected applications;
- Testing processing performance of a set of materials structures: metal composite, CerMet and Ni based high temperature alloys.
Further objective is the benchmarking against the current technology performed in order to verify the powder production process effectiveness.

Objective 4: Integration of HEBM materials into three product lines
Aim is to develop three main product lines (sintering, coatings and additive manufacturing) thanks to the upscale of HEBM pilot scale plant.
The SMEs and industrial partners in the consortium will cover the manufacturing chain from pilot powder production to
Further aim is to tune the pilot scale production for the thermal spraying material, for composites in abrasive tool and powder materials and for additive manufacturing during the development, assisted by the characterization and quality control of the products during the scale-up, performed according to consolidate protocols available at the companies' facilities.

Objective 5: Technological validation of the pilot line
Aim is to perform the technological validation at different levels: HEBM machine, materials and products.
- HEBM machine: validation to confirm that plant components and systems meet the technical targets defined during the modelling under industrial operating conditions.
- Materials and products: validation of the materials produced with pilot plant against the same materials produced by the actual technological facility; related products benchmarked with the products as performed during the respective development of applications with the current small scale HEBM.
In parallel, this objective includes also the energy evaluation of energy for each material and the implementation of a robust engineering methodology and on line/at line characterization/ monitoring systems to enhance product quality and batch-to-batch consistency of material.

Objective 6: Business plan
Aim is to develop a clear picture of the actual product benefits and cost bases of selected application cases, to enable refinement of Target Market Opportunities and pricings. Based upon a survey of each market, target lead adopter customers will be identified, contacted and encouraged to validate the perceived product benefit of each products and, ideally, confirm the postulated pricing models and evaluating the best method of market introduction and penetration in the case of each product.
Based upon confirmed product benefits, pricings, market opportunities and routes to market, the final objective of the project is to define a business plan each of the application cases, considering the penetration over a ten-year period, together with cumulative profit, having allowed for costs of goods sold plus any investment needed to deliver commercialization.

3 Science and Technology Results

3.1 HEBM Pilot machine and powders

MBN is a SME that is manufacturing nanostructured powder materials for several applications (from abrasive diamond tools to biomedical) through a proprietary High Energy Ball Milling (HEBM) process technology; during PilotManu project, MBN designed and assembled a pilot scale HEBM machine able to deliver larger powder batches (up to ten times more) compared to current facilities; the technology was then successfully validated through the three product lines of the project.

3.1.1 Description of the result and key achievements

MBN initially considered two different models for the HEBM pilot machine: both were analyzed with evaluation of the benefits for each model. The second model, based on a concept adopting a principle of oscillating masses equilibration, was finally selected after comparison with the other model based on the current oscillating machine concept by elastic dumping.
Second concept showed also best trade off characteristics: it guarantees the balance of masses, loads and forces during the HEBM process and the expected costs for realization and maintenance are considerably lower than the first concept.

MBN then proceeded with the construction of the Pilot machine following the subsequent steps:
- Detailed design of the machine were realized and stress-fatigue simulations were performed
- All machine components were defined: main mechanical components were manufactured following the executive design and commercial components were ordered
- Critical components were tested in a small-scale prototype machine based on the same Pilot concept and confirmed their reliability and durability before the assembling in PilotManu machine
- Fe-based material was milled in the prototype machine: the PilotManu HEBM process was compared and the obtained material was confirmed to be in line which the material resulted from current HEBM process
- Pilot machine was assembled and electrical parts and control panel were installed and checked
- Design and manufacturing of reaction vials based on new concept to increase the processable volume of powders per batch

Consequently, MBN proceed with the installation of the auxiliary system: sound proof cabin, safety system with integrated smoke and powder loss detectors, powder load/unload automated system and air extraction.
The HEBM pilot machine achieved an average batch productivity of ten times the current small scale facility, reducing current energy consumption of more than 50% per Kg of powder.

In order to validate the HEBM pilot machine, processing tests have been performed and compared with same powder produced by current HEBM facilities.
Three powders systems have been selected as representative materials for physical-chemical point of view and for product applications in the three project product lines:

- FeCu alloy and FeP based alloy processed for grinding tool application
- Ti-WC and WCCoCrAl Cermets for thermal spraying
- TiAlNb for additive manufacturing

All the materials produced by PilotManu were compared with the same powder variants produced with current HEBM facilities: the properties were confirmed to be comparable with the current product. In parallel, reliability of the mechanical and electrical components were tested and confirmed among several subsequent production sessions.

At the end of the project, the Pilot machine technology has been completely and successfully validated: several powder batches have been produced, confirming the process reliability and reproducibility and the expected reduction of operational costs

3.1.2 Progress beyond the state of the art

High Energy Ball Milling process (HEBM) is able to produce advanced materials and nanophased materials with great potential for a number of manufacturing processes, however, there is a "valley of death" between the possibilities of the HEBM technology and the low amount of already exploited applications. To progress towards industrial manufacturing, the technology was up-scaled to pilot production level: the technological facilities that have been used so far have a small scale production capacity that have limited the widespread use of HEBM powders. PilotManu project allowed to upscale the production in order to have sufficient volumes of powder batches and thus to bring materials in effective industrial deployment.

3.2 Diamond cutting tools by hot pressing and free sintering

DIAM is a SME specialized in manufacturing diamond abrasive tools with high degree of flexibility for satisfying both large and small volume production requirements. Thanks to the particular attention puts in R&D department, DIAM reveals an inclination to new technologies and material applications that was possible to further develop during PilotManu project.

3.2.1 Description of the result, key achievements and progress beyond the state of the art

DIAM optimized free sintering technique to produce cutting rims for medium size disks: due to excellent results achieved in terms of sinterability and cutting performance, DIAM selected this production route to manufacture the rims, using FeNiCuP powder provided by MBN.
For cutting sectors for big disks (3500 mm diameter) DIAM proceeded with the classic hot pressing technique using FeCuSn composition. A specific Fe-based composition was selected as best binder for beads (cutting wires), then DIAM proceed with pilot production by free sintering. For all selected rims and beads, the production costs were notably reduced compared to current benchmarks. The selected products were qualified by testing in operative environments among end users: cutting speed and tool life were evaluated against the currently used benchmarks.

Cutting sectors for discs:

It was obserseved a notable improvement in the tool life of both cutting segments as consequence of the optimization of the sintering conditions and adding of Nickel: PilotManu products achieved a good cutting speed and tool life equal to benchmark, confirmed by tests in operative environment.

Figure: Cutting disc (1200 mm) in operative environment

Also for the rims for big disks the cutting speed is higher and the tool life is lower: it is important to note that for cutting operations with discs the operative costs are more affected by the cutting speed rate and consequently by the cutting speed; due to this, also considering the absence of toxic elements as Cobalt, the PilotManu products can be considered really competitive in the market considering also the low price of binder powder.

Cutting beads for wires:

Several optimization tests of the one-step free sintering technique were performed: with PilotManu powder, the sintering of the beads and their brazing to the steel wire are made in one single step, allowing a notable cost and energy saving compared to the competitor benchmark powder, that need two separated steps of sintering and brazing to the wire.
Beads were tested in operative environment achieving very good results in terms of cutting speed and acceptable tool life.

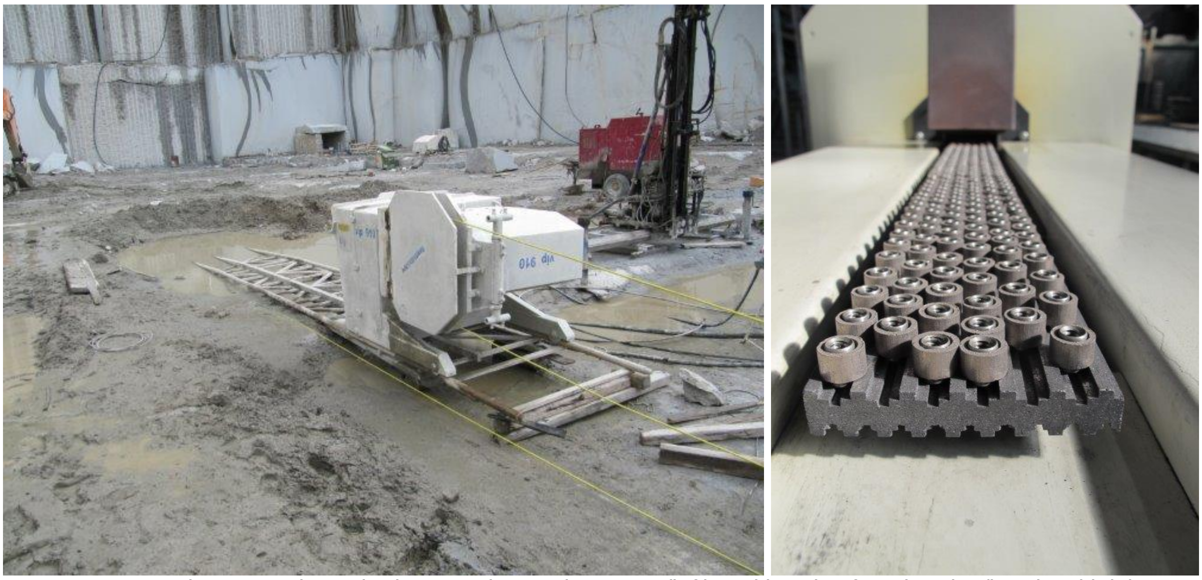

Figure: Cutting wire in operative environment (left) and beads after sintering/brazing (right)

For this product, the lower tool life of PilotManu wire is offset by the higher cut speed achieved by PilotManu, besides lower production cost due to sintering process in one single step and “green” composition without Cobalt usually in the range of 10-20% wt for current commercial product.

3.3 Components by advanced sintering techniques (SPS)

Metal Forming Institute (INOP) is an innovative, multidisciplined, industrially oriented research and development centre: INOP is currently developing SPS metal-ceramic composites and solid lubricant coatings with ultralow friction coefficient for forging tool and sliding components and, during PilotManu project, tested HEBM materials in the production of advanced components by such technique.

3.3.1 Description of the result, key achievements and progress beyond the state of the art

INOP developed spark plasma sintering technology of manufacturing: (i) diamond cutting sectors, (ii) sliding bearing sleeves and (iii) resistance spot weld tips.

(i) INOP tested two kinds of Fe-based powders: (i) FeCu and (ii) FeCuSn for spark plasma sintering the diamond cutting sectors. The influence of SPS parameters (sintering temperature and holding time) on microstructure and physical and mechanical properties were investigated. For producing the final shape diamond cutting sectors the new complex shape graphite tools for SPS technology were design and fabricated. The main achieved results are:

- Stable SPS process of diamond cutting sectors sintering
- Uniform distribution of Diamond particles in the binder matrix
- Preservation of a sharp angels of Diamond particles that is believed to indicate about absence of graphitization reactions at the Diamond-matrix interface,
- Good mechanical properties of SPSed diamond cutting sectors,
- Exploitation tests of diamond cutting discs show the possibility to use it for cutting hard materials.

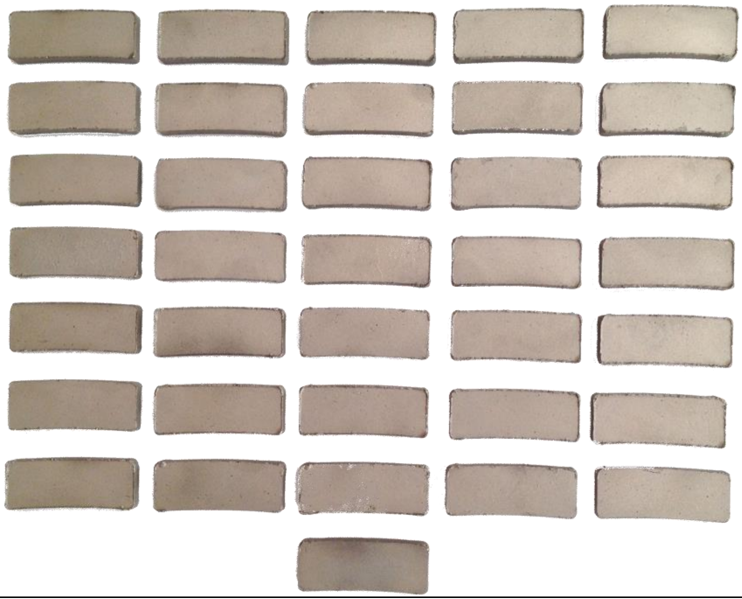

Figure: Diamond cutting sectors made from Fe-based HEBM powder fabricated by spark plasma sintering

(ii) INOP developed SPS process of new Ti-based powders assigned to advanced components characterized by low density and high hardness, fracture toughness and low wear at high temperature. A few types of Ti-SiC powder were tested. The influence of sintering temperature, holding time, compaction pressure and heating rate on microstructural evolution, phase transformation and physical and mechanical properties were investigated. For high pressure SPS of Ti-SiC powders, the special design of graphite tools were done and the die-in-die system was applied. The main results of SPS of Ti-SiC are:

- The basic structure formation processes of SPS of Ti-Si-C composite materials such as self-high temperature synthesis, Ti-Si, TiC and MAX phases (Ti_3SiC_2) reactions are found, characterized and controlled to achieve the high exploitation properties of the Ti-Si-C components.
- The Ti-Si-C nanostructured powder composite sliding bearing sleeves for high temperature application are made and tested. The results of high temperature friction tests reveal about achievement of self-lubricating regime at high temperatures. The great effect of wear rate diminishing is obtained at 700°C due to preservation of interaction of microasperities at sliding interface because of self-lubricating effect found.

Figure: Sliding bearing sleeve made from Ti-based HEBM powder fabricated by spark plasma sintering

(iii) INOP developed the new combination of SPS and KOBO cold extrusion technology of Cu-Al_2O_3 powders. It allows to utilize the nano-structuring effects of Cu-based powder mechanical alloying by HEBM to increase a longevity of the tips with compared electrical conductivity. The KOBO extrusion differs from the conventional extrusion process by additionally implemented cyclic torsion of metal, resulting from a forced reciprocal rotations of adequately configured die. A cyclic change of deformation path leads to shear banding and generates overbalance concentration of point defects, typically in the form of nano-dimensional clusters of interstitial atoms. It allows extrusion of Cu-Al_2O_3 composites at ambient temperature. A few types of Cu-Al_2O_3 powder were tested. The influence of sintering temperature on microstructure and physical and mechanical properties were investigated. The main results of SPS and KOBO extrusion are:

- High mechanical properties and electrical conductivity,
- High resistance to recrystallization due to decoration of grain boundaries with Al2O3 nanoparticles.

Figure: Resistance spot weld tips made from Cu-based HEBM powder fabricated by spark plasma sintering and KOBO cold extrusion technology

3.4 Coatings by Cold gas spraying

IMP is a SME specialized in R&D of cold spray applications; the company is now producing a new innovative high pressure cold spray unit as well as new components, nozzles and spare parts for cold gas spray equipment.
During PilotManu project, IMP found different cold spray coating applications in three industrial fields and utilizing the innovative HEBM powders:

Oil & Gas industry:

- Ti-WC hard coatings for bearing shafts for pumps and other industrial bearings
- Ti-WC hard coatings for pistons for piston pumps

Printing roll manufacturing industry:

- Ti-WC hard coatings for printing rolls

Automotive industry:

- Fe-based coatings, for automotive combustion engine cylinder bore ID coatings

3.4.1 Description of the result and key achievements

The Ti-WC coatings for bearing shafts were functionally tested in real industrial conditions and after 24 months in operation qualified as a hard coating suitable for the application for industrial bearings. The results of the piston demonstrator showed, that the innovative Ti-WC hard coating is suitable for the application as a wear resistance coating for pistons of piston pumps in the Oil & Gas industry. The Ti-WC hard coatings for printing rolls were functionally tested by a printing roll manufacturer, and qualified as a new innovative material with high potential for this industrial segment.

Figure: From left to right: Ti-WC coated bearing shaft, Ti-WC coated piston, Ti-WC coated printing roll and Fe-based alloy ID coated engine block

In case of the cold sprayed ID coating of combustion engine's cylinder bores, except the Fe-based HEBM powders, the ID coating device was re-designed to implement the nozzle water cooling system, keeping the nozzle wall cold, preventing the softened powder particles to stick on it, causing nozzle clogging. To be possible to deposit Fe-based coatings at high deposition efficiencies, the ID coating device was improved implementing better heat insulation, to prevent losing heat between the heater and the cold spray nozzle. The better heat insulation led to higher kinetic energy of the particles, hence to increased deposition efficiency. With the new ID coating device, the ID cold spray coating using the innovative Fe-based HEBM materials were demonstrated on real cylinder block.

3.4.2 Progress beyond the state of the art

The Ti-WC hard coatings deposited by cold spray, compared to thermal sprayed hard coatings enable to build-up up to 4mm thick coatings, giving an advantage of longer component lifetime and shorter service periods. Except that, the cold spray process is a solid state low temperature process giving an advantage to deposit hard coating to heat sensitive substrates.

The Fe-based coatings together with the ID coating device, developed for the combustion engine cylinder bore inner deposition, combines the advantages of the cold spray process and the innovative mechanically alloyed materials, which leads to specific coating properties, at high productivity, high deposition efficiencies. Recent thermal spray technologies used for the inner cylinder coatings in the automotive industry have to overcome several technical limitations, due to the character of the thermal process itself. Cold gas spray deposition offers many advantages compared to other thermal spray techniques, which can lead to better material utilization, cost reduction and process simplification preventing surface roughening, preheating/cooling of the engine blocks and complex overspray dust collection.

3.5 Coatings by High Velocity Air fuel spraying

PUT is a SME specialized in the manufacturing of high quality mechanical components including thermal sprayed coatings. During PilotManu, the company investigated the application of HEBM powders in High Velocity Air Fuel (HVAF) technology, that was validated through the production of industrial application cases.

3.5.1 Description of the result and key achievements

Putzier has thermally sprayed protective coatings for new advanced coating materials developed by PilotManu partners. Particular focus was set on the WC-based cermets. Corresponding powders produced by HEBM exhibit an irregular, not spherical particle shape and a size distribution adapted to meet the HVAF specification. Their apparent density is comparable to the agglomerated-sintered benchmark powder. All powders can be fed by the standard HVAF powder feeder and are therefore suitable for production purposes.

Benchmark WC-Co-Cr 86-10-4 | **PilotManu powder: WC-Co-Cr-Al 85-10-4-1**

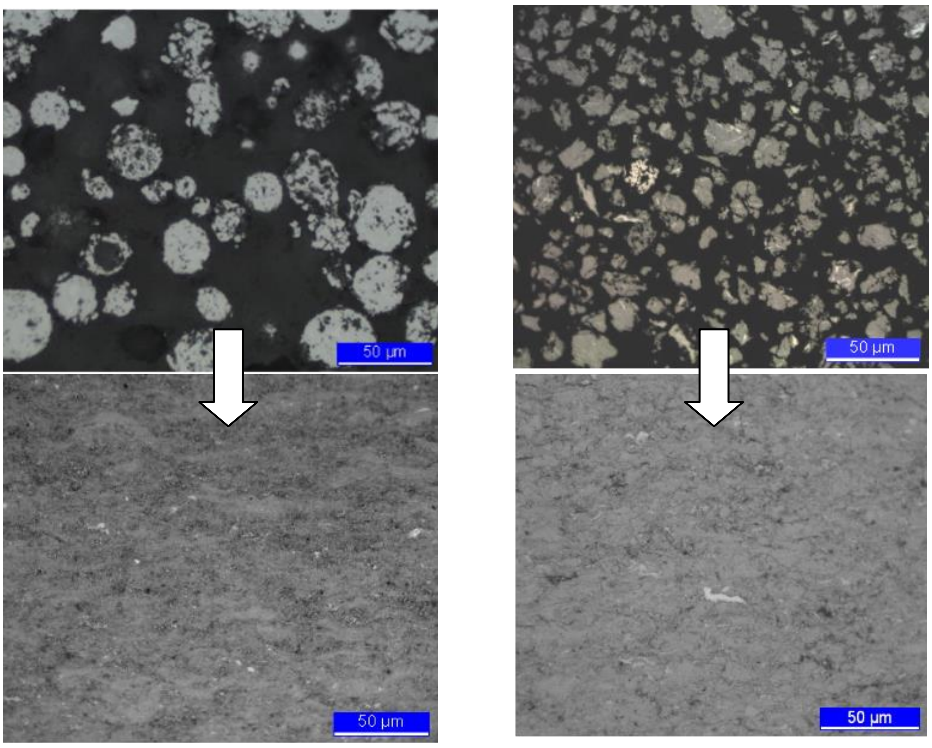

Figure: Microstructure of coatings sprayed with the agglomerated-sintered benchmark powder and the HEBM PilotManu powder

Coatings' properties were intensively analysed in laboratory tests, for example hardness, wear and corrosion testing. In the first part of the project, powders were produced in the small-scale HEBM machine. In the second part of the project, PilotManu powders were produced in the newly developed HEBM pilot machine. All tests of the coatings were repeated

and it was found that the coating properties are the same for both powder types. This means that the upscaling of the laboratory-scaled HEBM to a mass-production HEBM process was successful.
For the next step the optimized HVAF WC-Co-Cr-Al coatings were sprayed onto various components and tested in several industrial tests in customer's facilities. The figure below gives one example for the application of PilotManu coatings, rods for hydraulic cylinders.

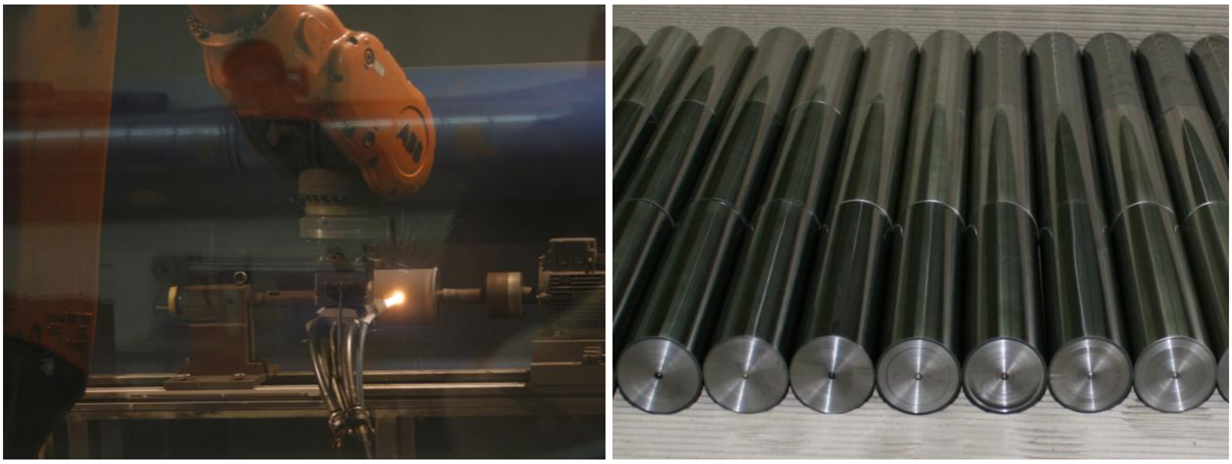

Figure: HVAF-spraying and mechanically ground rods for hydraulic cylinders.

Customers tested the coated parts dependent on their internal quality standards and test procedures. For hydropower generation, erosion and cavitation tests were performed. When the coating acts as a counterpart for a sealing, a porous-free coating with a good abrasive resistance is needed. For applications in plastic processing industry, erosion resistance against solid particle impact is of significant importance. All applications in corrosive environment, either hydraulic cylinders in marine environment or parts in contact with aggressive chemicals like detergents or fertilizers, need a certain resistance against the corresponding type of corrosion. PilotManu coatings were tested in all these types of applications and feedback by the customers was collected in order to improve the powder production process.

As a summary, from the technical point of view, PilotManu WC-Co-Cr-Al coatings can be used for applications in which protection against abrasive wear and/or erosion by solid particles is needed. Their performance will be comparable but slightly inferior to that of the benchmark coating. In applications in which strong corrosive attacks may occur, PilotManu powders may need to be modified.

From the economical point of view, the PilotManu WC-Co-Cr-Al powders may be competitive, as their deposition efficiency during the HVAF spray process is higher compared to the benchmark for some cases. In combination with their expected lower powder price and the good quality of the resulting coatings, PilotManu HEBM powders are an interesting alternative to the current agglomerated and sintered benchmark powders.

Apart from WC-Co-Cr-Al powders, also Fe-based powders with Al_2O_3 reinforcement (FAC-Al) were analysed and sprayed. The figure below compares the coating microstructures of the same powder sprayed with different spray processes (APS, HVOF, HVAF). It is evident that the HVAF process best preserves the powder structure while avoiding oxidation. Potential applications for this powder type is still in progress, but FAC-Al is seen as a cheaper alternative compared to WC-based powders in wear-protecting applications.

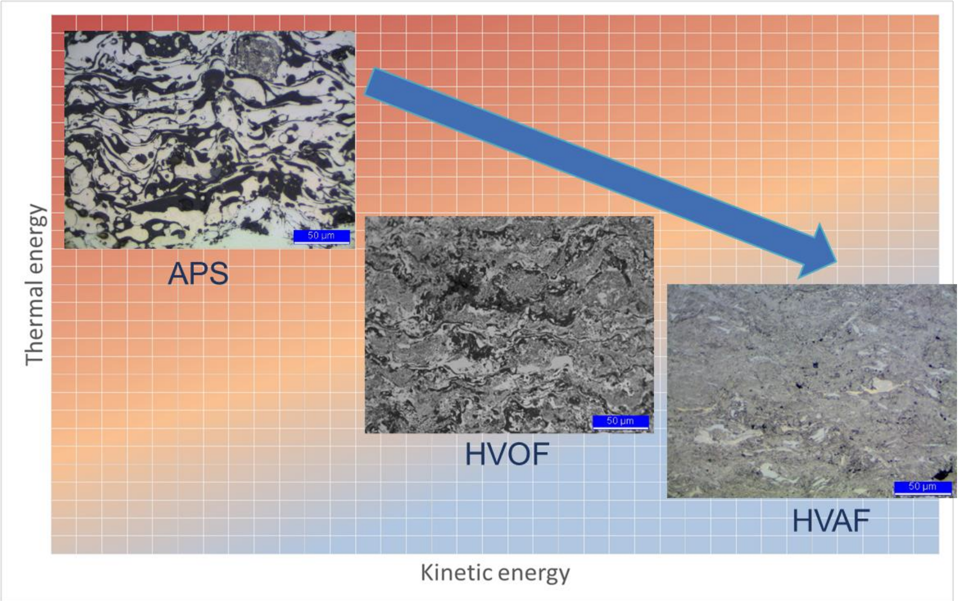

Figure: Microstructure of FAC-Al coatings, thermally sprayed using different spray processes

3.5.2 Progress beyond the state of the art

Current products in the market are hard chromium plated, this process involves formation of toxic / carcinogenic Cr(VI). Thermal spraying process used by Putzier avoids this formation.
Thicker coatings with HVAF are possible as compared to HVOF, this increases the life time of parts and opens up new applications e.g. in harsher erosive environment.
HVAF spraying preserves the powder properties much better than other thermal spray processes. Due to the lower gas temperature during the process, undesired thermal phase transformations, which can lead to decreased mechanical properties and corrosion resistance and thus decreased lifetime of components, are significantly reduced.

3.6 Direct laser sintering

Direct Laser Sintering (DLS) is an additive manufacturing technology for the fabrication of nearly net shape objects. Carried by an inert gas jet, a stream of powders is injected in a melt pool provided by a power laser as shown at left.
MAN is a SME that develops and supplies laser micro-sintering equipment and offers solutions that guarantee reliability and flexibility thanks to a modular and expandable development: during the project the company has tested HEBM materials to be integrated in a DLS production and relevant for the end users application of some industrial cases study.

Figure (left): DLS machine

3.6.1 Description of the result and key achievements

Direct Laser Sintering (DLS) of nearly net shape objects requires the optimization of numerous inter-dependent process parameters. This time consuming task has to be carried on for any new material adopted for the deposition. MAN developed a method to relatively quickly obtain an overview of the possible deposition conditions. This allows the user to select the best conditions for the application with just a qualitative observation.
The first case study, that was stopped at first stage due to encountered issues to sinter the Ti-based cermet materials, was the patterning of biomedical implants with micro-structures enhancing the adhesion and integration with a living tissue (eg. osteo-integration in dental implants).
MAN was then been able to deposit HEBM TiAlNb (material ofinterest in the aeronautical turbine industry) with very stable growth conditions resulting in accurate geometries, smooth surfaces and almost full dense material.
Microfluidics is a technology for the manipulation of fluids at the sub-millimetric scale. The fabrication of large numbers of inexpensive and disposable lab-on-a-chip devices has proven to be challenging since it involves scales that are in-between the few micrometer range for surface finishing and cell interacting objects and the few millimeter range for pipes interconnections and mountings for analysis systems. Micro additive manufacturing falls within these ranges of dimensions

and may be used to directly fabricate metal dies for injection moldings. Here is showed an example of a commissioned study for the fabrication of a die that MAN realized in stainless steel (AISI 316L).

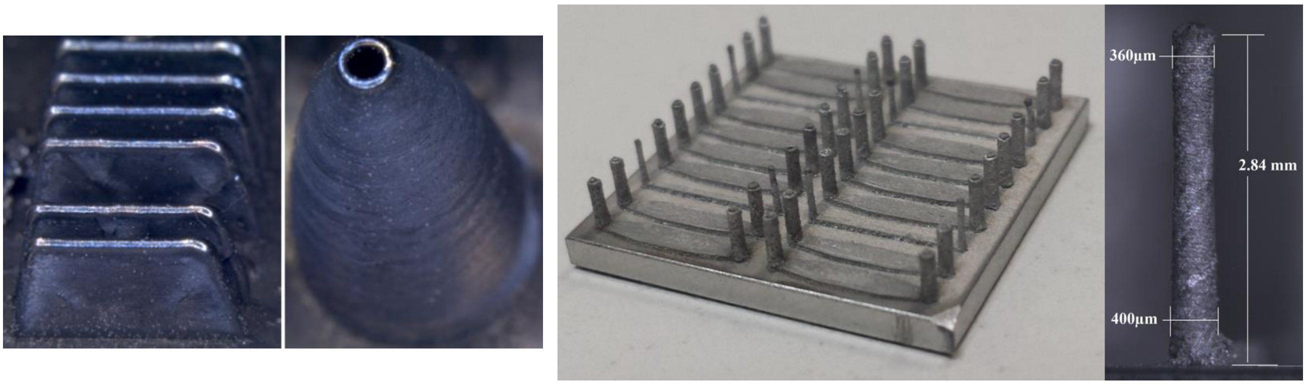

Figure: Samples sintered by DLS technology

3.6.2 Progress beyond the state of the art

MAN validated a method to attain DLS process parameters that constitute a guideline for the use of HEBM materials in additive manufacturing technology. The method has been applied to four materials and allowed to reach satisfying deposition conditions in much shorter times than by trial and error empirical methods.

3.7 Selective laser sintering

+90 is a recently funded SME with the objective of providing rapid prototyping, rapid tooling and rapid manufacturing service through Selective Laser Sintering (SLS) technology: PilotManu project allow to develop and define the process parameters for this technology for a plastic material (polypropylene) novel for this application.

3.7.1 Description of the result, key achievements and progress beyond the state of the art

For more than last 30 years, the term of “Additive Manufacturing” have been used for very different techniques of layered production methods. The most promising ones are being used for production of functional prototypes, models, sacrificial tools, end use parts in different applications for different industries.
In general in last 3-4 years, the interest of developing new materials for different needs in different 3D printing technologies has been greatly increased. Although these technologies have already reached a certain level of development, they still are very complex regarding to the considerable number of different production parameters and affects.
PilotManu has focused on developing a more economical PP-like material which is mostly used automotive industry and also does not exist in the SLS market. In the project material and process based parameters are investigated to understand the role for the quality of SLS parts.
To summarize the work done on the process based, there are 3 different factors effecting the SLS process; layer thickness, pre-sintering temperature and laser based parameters; laser power and laser speed.
The very first studies show that according to the unstable micro-shape of the PP-like powder, the recoating operation during the SLS process is very affected with the layer thickness. It studies resulted in better recoating with a greater layer thickness. 2 different layer thicknesses; 100 µm and 150 µm; are studied and the production trial continued with 150 µm.
One another key factor that affects the success rate of SLS production is pre-heating temperature. SLS is processed under high temperatures, roughly up to melting temperature of the material as it is for conventional sintering methods. Failure to adequately preheat the powder reservoirs or the workspace can lead to poor adhesion. Overheating produces the opposite effect, possibly leading to over-sintering more material than is desired and producing parts with poor dimensional tolerances. In addition, improperly regulating heat distribution of the layers may result in curling, a phenomenon in which the gradient of layers undergoes irregular thermal contraction and physically bends the part structure. Within the determination of pre-heating temperature trials the initial temperature is set to 150 ºC and raised by 2 ºC up to 156 ºC for successful parts.
The development of the laser based parameters is the most complex stage of the process development procedure. In SLS, laser based parameters are separated in different groups; contours and rasters. Each group has own variables. As the TRL level on sintering the PP-like powder is quite low compared to the commercial SLS powders in the market, at this level a few of these variables are selected and studied in PilotManu.

As a result of these studies, it shows us the increase in the laser power during the sintering increases the level of sinterization. However, this increase should be very slight for better parts. Also the surface roughness of the sintered parts are present for most of the demonstrators.

To sum up it can be stated that the development of new suitable SLS materials establishes a real challenging task due to the complexity of the SLS process and the large amount of variables and setting parameters involved during the build stage. The use of PP polymer in SLS application is still a novelty and consequently the availability of PP polymer specifically suitable for SLS. The results achieved for PP in terms of material and SLS parameters optimization are a notable improvement compared to the current know-how. The overall quality of the sintered PP material needs more improvement in terms of surface quality and robustness. At this stage of process investigation the parts can printed with relatively higher dimensional tolerances and rough surface quality.

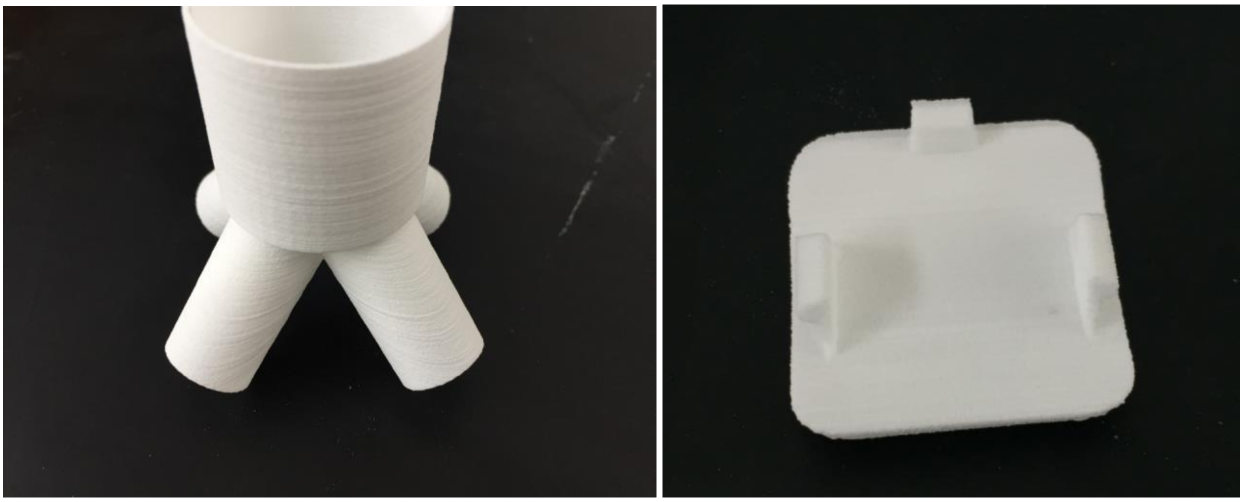

Figure: Examples of laser sintered components in polypropylene

3.8 Description of the LCA analysis performed

Matres compared the energetic performance of the newly developed pilot scale manufacturing line against currently applied competing technology in the three main product lines: due to complexity of the product lines, that involved very different production technologies and applications, it was not performed a cradle-to-grave analysis for the materials/products selected for the validation but to focus the analysis in the following steps:

- Comparison of the production of a number of powders developed during the project for each product line: MTS evaluated the overall energy consumption for the production of a fixed amount of powder with the current HEBM facilities and with the PilotManu plant

- Comparison of the production of the selected components: for each product line, MTS and the involved partners defined the energy consumption for the manufacturing of the components with PilotManu powders and with currently used materials

- Comparison of the selected components: only for diamond cutting tools, PilotManu products were tested in operative environment and compared to current benchmarks

- Evaluation of the energy consumption for HEBM process confirmed that the energy consumption is reduced in the range of 40-50% for powders produced with PilotManu compared to current facilities, with relevant cost saving.

In parallel to the LCA, it was conducted a similar analysis focused on the economic aspects of the new materials (Life Cycle Cost Analysis, LCCA), in connection with the economic assessment of the pilot line.

Evaluation of the energy consumption for HEBM process (including powder post-processing) confirmed that the energy consumption is consistently reduced for powders produced with PilotManu compared to current facilities, with reduction of the direct production costs of 50%.

Table: Difference in energy consumption for PilotManu compared to current HEBM facilities

Product line	***Powder***	***Difference PilotManu vs Benchmark***
1 – Sintering	Fe-based A	-43%
1 – Sintering	Fe based B	-59%
2 - Thermal Spraying	Ti-WC	-43%
3 – Additive Manufacturing	TiAlNb	-32%

The energy consumption and the production costs for the manufacturing of the products was notably reduced for the analysed products in the three lines: in particular for the diamond abrasive rims for cutting disks the PilotManu product achieved lower energy (up to 80% reduction) and production costs (up to 40% reduction) with, at same time, better cutting performance and tool life (+10%).

4 Potential impact, main dissemination activities and exploitation

4.1 HEBM Pilot machine and powders

PilotManu plant will allow to produce much larger powder batches than current facilities, with consequent reduction of operational cost and selling price per Kg of produced powders for a wide range of applications. The specific energy consumption reduction of PilotManu per Kg of processed powder is more than 50% compared to current HEBM Technology.

The advanced micro/nanostructured powders have improved properties compared to the competitors' powders in terms of performance achieved in the components: the adoption of HEBM as manufacturing technique brings to different advantages, like nano-structuring and homogeneous fine distribution of the elements, that cannot be achieved with standard techniques as atomization or re-agglomeration. The mixing achieved through mechanical alloying with the MBN technique is refined up to the nanoscale level and the components (sintered tools, coatings, etc.) made by such powders are positively affected in terms of surface quality, toughness, tribology and mechanical properties. Other advantages are the possibility to obtain a wide range of materials and compositions at solid state and to manufacture powders starting from not miscible materials but achieving a strong refinement and microstructure; furthermore the handling of powder is safe because the powders are nanostructured but have a micrometric size and the process has high flexibility because HEBM allows to process materials based on customer demand.

4.2 Diamond cutting tools by hot pressing and free sintering

DIAM will produce the cutting tools with HEBM powders and, for the beads for cutting wires the development of these new materials with nanostructured multi-phases will allow to merge the manufacturing steps in a single process step due to the self- brazing behavior of the material and consequent lower cost of the process production.
Better performance on the cutting process and on the manufacturing of the cutting tools will allow to sell cutting tools at more competitive price in very short time. The market dimension is notable, due to large number of potentially users of cutting tools for marble and stone cut (mainly construction industry, marble/stone mining sector).
DIAM besides the selling of cutting tools, can provide cutting service for external customers. Based on the elaborated technology of spark plasma sintering of the diamond cutting sectors, the combined SPS – Cold extrusion technology of nanostructured Cu-based composites for manufacturing of Resistance Spot Weld Tips was developed by INOP. The SPS-Extruded Cu-based composite profiles are designed for RSW tips of various diameters production. The demand of the one plant equipped with 50 RSW machines is about 1ton/year of profiles. The Spark Plasma Sintered rings made of Ti-SiC HEBM powder are designed for aircraft high temperature bearing applications and high temperature conveyers applications. The demand of high temperature bearing and bushings is estimated to be about 20 000 pieces per year for two plants equipped with high temperature conveyer furnaces. The future plan is to start production of bushings at the next year.

4.3 Coatings by Cold gas spraying

The intention of IMP is to sell cold spray equipment for deposition of the newly developed HEBM powder materials. The Ti-WC; Ti-SiC hard coatings developed in the project are highly innovative, new materials, commercially available on the market, opening new opportunities on the cold spray coating market, since there are no other commercially available powders for hard coating. IMP is providing the powder datasheets and coating information of the Ti-WC & Ti-SiC materials to each request for hard coatings by cold spraying (Impact Innovations is a global leader in cold spray equipment manufacturing). Except that, the coating and powder datasheets are available in IMP's meeting rooms, exhibitions and distributed to existing customer. The results of the development in the PILOTMANU project are involved in every presentation of IMP. Since the powder market for cold spray applications is just being created, the timing to enter the powder market is ideal

Recently IMP is the only cold spray ID gun supplier on the world suitable for industrial automotive cylinder block ID coating. Based on the positive development results of the PILOTMANU project, IMP exhibited the ID coating device and one ID coated engine block on the International Thermal Spray Conference & Exhibition in Düsseldorf in June 2017. Due to the many advantages of the cold spray process, compared to the benchmark thermal spray process there was a big interest of the automotive industry in the ID coating technology for the cylinder bore ID coating application, assuming successful business cases for both cold spray equipment and HEBM powders in the future.
IMP is providing the powder datasheets and coating information of the FAC-Al and other possible Fe-based materials to each request for ID coating from the automotive industry, the results of the development in the PILOTMANU project are involved in separate presentation of IMP and the prototype video was published on YouTube and other social media channels of IMP.

4.4 Coatings by High Velocity Air fuel spraying

Putzier will provide spraying service to customers interested in wear-resistance coatings. The market opportunities are promising since the global thermal spray market is expected to grow at a compound annual growth rate of 7.79%. Especially replacement of hardchrome is growing as Cr(IV) might be banned by REACH (European regulation for chemicals) in near future. HVAF process coated products have a higher tool life and lower replacement rate. Thus, Putzier will target additional market applications in various sectors such as energy production in marine environment (wind energy, oil&gas), agricultural machinery industry or materials processing industry. HVAF technique is a most efficient deposition process and therefore allows lower cost for the deposition of the coatings.
PilotManu has helped Putzier expanding valuable process knowledge in HVAF and other thermal processes to produce TS coatings, especially from HEBM WC-based as well as FAC powders. Putzier has gained knowledge of the high-energy ball-milling (HEBM) process as an alternative source for high-quality thermal spray powders with nano-structured compositions, which cannot be produced in other commercial routes. Comparison of own laboratory test results to the PilotManu partner's investigations helped Putzier to gain better knowledge of the characterization methods of coatings and the correct interpretation of measurements and results. Practical tests at customer's facilities gave deeper insight into the requirements of the respective industry, making Putzier better able to understand and improve coating applications.
PilotManu has helped to establish an international network of research partners in the field of powders, thermal and cold spraying as well as characterization methods. This network will be of use for Putzier in the future, as the collaboration can be employed to other spray materials and applications.
Putzier plans to expand the collaboration with customers, which have already tested the newly developed PilotManu coatings, to build up a series production. This step towards commercialisation is planned for the next 1-3 years and will probable involve further testing of parts in customers' facilities, before setting up the production.
Apart from that, Putzier will benefit from the knowledge gained in the PilotManu project in addressing new customers for wear and corrosion protection. This is not limited to the powders developed in the project, as the background knowledge can be transferred to other cermet powders as well. This will significantly shorten the time needed for development of new coatings in the future and thus giving Putzier a market advantage.

The HEBM process opens new possibilities for mechanical alloying of different powder materials, as for example low-melting materials with high-melting materials, which cannot be processed by thermal alloying. Putzier will cooperate with MBN to find new customer applications for these types of powders.

4.5 Direct laser sintering

Additive manufacturing sector is fast growing worldwide but limited to few processable materials: for PilotManu materials suitable for DLS processing the possibilities in the market will be numerous in a very short time. Manudirect can provide advisory of manufacturing strategies for deposition in surfaces and construction of features by additive manufacturing with materials not yet developed to be used with this technique and directly assemble and sell optimized DLS machines directly to end-users that use DLS technology in their facilities

For the materials and applications developed during the project, procedures for steel and WC-Co powder materials are finalized, instead the procedures for Ti-based and Ni-alloy will be developed further in the next 1-2 years. Manudirect is already in discussions with three potential collaborations which will help accelerate commercialization and help position DLS machine in the market.

- For Microfluidic injection mould: currently at Technology Readiness Level (TRL) 3. Manudirect is discussions with an Italian University
- Reverse drawing tool: currently at TRL 5. Manudirect is in working with an industrial partner on a project proposal.

4.6 Selective laser sintering

In the market of SLS powder, most of the materials are PA based. In general in last 3-4 years, the interest of developing new materials for different needs in different 3D printing technologies has been greatly increased. Materials with different fillers or elastic materials are quite interesting for several applications. However, since the patents are still present and the powder manufacturers are still holding the price quite high. The recent price of PA2200 (one of the mostly used material), is around 60€/kg. The impact of PilotManu takes place at this point with its 30€/kg for PP-like powder.