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.

Contents

Glossary of Terms

AM	Additive Manufacturing
CAD	Computer Aided Design
CAM	Computer Aided Manufacture
CNC	Computer Numerical Control
CTE	Coefficient of Thermal Expansion
EBSD	Electron Back Scattered Diffraction
F-P	Faby-Perot
FBG	Fibre Bragg Grating
HEBM	High Energy Ball Milling
LMD	Laser Metal Deposition
MA	Mechanical alloying
ODS	Oxide Dispersion Strengthened
PSD	Particle Size Distribution
SEM	Scanning Electron Microscopy
SLM	Selective Laser Melting
SPS	Spark Plasma Sintering
SMF	Single Mode Fibre
STEM	Scanning Transmission Electron Microscopy

1 Executive Summary

The concept of OXIGEN is to achieve increased efficiencies (>30%) in power generation by enabling higher operating temperatures of gas and steam turbines. This will be achieved by the development of Oxide dispersion strengthened alloys, a class of materials that offer exceptional high temperature strength, oxidation and corrosion resistance at high temperatures exceeding 1000°C. While the fundamental material properties of ODS alloys are exceptionally well suited to power generation, the manufacture of components using ODS alloys are currently subject to a number of economic and technical barriers including (1) no means of efficient powder production (2) low volume supply chain, (3) difficult to process and (4) coarse grain size can give variability in creep.

The main thrust of 'OXIGEN' was the development of optimised materials for high temperature operation of SLM and LMD manufactured parts. WP2 extended that thrust and provided “significant added value” by demonstrating capabilities to generate additional functionality and intelligence in the form of in-situ condition monitoring at very high temperatures in components manufactured utilising the unique opportunities offered through the layer by layer build processes of SLM and LMD additive manufacturing technologies.

WP2: Fibre optic sensors, capable of operating at temperatures in excess of 800°C were developed, and embedded into components as part of the SLM/LMD manufacturing process, providing structural and condition information from within a part.

In order to achieve these goals, in-fibre Fabry-Perot type sensors temperature sensors were developed and tested up to 1000°C, fibre Bragg grating based strain sensors were embedded as an integral part of a SLM manufactured part and permit in-situ strain monitoring up to 800°C, far in excess of previously attained temperatures. The detailed understanding of the behaviour and stability of fibre sensors under high temperatures and the challenges of processing fibre material for use at these conditions went beyond state of the art and have been published and presented in per reviewed journals and conferences.

Many of the individual R&D findings leading up to these capabilities represent individual key achievements significantly beyond the state of the art. Embedded strain sensors can for example also be used for process monitoring in the SLM manufacturing process per se, assisting in process optimisation, reduction of stress in parts.

Although WP2 had no direct interface with our eastern European partners, the positive experiences of working and interacting with these partners in the project were a very positive element and contacts formed will be utilised for dissemination into new geographical areas.

WP3: was concerned with the selection and development of Oxide Dispersion Strengthened (ODS) alloys for the project, and their subsequent testing. In conjunction with our end users, two existing Ni-based alloys and one new Ti-Al-Nb alloy type were eventually selected for ODS addition and processing. Phase diagrams were modelled for all the alloys selected and the effects of small additions of Y and Hf, which might be incorporated via ODS particles, were investigated. No new detrimental phases were predicted, but it was found that Hf could depress the solidus temperatures. Phase fractions in Ti-Al-Nb alloy were also very sensitive to Al content. We successfully demonstrated that yttria based particles could be incorporated into powders of all the selected alloys by mechanical alloying, although the use of nm sized yttria additions was shown to give much improved homogeneity and distribution of oxide particles after mechanical alloying.

The addition of ODS particles to a new Ti-45Al-3Nb alloy led to grain refinement, grain boundary pinning and enhanced mechanical properties over a wide range of temperatures, with little segregation after SLM or LMD processing; and the alloy had lower weight gain and good scale adherence when oxidised at 650°C when compared with existing commercial alloys. Cracking on cooling could be minimised by use of a heated substrate and slow cooling during LMD processing. ODS additions to IN625 gave x40 improvement in oxidation resistance compared with the non-ODS variant; while prolonged high temperature testing produced little coarsening of oxide dispersoids. Excellent samples of ODS IN625 and ODS H230 could be fabricated by SPS but it proved difficult to optimise build parameters for SLM and LMD to give uniform, defect-free samples. The new ODS variant of a Ti-Al-Nb alloy is now being fabricated into components for turbines, while the ODS variant of IN625 is a promising addition to the existing range of high temperature superalloys for use in high temperature turbines.

WP4: powder manufacturing routes based on mechanical alloying have been developed, optimized and scaled-up for Ti-based and Ni-based ODS alloys for Additive Manufacturing. Characterization and quality control procedures have been implemented to verify the achievement of microstructural and morphological requirements.
Homogeneous dispersion of nano-oxides in metallic matrix has been obtained by an efficient solid state process based on High Energy Ball Milling (HEBM). Additional procedures for size and shape control and for alloy annealing have been implemented, allowing to select the most suitable particle size distribution and microstructure for both LMD and SLM techniques.
The efficiency of powder manufacturing cycle has been improved by refining process conditions and by integrating the recycling of unused fractions, allowing to increase the process yield in addressed Particle Size Distribution (PSD). This, together with an accurate selection of raw materials, led to competitive projected costs for the resulting powder, compared to conventional non-ODS powders available in the market.
The outcomes of Oxigen project have a relevant exploitation potential for MBN and Matres, with the possibility of commercialization of ODS and non ODS TiAl-based alloys for AM and of further development and customization of Ni-based ODS powders.

WP5:

Selective laser melting, laser metal deposition and conventional sintering techniques were used to validate the manufacture of test specimens using the ODS powder developed in WP3 and manufactured in WP4. For LMD success was achieved in the deposition of TiAl materials giving a 2 fold increased in corrosion resistance. Mechanical alloyed powders were also successfully processed using SLM but those alloys with inclusion of ODS particles proved more problematic from a thermomechanical viewpoint. This was somewhat overcome by adopting a blended approach to ODS inclusion which gave material benefits but with a reduced number of oxide particles remaining in the matrix.

WP6:

WP6 was concerned with technology demonstration and validation in the framework of the OXIGEN project. The project scope was to develop materials that allow operation of power plants at high temperatures as well as materials and methods for high temperature structural monitoring, all in order to increase efficiency, reduce emissions and increase reliability. All of these goals were met to a certain extent as could be shown during this phase of technology demonstration and validation.

In WP6 demonstrators were built from 4 different materials or material combinations. These materials were Haynes 230, IN625-ODS, TiAl and TiAl-ODS applied on the following demonstrators: Burner Segment, Turbine Blade Coupon, Turbine Blade and Heat Shield.

Along with the making of the demonstrators from SLM and LMD, test specimens were produced as well and further tasks were performed such as cutting, machining, polishing, optical measurements, roughness measurements, sensor integration, CT scanning and heat treatment.

WP6 was subdivided into 10 tasks. The first task was preparational where sufficient amounts of ODS powder materials for manufacturing of the demonstrator parts as well as all test specimens were supplied by MBN (mechanically alloyed TiAl-ODS) and by Inspire (blended IN625-ODS). SLM and LMD build strategies were finalized by Inspire and TWI to obtain the best results possible from manufacturing. HWU prepared all final tools and strategies for implementing sensors into the demonstrator parts. All end users (GE, Ivchenko, Siemens) prepared the 3D CAD files required for manufacturing.

The second, third and fourth task comprised the manufacturing of all demonstrators using SLM, LMD and hybrid approaches. The fifth task was concerned with embedment of fibre optic sensors and their respective testing while the sixth task took care of all post-machining and metrology issues.

The seventh task was the NDT validation and QA of the specimens also produced in tasks 2, 3 and 4. The tasks 8, 9 and 10 demonstrated performance benchmarking, powder production capabilities and life cycle impact.

2 Project Context and Main Objectives

The concept of OXIGEN is to achieve increased efficiencies (>30%) in power generation by enabling higher operating temperatures of gas and steam turbines. This will be achieved by the development of Oxide dispersion strengthened alloys, a class of materials that offer exceptional high temperature strength, oxidation and corrosion resistance at high temperatures exceeding 1000°C. While the fundamental material properties of ODS alloys are exceptionally well suited to power generation, the manufacture of components using ODS alloys are currently subject to a number of economic and technical barriers:

- Currently available mechanical alloying processing equipment for production of ODS alloys are time consuming and not effective, leading to high production cost.
- Low volume supply chain lacks major industrial producers i.e. Special Metals and Plansee have stopped ODS production.
- Oxide particle coarsening using conventional fusion (high heat input) joining techniques can lead to reduced high temperature creep strength.
- Difficult to repair for reasons given above.
- Difficult to manufacture with traditional machining techniques (drilling, milling, grinding) due to their superior properties.
- Superior high temperature creep strength in an ODS material requires recrystallization which produces coarse, usually high anisotropic grain structure.
- Coarse grained ODS alloys can give significant component to component variability in creep life. Moreover, these alloys tend to be creep brittle (e.g. <1% creep elongation to failure), so there can be little warning of imminent failure using time averaging approaches, increasing the risk of unplanned downtime.

OXIGEN will address the limitations given above for existing ODS alloys, focusing on the manufacturing of gas and steam turbine engine components for power generators. To achieve this, OXIGEN proposes to undertake development in four areas; (1) Development of new ODS powder materials, (2) Development of ODS powder production techniques, (3) Development of flexible and efficient powder-based additive manufacturing routes for component manufacture, and (4) Embedded sensing for in-service monitoring.

These four development areas will be combined within OXIGEN to enable the end-users (generator manufacturers) to be able to select components, and; improve their high-temperature performance (based on new ODS materials), optimise their design (based on new flexible AM manufacture) and embed sensors/features. The project will also compare the ODS materials developed and AM manufacturing to traditional materials and conventional powder metallurgy processes (sintering, HIPing). This will allow simple selection of the most cost-effective methods for production of higher efficiency generators. Each of these development areas will also consider comparisons with more common material classes for the purposes of benchmarking and knowledge based design opportunities.

The OXIGEN consortium represents the two largest European-based power generation turbines manufacturers (SIEMENS and ALSTOM); who represent ~30% of world-wide orders for gas and steam turbines. In addition, IVCHENKO represent approximately 70% of gas and steam turbines (and compressors) in Ukraine and significant reach in Eastern Europe, Russia and India and will seek to introduce new turbine components to Eastern Europe (see Figure 2a).

These end-users will look to apply the results of the OXIGEN project approach to improve the performance and efficiency of their next generation gas and steam turbine. A number of suggested 'demonstrator' parts have been identified by SIEMENS, ALSTOM and IVCHENKO and described as part of WP1 deliverable.

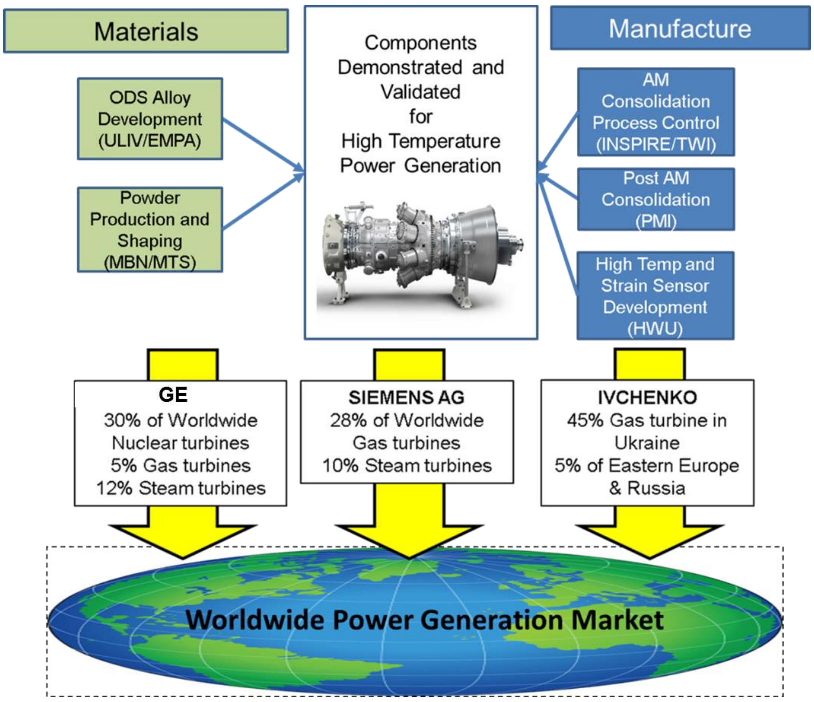

Figure 2a: Outline of the OXIGEN consortium and roles in the project

The OXIGEN project is focussed on achieving six key objectives:

1. <u>Project Specification:</u> *To identify existing components and detail required materials performance to enable higher working temperature; define required materials performance targets and identify target ODS materials and alloys*.

2. <u>Materials Development, Modelling and Screening:</u> *To develop high performance alloys that will compete with existing high temperature performing materials; Develop materials to achieve performance targets, as defined in Objective 1 above.*

3. <u>Powder Material Production, Characterisation and Optimisation:</u> *To develop equipment and procedures to produce powder alloys from material grades developed within OXIGEN*

4. Procedures Development and Test Specimen Manufacture: *To develop procedures using ODS powders to manufacture demonstrator parts for validation in increased turbine efficiency.*

5. Sensor Design, Development and Integration: *To develop and embed fibre sensing technology for in-service monitoring of optimised components; develop Fibre Bragg Gratings (FBG) to facilitate high temperature strain sensing arrays capable of continuous in-service monitoring for stress corrosion cracking.*

6. Demonstrator Manufacture and Validation: *To produce at least 3 components/optimised components, some with integral sensors, for performance testing and monitoring by end-users; achieve up to a 30% increase in engine efficiency by operating with exhaust temperatures above 615 °C.*

The impacts include:

1. Increased power plant efficiency by at least 30% allowing operations at substantially higher temperatures
2. Lower, cost efficient emissions (e.g. CO_2 and/or other pollutants)
3. Improved Reliability of in-service materials
4. Increased safety in the plants of application
5. Boosted cooperation between the EU and the Eastern partnership countries.

To achieve the OXIGEN key objectives the work is divided into six technical work packages, five will involve high technology research and development and one will encapsulate all demonstration activity. A further two work packages, Project Management and Dissemination and Exploitation, will support the technical activity.

3 OXIGEN Partnership

The OXIGEN project consortium has been selected due to their relevance and suitability of previous experience and expertise in the fields of machine construction, robotics, mass manufacture, CAD-CAM programming, process monitoring and control, systems integration, sensor development, non-destructive testing and end use. This will provide the necessary skills and expertise for such as ambitious project to develop automated laser metal deposition manufacturing systems technology that is both sustainable and low resource consuming, whilst being flexible to new applications, cost efficient and capable of adding enhanced functionality to existing components.

The industrial interest in the OXIGEN concept falls within the power industry for high temperature power generation and this is reflected by the strong industrial end user participation. The end users in the project represent significant market share of the high temperature power generation within Europe and the global markets. The strong end user participation will ensure that there is a focussed commercial drive in the development and demonstration phases of the project. There is also a strong SME focus, with 3 SME's from with the materials and powder industry offering key enabling materials and systems supply post project that, through a coordinated effort made possible by OXIGEN and supported by 4 research technology developers.

The RTD partners will provide a turn-key solution to flexible, efficient and cost effective manufacturing. The OXIGEN end-users and supply chain distribution across Europe is given in Figure 3a and the suitability of individual participants is given in Figure 3b

Table 3a: OXIGEN Consortium

Part no.	Participant legal name		Country		Organisation Type
1	TWI Ltd (co-ordinator)	TWI		UK	RTO
2	Inspire Corporation for mechatronic systems and manufacturing technology	INSPIRE		Switzerland	RTO
3	The University of Liverpool	ULIV		UK	Higher Education
4	MBN NANOMATERIALIA SPA	MBN		Italy	SME
5	Eidgenössische Materialprüfungs- und Forschungsanstalt	EMPA		Switzerland	RTO
6	MATRES SCRL	MTS		Italy	SME
7	State Scientific Institution "Powder Metallurgy Institute"	PMI		Belarus	RTO
8	Siemens AG	SIEMENS		Germany	End user
9	Heriot-Watt University	HWU		UK	Higher Education
10	Zaporozhye Machine-Building Design Bureau Progress State Enterprise Named After Academician A.G. Ivchenko	IVCHENKO		Ukraine	End user
11	GE Power Services	GE		Switzerland	End user

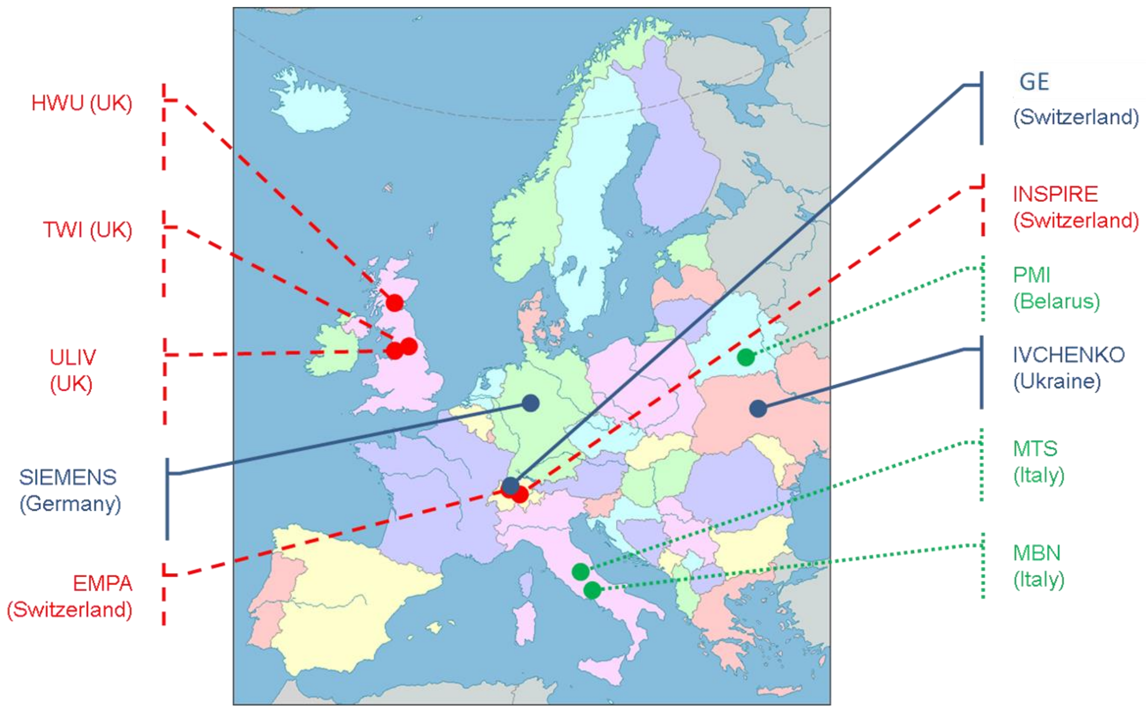

Figure 3a: EU distribution of end users (solid blue line), RTD AND University partners (dashed red line) and SME's (spotted green line).

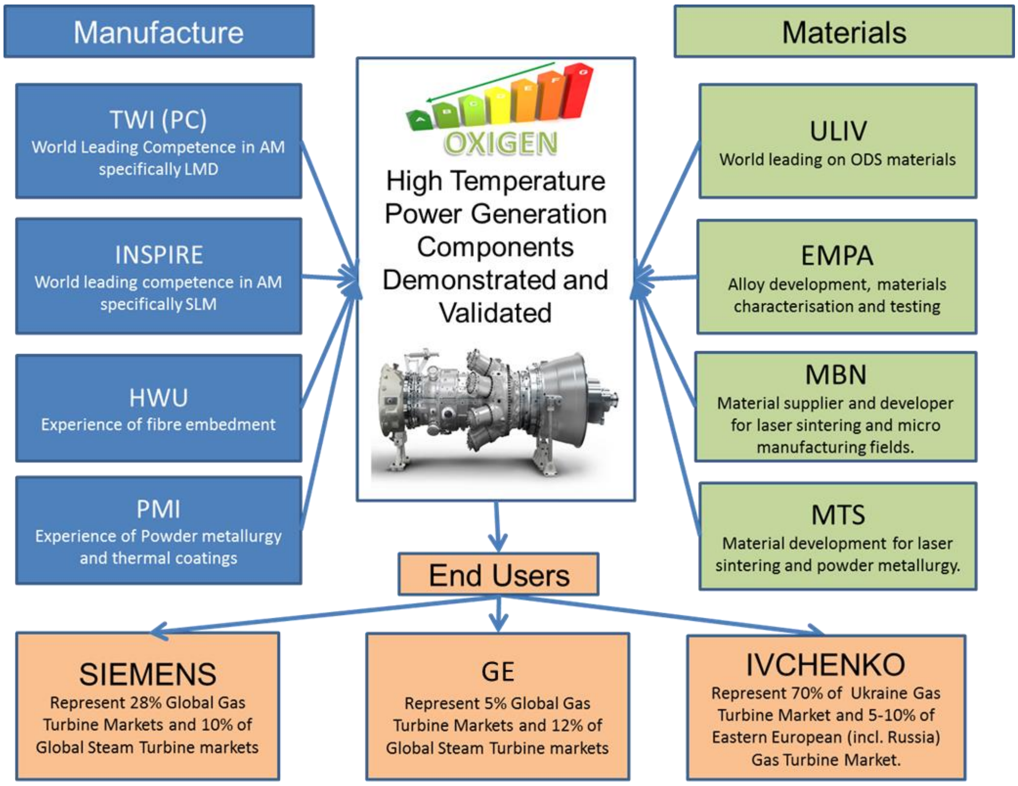

Figure 3b Suitability of Consortium members to deliver the OXIGEN project.

4 Science and Technology Results

The overall work plan strategy for the OXIGEN project has been defined in the Pert diagram (see Figure 4a). There are 3 phases to the project: (1) Demonstrator Road-mapping (component, materials and technologies specifications), (2) technology, material and procedures development for component manufacture and (3) demonstration, testing and validation (relevant demonstrator applications manufactured using ODS alloys).

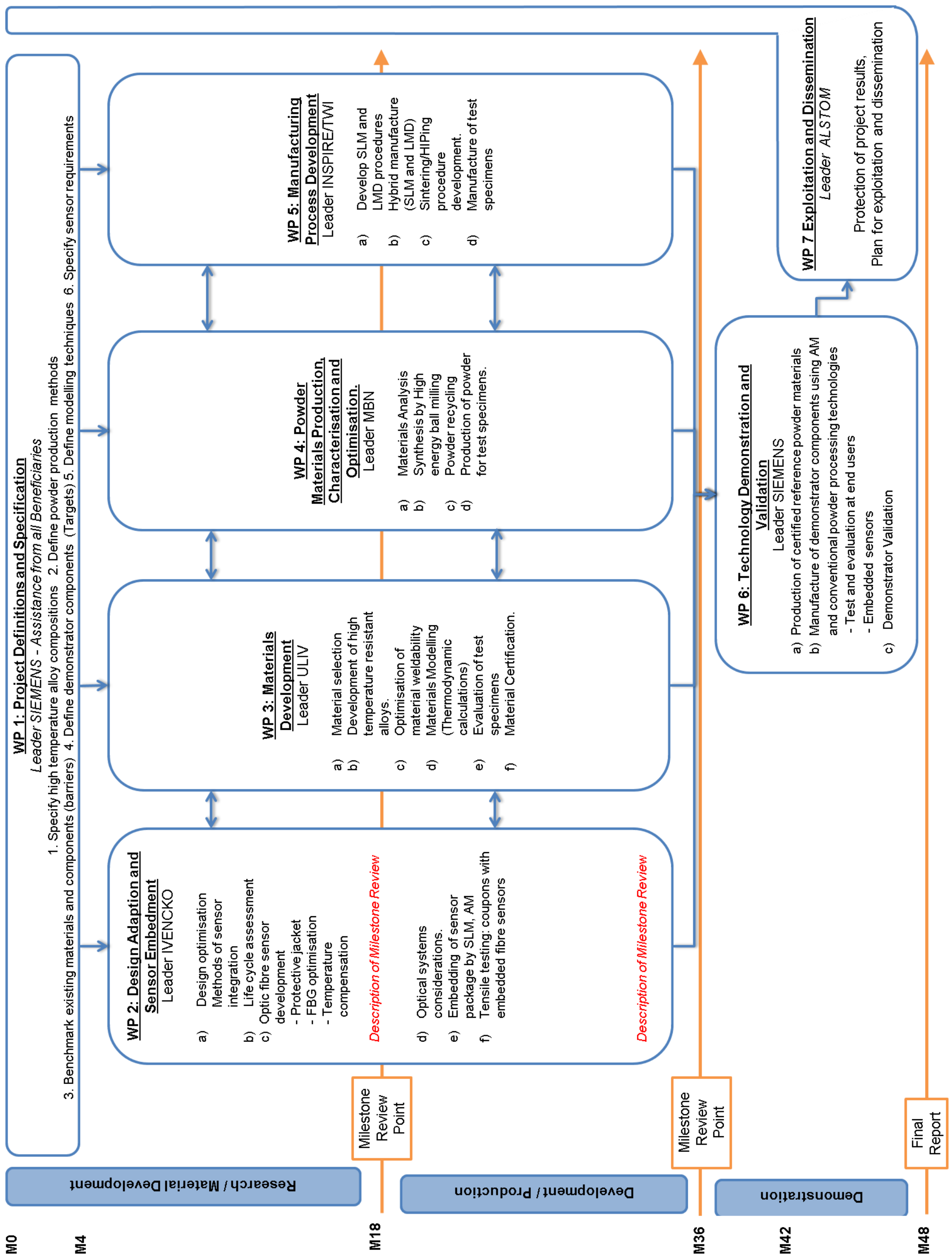

Figure 4a: OXIGEN Pert Chart

4.1 Work Package 1: Project Definitions and Specification

Start month: ***Month 1***
Schedule Completion: ***Month 4***
Status: Completed
Lead Beneficiary: SIEMENS

Task	Task Title	Start Month	Scheduled End Month	Status
1.1	Demonstrator Identification and Benchmarking	1	4	Complete
1.2	ODS Alloy Material Specifications	1	4	Complete
1.3	Powder Production Methodology	1	4	Complete
1.4	Consolidation Methodology:	1	4	Complete
1.5	Sensor Embedment and Validation:	1	4	Complete
1.6	Materials and Demonstrator Testing	1	4	Complete
1.7	Powder Material Recycling:	1	4	Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D1.1	OXIGEN Specification Document	4	Complete	

Milestones	Milestone Title	Delivery Date	Status	Comments
MS1	OXIGEN specification defined	4	Complete	

4.1.1 WP Summary Description and main objectives

WP1 focused on gathering all specifications related to the demonstrators as needed for the project. This work culminated in the deliverable document D1.1 “OXIGEN Specification Document”. It comprised of the entire work of WP1 achieved towards the end of May 2013 (M4) and concluded the results from all seven tasks in this work package.

4.1.2 Specification document updates.

As agreed in the OXIGEN description of work (DoW), the Oxigen specification document was subjected to an annual review and updated as necessary. The review and update process was carried out by Siemens as part of WP1 activity. Within this review, minor updates to the contents of the specification document took place to promote the developments in the project. These changes do not affect deliverable D1.1. D1.1 reflected the status of all aspects of the project to the best of knowledge at the time of its submission.

The only key change was the removal of the IVCHECNKO compressor wheel. Due to the difficulties in processing Titanium aluminide powder by LMD, coupled with the late supply of the material into the project (Nickel based alloys took priority in the early stages of Oxigen) meant that the capability to process TiAl by LMD did not extend to a full compressor wheel demonstrator. This short fall does not diminish project outcomes and developments to successfully manufacture test blocks in a new TiAl material with exceptionally superior material properties to any previous commercially available TiAl powder, has shown great success.

4.2 Work Package 2: Design Adaption and Sensor Embedment

Start month: ***Month 4***
Schedule Completion: ***Month 36***
Status: Completed
Lead Beneficiary: HWU

Task	Task Title	Start Month	Scheduled End Month	Status
2.1	Sensor structures for high temperature sensing	4	9	Complete
2.2	Protective jacketing of sensor fibres	4	9	Complete
2.3	Supply of jacketed sensor fibres for embedding by LMD	4	15	Complete
2.4	LMD fibre embedment optimisation	4	15	Complete
2.5	Embedding of sensor fibres into metallic coupons using a SLM process	4	27	Complete
2.6	Process optimisation during SLM process	4	42	Complete
2.7	LMD embedding of sensor coupons	4	30	Complete
2.8	Testing of sensor coupons	4	30	Complete
2.9	Interrogation and system considerations	4	46	Complete
2.10	Miniaturisation and customisation for embedment using LMD	4	45	Complete
2.11	Design adaption of demonstrator component for SLM	4	44	Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D2.1	Tested Optic with FBG for strain and temperature monitoring	9	Complete	Tested optical fibre F-P sensor for temperature monitoring
D2.2	Supply of protective jacket fibres for LMD Embedment	12	Complete	Direct LMD embedment was shown to be not viable using metal coated fibres with diameters <0.5mm as required by end users
D2.3	LMD embedded sensor optimised for temperature monitoring up to >1000°C (dependant on finalised service)	16	Complete	Successful sensor for temperature monitoring up to 1000°C when using hybrid embedment technology
D2.4	Supply of SLM coupons for LMD Embedment	22	Complete	SLM coupons with embedded F-P sensors have been manufactured, tested and embedded by LMD onto GE supplied SLM parts and have been tested further tested in WP6 as part of the demonstrator components.
D2.5	LMD embedded conformal sensor coupon optimised for strain monitoring	30	Complete	Strain monitoring was not a requirement by end user, hence LMD embedment of a strain sensing coupon was not taken forward. However, strain sensing capability in a coupon similar to D2.4 has been developed and LMD embedment has been demonstrated in D2.4 hence this element of 2.5 would have duplicated these efforts.
D2.6	Modelling evolution report.	36	Complete	This final MS was achieved with the successful build of two demonstrator parts for the end user (GE), utilising a multi level hybrid approach

Milestones	Milestone Title	Delivery Date	Status	Comments
MS2	Embedded and tested sensor fibre using LMD	18	Complete	The fibre was unsuccessful for direct embedment by LMD
MS3	Embedded and tested SLM manufactured fibre sensor coupon using LMD	30	Complete	Embedded and tested sensor fibre using SLM.
MS4	Miniaturised / customised fibre sensor element embedded using LMD	45	Complete d	This final MS was achieved with the successful build of two demonstrator parts for the end user (GE), utilising a multi level hybrid approach

4.2.1 WP Summary Description and main objectives

The utilisation of AM technology and advanced ODS alloys for the manufacturing of power generation components enabled facilitates the addition of added functionalities into the component. In particular, the nature of the layer by layer build process inherent to the SLM and LMD AM technologies enables the integration of in-situ, condition monitoring capabilities providing added value and assisting in the safe operation of a component operating at high temperature.

The key to deliver this objective has been i) the successful development of high temperature compatible fibre optic sensor and ii) the development of embedment processes using hybrid SLM and LMD technology to enable high temperature monitoring by in-situ sensors in the power generation components. The original DOW stipulated temperature and strain monitoring, however the outcome of WP1, preceding WP2, indicated a primary need for temperature monitoring only, in the components identified and selected by the end users Siemens and Alstom, with Alstom later becoming GE.

The embedded, in-situ temperature and condition monitoring sensor technology developed as the final outcome of WP2, were then deployed in two dedicated demonstrator parts built as part of WP6 and tested at the facilities of the end user. The design of these demonstrator parts were adapted in WP6 to facilitate effective and efficient manufacture using SLM and at the same time to accept the conformal sensor coupons with embedded fibre optic temperature sensors, attached by LMD

4.2.2 Main Science and Technology Results

WP2 has delivered significant advances in the area of in-situ condition monitoring of structures and components operating at elevated temperatures far beyond what has been reported to date in the open literature. This has been achieved by developing high temperature compatible fibre sensors and technologies to enable the embedment of such high temperature optical fibre sensors into SLM manufactured components by direct SLM or SLM / LMD hybrid technology. Embedded sensors as developed in the 'OXIGEN' project enable in-situ temperature and strain monitoring at temperatures of up to 900°C and 800°C respectively.

Starting from a lab based feasibility studies on suitable sensor technology for use at high temperatures, embedment technologies and strategies, fibre coating and protection to individual procedural stages and steps, required to deliver the outcomes of the project, work progressed highly satisfactorily in close collaboration with significant input by Inspire, EMPA, TWI and GE at all stages throughout the project. In addition to the named project partners in 'OXIGEN', all other partners in 'OXIGEN' and unfunded PhD and UG students at HWU provided further supportive input.

The R&D program followed the prescribed Deliverables and Task as described in the DOW; and the amendments as reported to, and agreed to, by the commission. Deviations from the original proposed DOW arose from i) revisions to need of the end users, ii) findings of the ongoing R&D indicating a need to revise technological approaches to the proposed manufacturing process, iii) identification of incompatibilities of, for example, direct LMD embedment of fibres and iv) improved understanding of limitations to the original proposed fibre Bragg grating [FBG] based high temperature sensing capabilities.

All the key achievements of WP2 have been reported not only to the project partners at the monthly WP lead conference calls and the 6 monthly PSC review meetings, but have also been reported widely at relevant conferences and in peer reviewed journals as part of WP7 (Dissemination). This dissemination activity is still in progress with 2 further journal papers in progress, one currently under peer review and the second in the final stages of draft. The paper currently still being drafted on “embedded fibres under compression” is describing the technology which underlies the Patent application (GB1622357.0) submitted on 29 December 2016.

The key achievements of WP2 during the duration of the project were:

Development of:

- **multi-layer coating technology to protect fibres and glass components during SLM and LMD embedment from the incident high power laser radiation during SLM/LMD processing**
- sensing capabilities for temperature and strain operating at very high temperature of to 1000 °C and the study of the long term stability of these sensors systems at high temperature.
- **optical coating, splicing and termination technologies for Fabry-Perot type temperature sensors and the behaviour of such systems at temperatures of up to 950°C .**
- **embedment strategies and process using SLM, LMD and hybrid technologies in lab based systems.**
- embedment strategies to commercial SLM and LMD processes (and technology transfer). Supply and provision of sensors, fibres, interrogation and support to partners unfamiliar with fibre optic sensors.
- optical interface and interrogation technology compatible with the high temperature environment.
- capillary embedment, sensor fibre insertion and sealing.
- miniaturisation of F-P based sensor heads to facilitate micro encapsulation.
- **fibre embedment under compression to enable high temperature strain sensing (Patent applied)**
- extension of temperature sensing capabilities to >1000 °C by use of sapphire fibres and ultrafast laser based micro glass to glass welding at dimensions in the order of µm (work in progress) .
- and correlation of theoretical models of sensor response with calibration values for sensors over a narrow temperature range to ~200 °C and extrapolation of the calibration to 800°C based on theoretical models.
- high temperature compatible fibre in/out leads and interconnection to (Au coated).
- conformal sensors coupons for LMD embedment and hybrid processing at partners .
- Integration of fibres into coupons during SLM manufacturing on a **commercial SLM system**. This result enables a transfer of the embedment strategies from lab environment to the next level.
- The on a commercial SLM system embedded fibre is exhibiting a proper bonding between its coating and the surrounding coupon`s material (360°) assuring a sufficient heat transfer from the coupon to the fibre. This is of significant importance for the quality of the measurement results.
- Optical fibre proofed to withstand the harsh environmental conditions in a commercial SLM system, including both the powder and the small bending radii, as well as the embedment process itself → merely negligible losses in light transmissivity after embedment

The overall outcome of the project was made possible by the summation of each successfully developing each individual step shown above, where each bullet point is a summary of a multitude of individual steps. The first bullet, the build of a Fabry-Perot sensor element by itself is a 15 step manufacturing process followed by multiple thermal cycling and ageing process and a number of repeated thermal cycling and calibrations runs.

The above listed key achievements culminated in the manufacture of 2 in-fibre Fabry-Perot type sensor coupons embedded by SLM for subsequent LMD based embedment and over coat demonstrating viability of the hybrid manufacturing approach as final outcome of WP2.

The majority of steps indicated above represent progress beyond the state of the art since fibre embedment by SLM and temperature and strain sensing at the level demonstrated in 'OXIGEN' has not been reported before. Individual steps were tailored and fine-tuned to achieve a particular and optimised function and the successful overall outcome of the project is a sum of the whole.

The following two aspects represent the key progress beyond the state of the art for the developed system:

- The ability to monitor strain at the µ-strain level at temperatures in excess of 700°C in metals, with sensors becoming an integral part of a component, represents a step change in strain monitoring technology. Embedment by SLM and LMD and hybrid technologies into and onto metallic systems will enable new levels of metrology in a wide range of engineering, process and power industries. This work has resulted in a patent (applied for).

- Although in-situ strain sensing by embedded fibres was originally conceived to allow condition monitoring in SLM built components (in a gas turbine environment), strain sensing at the demonstrated level has given a deeper insight into the development of stress in the SLM manufacturing process itself. This ability will assist in the improvement of the SLM manufacturing process by a better understanding and potential minimisation of the of build-up of residual stress.

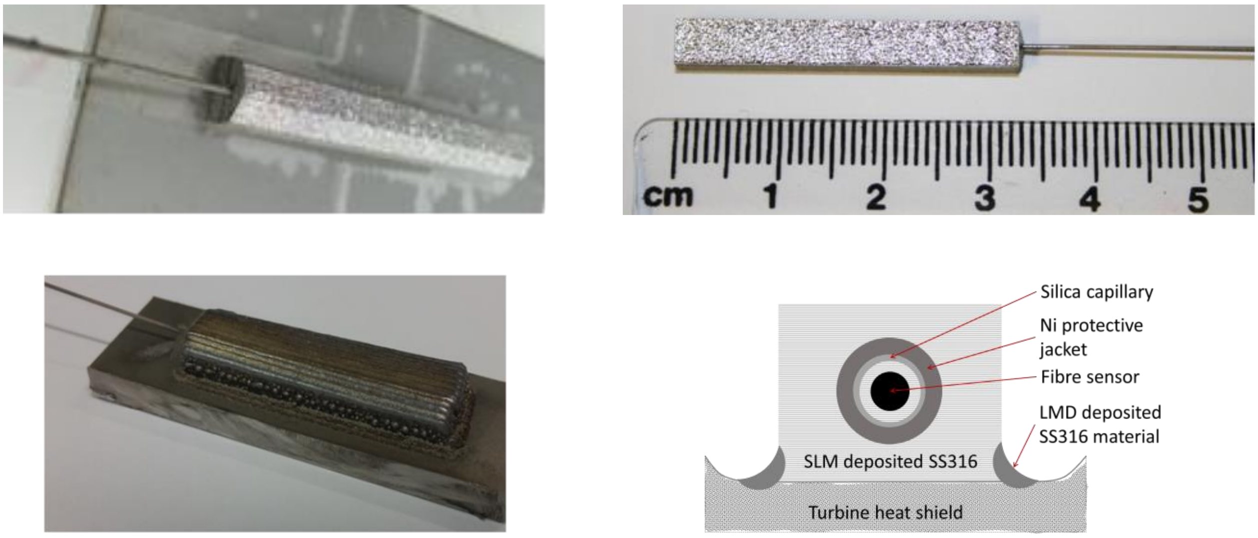

Figure 4.2a: Top left: SLM coupon with embedded fibre under compression for strain sensing; top right: conformal SLM coupon with embedded capillary for insertion of F-P based temperature sensor fibre; Bottom left: LMD embedded conformal sensor coupon; Bottom right Schematic of cross section of embedded F-P sensor with attachment to Demonstrator heat shield by LMD (in WP6)

4.2.3 Potential Impacts and Exploitation

Prior to 'OXIGEN', the prospect of embedded in-situ condition (strain) monitoring in metals at elevated temperatures in the order of 700 °C was raised in conversations with metallurgists involved in the design of super saturated steam systems for power generation and their response was that such a technology would address “a very urgent need” and could constitute “the holy grail in metallurgy” by enabling the in-process detection of stress corrosion cracking in [SCC] such high temperature, high pressure environments. The ability to detect SCC in operation will enable the safe and effective operation of power generation systems at ever higher temperatures, thus enhancing the thermos dynamic efficiency of the plant, and mitigate against unexpected shutdowns.

The key element to commercial and technological exploitation for such applications is the patented process (applied for) enabling the embedment of fibre strain sensors in a compressed state. HWU will drive forward the exploitation of this technology by continuing to work on refinement and improvement of the technology in collaboration with relevant industry partners. Initial links with Doosan Babcock have been established. We will also continue to work with TWI, our partner in 'OXIGEN', to utilise dissemination to their network of members through workshops and industry relevant publications.

Within 'OXIGEN' the outcomes of WP2 have primarily been demonstrated in two end user components which were successfully tested at GE’s facilities and future exploitation and use within GE is under review. HWU, the lead in WP2 will maintain links with GE and support their future in-house exploitation.

SLM manufacturing technology, used for the embedding of fibre optic sensors, by itself requires optimisation and more fundamental research to identify and mitigate processes leading to intrinsic stresses present in manufactured components. Research in WP2 has shown that embedded fibre strain sensors can provide an enhanced level of understanding of the SLM manufacturing process and HWU will develop the technology in collaboration with Inspire and TWI, 2 of our 'OXIGEN' partners and Renishaw, a strategic research partner to HWU and a leading manufacturer of SLM systems and metrology instrumentation.

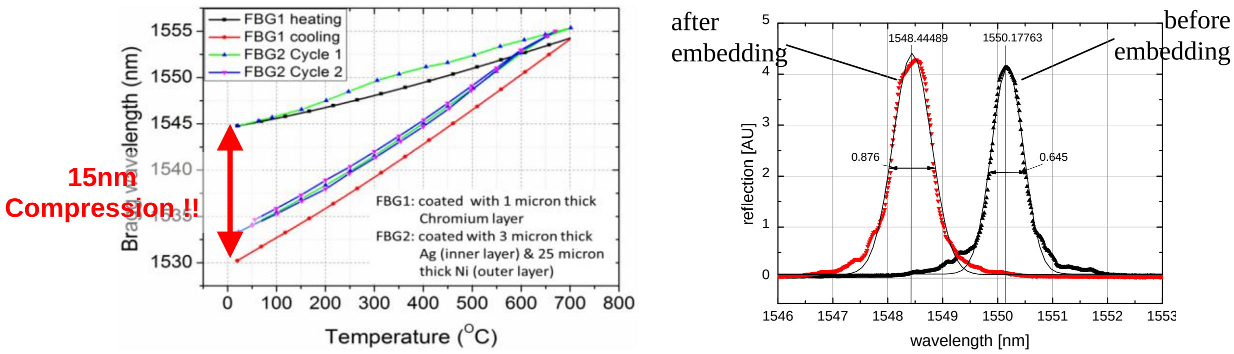

Figure 4.2b: Left: Compression of an embedded fibre achieved by a multi stage embedment process, enabling the use of FBGs for strain sensing to temperatures far in excess of any previously reported patented values. The process has been a key achievement of 'OXIGEN' and a patent has been applied for. Right: FBG response prior and post embedding, indicating a stress in the material due to the wavelength shift, indicating stress in the material due to the SLM process.

4.3 Work Package 3: Materials Development

Start month: ***Month 4***
Schedule Completion: ***Month 36***
Status: Completed
Lead Beneficiary: UoL

Task	Task Title	Start Month	Scheduled End Month	Status
3.1	Definition of materials performance baseline	4	9	Complete
3.2	Alloy composition modelling	4	12	Complete
3.3	Initial microstructural studies and screening of new alloys and powders	4	24	Complete
3.4	Evaluation of test specimens made by additive manufacturing processes.	4	36	Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D3.1	Report on limitations of existing alloys	9	Complete	
D3.2	Report on predicted solidification phases of chosen ODS alloy systems	12	Complete	
D3.3	Report on limitations of existing alloys for use in high temperature components.	15	Complete	
D3.4	Degradation data from test samples manufactured from the new alloy powders	24	Complete	
D3.5	Microstructural characterisation of test specimens prior to demonstrator manufacture	36	Complete	
D3.6	Modelling evolution report.	36	Complete	

Milestones	Milestone Title	Delivery Date	Status	Comments
MS5	Full material specification for 3 demonstrator components	12	Complete	Linked to D3.2
MS6	Material grades produced and final selection made for manufacture of optimised test samples	24	Complete	Shifted to M36 due to unforeseen difficulties in powder processing and consolidation. Linked to D3.5 and D3.6
MS7	First test samples characterised and assessed for high temperature operation	36	Complete	Most of the work completed as part of D3.5

4.3.1 WP Summary Description and main objectives

This work package targeted the development of oxide dispersion strengthened (ODS) variants of two commercially available nickel alloys, together with a Ti-Al-Nb based alloy, for use in applications at temperatures in excess of 650°C. An additional requirement was that the material should be processed to a final powder form that was suitable for consolidation by additive manufacture techniques such as selective laser melting (SLM) and laser metal deposition (LMD). The objective of this work was to characterise material throughout all stages of production and provide feedback to consortium partners to enable the development and optimisation of mechanical alloying and laser consolidation parameters. Although Liverpool and EMPA were the main partners in this task, there was considerable interaction and iterative developments with the powder producer, MBN, via WP4 and organisations such as INSPIRE and TWI, who were responsible for processing the powders within WP5.

4.3.2 Main Science and Technology Results

During the alloy development and testing phases of this project, different approaches were used for the two different alloy classes. For the Ti-Al-Nb alloy a bottom-up-approach was taken, which gave more flexibility in alloy development and higher added value, but with greater risk. For the Ni based alloys the end users were keen that conventional commercial alloy powders should be the starting point, to ensure that powders could be processed into acceptable components, even if there were some modifications to the microstructures. The main outcomes of the project were the successful development a new Ti-Al-Nb alloy with enhanced mechanical and high temperature corrosion properties, and a modified Ni-based alloy, IN625, with yttrium rich ODS additions which gave considerably enhanced high temperature oxidation performance.

Phase diagrams were modelled for all the alloys selected and the effects of additions of Y and Hf, in particular, were investigated. No new detrimental phases were predicted, but it was shown that Hf could depress the solidus temperatures in some alloys, which might lead to greater segregation during solidification. The predicted phase fractions in the Ti-Al-Nb alloys were also shown to be very sensitive to Al content. Modelling of the Ti-Al-Nb system also allowed the determination of the best composition range for the production of an optimised microstructure.

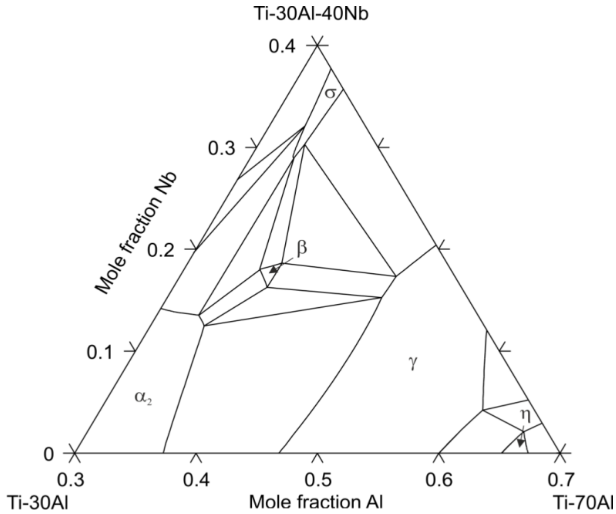

Figure 4.3.1 *Re-calculated partial ternary Ti-Al-Nb isothermal phase diagram at 1273 K*

The addition of yttrium rich ODS particles to the new Ti-45Al-3Nb alloy led to grain refinement, grain boundary pinning and enhanced mechanical properties over a wide range of temperatures, with little segregation after SLM or LMD processing. It was demonstrated that advanced thermodynamic simulation could be used to predict the solidification path and correlated microstructure formation in Ti-Al and Ti-Al-Nb alloys. The modelling could also be used to predict the outcomes of processing of Ti-Al alloy powders under non-equilibrium conditions, such as found in beam-based additive manufacture where solidification rates are very high.

The new Ti-45Al-3Nb ODS alloy had a lower weight gain and good scale adherence when oxidised at 650°C when compared with the nearest commercially equivalent alloy. There was some cracking on cooling, but this could be minimised by use of a heated substrate and slow cooling during LMD processing.

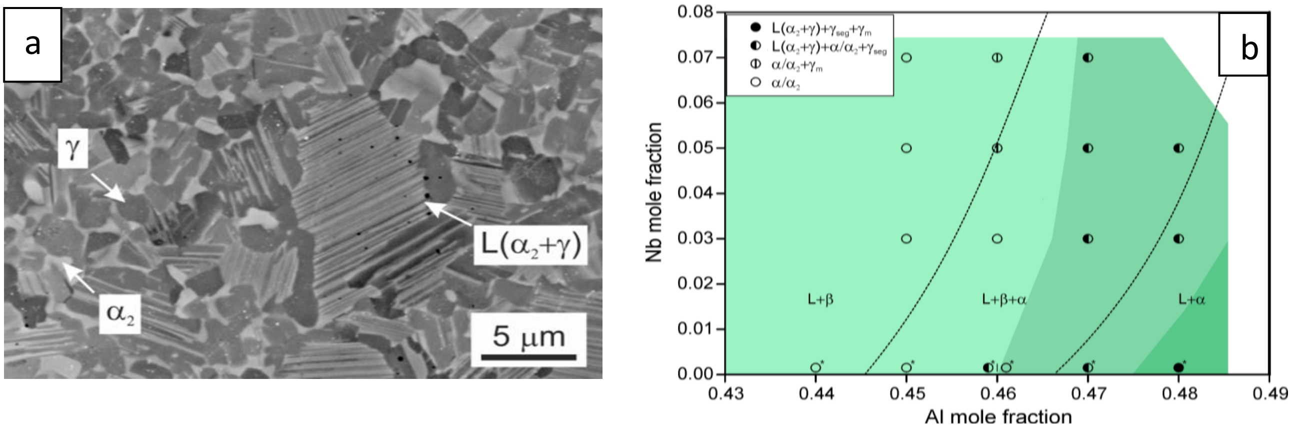

Figure 4.3.2 *(a) Microstructure of SPS ODS Ti-45Al-3Nb after a stabilization heat treatment at 850°C for 12h (b) Experimental composition – microstructure map of Ti-(44-48)Al-(0-8)Nb rapidly solidified at 4.5 (±0.2) ·10^3 K·s^{-1}.*

For the Ni-based alloys, it was successfully demonstrated that yttria based particles could be incorporated into powders of all the alloys by mechanical alloying. However, the use of nm sized yttria additions was shown to give much improved homogeneity and distribution of oxide particles after mechanical alloying; and prolonged high temperature testing produced little coarsening of oxide dispersoids.

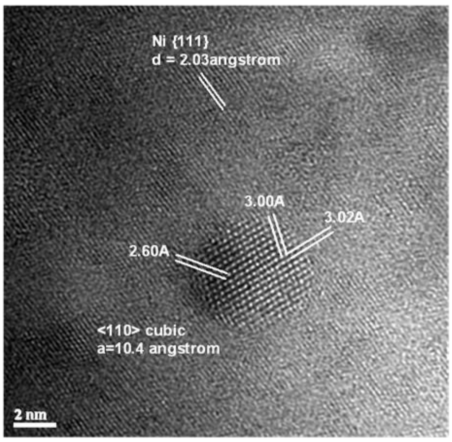

Figure 4.3.3 *Second phase particle, displaying a crystal structure consistent with yttria, observed in a Ni-based alloy.*

Excellent samples of ODS IN625 and ODS H230 could be fabricated by SPS but it was difficult to optimise build parameters for SLM and LMD to give uniform, defect-free samples. However, it was shown that similar enhanced performance may be achieved by blending with yttria powder rather than mechanically alloying. Thermobaric sintering of samples by PMI also gave new insights into the microstructural changes which occur during consolidation of ODS powders.

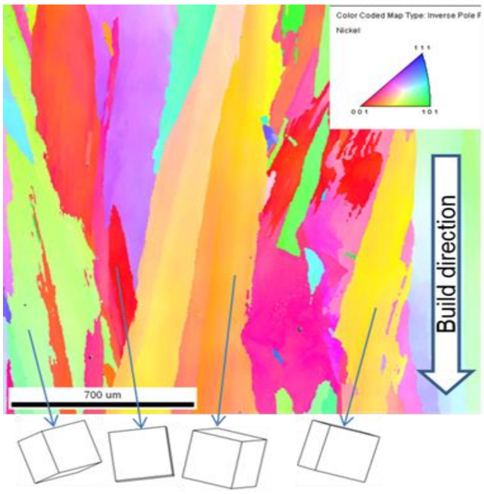

Figure 4.3.4 *EBSD orientation map showing the ⟨100⟩ solidification texture of LMD built Haynes 230 - ODS*

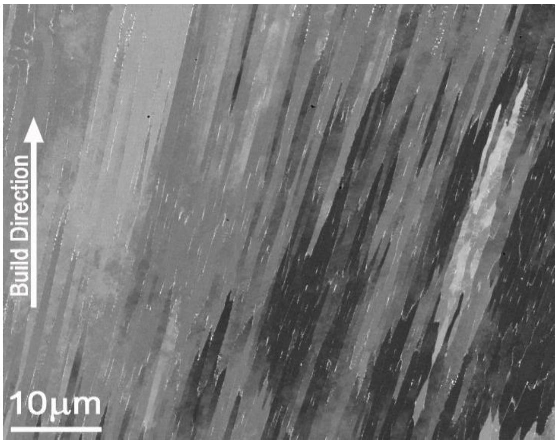

Figure 4.3.5 *Dendritic structure of columnar grains in SLM deposited Haynes 230*

The use of ODS additions to IN625 opens up the possibility of recrystallising the alloy, after consolidation by SLM, to produce components with equiaxed grain structures instead of the strongly columnar structures usually produced during SLM. ODS additions to IN625 also gave x40 improvement in oxidation resistance compared with the non-ODS variant, probably by the incorporation of small traces of yttrium into the oxide scale and scale / metal interface.

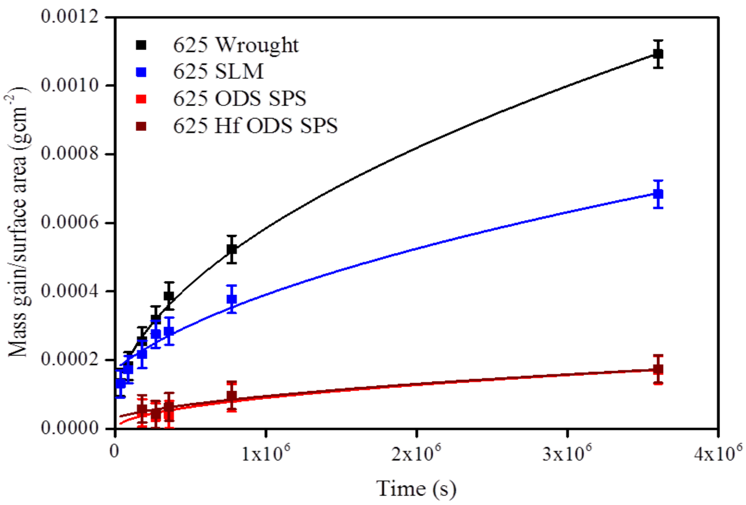

Figure 4.3.6 *Mass gain per unit area (gcm^{-2}) against isothermal oxidation time (s) at 900°C in air for the various IN625 alloys.*

Summary of the main outputs from WP3

- A new Ti-Al-Nb ODS alloy has been developed with enhanced oxidation and mechanical properties at high temperatures compared with conventionally produced, commercially available alloys of this type;

- ODS additions to an IN625 alloy have resulted in a x40 improvement in its oxidation resistance at 900°C;
- Mechanically alloyed powders of IN625 and Haynes 230 can be consolidated by SPS into acceptable components, but SLM or LMD processing gave samples with inhomogeneous microstructures;
- The use of blended powders of alloys plus yttria gave more promising microstructures after SLM processing;
- The use of ODS additions to IN625 opens up the possibility of recrystallising the alloy, after consolidation by SLM, to produce components with equiaxed grain structures;
- Advanced thermodynamic modelling can be used successfully to predict the solidification path and microstructure of samples consolidated under non-equilibrium conditions such as those found during SLM or LMD processing.

4.3.3 Potential Impacts and Exploitation

The Ti-Al-Nb alloy development is a major step forward and can be further refined. It will be exploited by Ivckenco in the production of turbine components. The basic alloy composition will also be published, and may be exploited by other alloy manufacturers, although the precise additions and processing of the alloys will remain proprietary. The enhanced modelling approach used for the design of this new alloy can be applied to other complex alloys and will be used by EMPA in other systems subjected to non-equilibrium processing conditions.

ODS additions to IN625 have given a x40 improvement in high temperature oxidation resistance; and this will be exploited by the turbine companies Siemens and GE as an attractive alternative material to existing superalloys. ODS additions to IN625 also open up the possibility of new heat treatment regimes for SLM processed IN625, since the alloy can be forced to recrystallise at high temperatures and hence the columnar grain structure usually produced by SLM processing can be removed by heat treatment.

The development of advanced techniques for observation and characterisation of 2-10nm size particles in metal matrices was necessary for this project and was carried out by ULIV. They will use a similar approach when studying nano-scale particles in other alloy systems in the future.

4.4 Work Package 4: Powder Materials Production, Optimisation and Characterisation.

Start month: ***Month 4***
Schedule Completion: ***Month 36***
Status: Completed
Lead Beneficiary: MBN

Task	Task Title	Start Month	Scheduled End Month	Status
4.1	Powder Synthesis by high energy ball milling	4	16	Complete
4.2	Powder engineering and recycling	4	31	Complete
4.3	Powder and milling process optimization	4	36	Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D4.1	Synthesis route and powder production methodology for each application case and characterization	12	Complete	Submitted
D4.2	Powder engineering and post treatments of powder materials	24	Complete	Submitted
D4.3	Production of reliable ODS alloy powders for the laser sintering process: characterization protocols	30	Complete	Submitted
D4.4	Powder product analysis: effectiveness of the manufacturing cycle and cost analysis	36	Complete	Submitted

Milestones	Milestone Title	Delivery Date	Status	Comments
MS8	Availability of 12 material variants for processing by SLM and LMD	18	Achieved	Linked to D4.1 and D4.2
MS9	Availability of optimised ODS alloy powder materials for demonstrators	36	Achieved	Linked to D4.3 and D4.4

4.4.1 WP Summary Description and main objectives

The objective of this work package was the development of advanced powders by mechanical alloying suitable for additive manufacturing of high temperature resistant components. An effective production route has been developed for the powder materials selected for each application case, including the definition of characterization and quality control protocols. In particular, the activities of WP4 were focused on TiAl-based and Ni-based ODS alloys for SLM and LMD techniques, in close collaboration with the material design activities of WP3 and with processability evaluations performed within WP5.

Within Task 4.1 the synthesis route has been defined for each material variant, modulating the process conditions to achieve homogenous dispersion of nano-oxides in metallic matrix and to meet microstructural requirements. Within task 4.2 the activities were focused on refining both the powder microstructure and the morphology, in order to improve SLM and LMD processability and to deliver required mechanical properties. Finally, within task 4.3, both the powder properties and the manufacturing process have been optimized, applying the manufacturing procedures to pilot scale, improving the process yield and the management of powder quality.

4.4.2 Main Science and Technology Results

Task 4.1

A solid state synthesis process for ODS alloys has been developed for TiAlNb and Ni-based alloys, based on High Energy Ball Milling. By managing process parameters it was possible to achieve an homogeneous dispersion of nano-oxides in metallic matrix, with an efficient process compared to

the state of the art, characterized by a shorter process – more than 10 times – with lowered processing costs. Dedicated Ti and Ni lined vials and milling means have been employed for excluding any cross contamination from the grinding means used during mechanical alloying .
The solid state synthesis of TiAlNb starting from raw elements has been achieved. This approach allows to process a wide range of possible compositions (i..e high Nb content), to control the content of low melting elements (i.e. Al) and to obtain a finer microstructure compared to conventional atomization process.

Task 4.2
The particles morphology has been optimized, targeting the particle size distribution for SLM and LMD processes, by refining the conditions of post-synthesis resizing and classification steps. The processability of resulting powders has been validated for both Ni-based and TiAl-based alloys within WP5 activities.
The post-synthesis annealing of TiAlNb alloy was refined, identifying the process conditions suitable for the management of alloy microstructure.

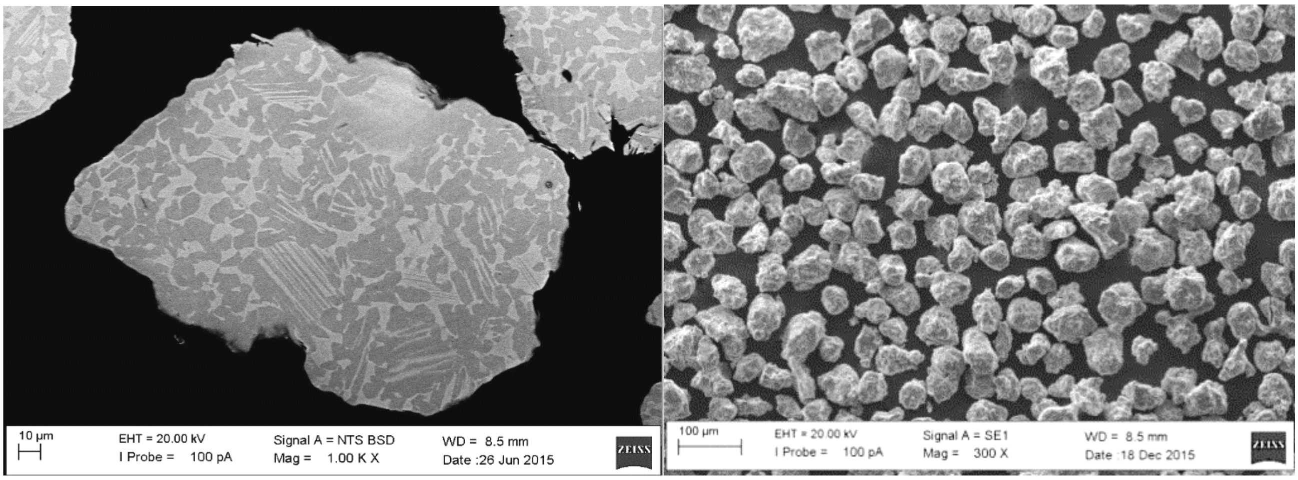

Figure 4.4a: SEM images of TiAlNb globular and lamellar microstructure of particles (left) and powder morphology optimized for LMD process (right).

Specific characterization protocols have been developed and integrated with manufacturing cycle to support the manufacturing procedures, allowing the optimization and the management of powder quality. More in detail, the content of interstitial elements, the particle size distribution and the powder flowability have been mainly taken into account.

Task 4.3

The time and cost effectiveness of powder manufacturing cycle has been evaluated, allowing to identify and optimize the most critical steps. The recycling of unused powder fraction has been implemented at pilot manufacturing scale leading to an increase of overall process yield for the SLM and LMD complementary fractions, without compromising material quality. An extensive screening of raw material sources has been performed too, allowing to identify additional solutions for powder cost limitation, in view of post-project exploitation.
More than 100kg of powder have been supplied into the project.
Particularly for TiAlNb ODS powder for LMD, the evaluated powder costs resulted competitive compared to similar commercially available powders for AM.

4.4.3 Potential Impacts and Exploitation

The outcomes of Oxigen project have a relevant impact for MBN:

- novel powder materials by HEBM are available, i.e. TiAlNb ODS alloys and NiCr-basd ODS alloys, produced by mechanical alloying approach;
- A pre-commercial production scale is available for TiAlNb ODS and non ODS powder for AM;
- The synthesis and processing capabilities by HEBM have been widened, developing know-how in:
    - the dispersion of nano-oxides in metallic matrices
    - the achievement of suitable morphology for LMD and SLM processes
    - the solid state synthesis of TiAl alloys from metallic elements
    - the management of heat treatment for microstructure refinement of TiAl alloys
- the output of modelling based approach of material for laser processing techniques has been implemented into powder manufacturing by mechanical alloying

These outcomes pave the way for the exploitation of project results, enabling the perspectives of:

- commercialization of ODS and non ODS TiAl based alloys for AM
- further developments and customization of Ni-based ODS powders for powder metallurgy techniques (e.g. SPS, extrusion)

It worth noting that initial requests have been already received for TiAlNb powders, suggesting the high exploitation potential of this project result.

4.5 Work Package 5: Manufacturing Process Development

Start month: ***Month 4***
Schedule Completion: ***Month 36***
Status: Completed
Lead Beneficiary: Inspire

Task	Task Title	Start Month	Scheduled End Month	Status
5.1	Selective Laser Melting Process	4	36	Complete
5.2	Laser Metal Deposition Process Development	4	36	Complete
5.3	Conventional Sintering Process Development	4	36	Complete
5.4	Manufacturing of test specimens	4	42	Complete
5.5	Microstructure and Geometry Analysis	4	42	Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D5.1	SLM-processing and post-processing parameter set for all materials	36	Complete	
D5.2	LMD-processing and post-processing parameter set for all materials	24	Complete	
D5.3	Processing and post-processing parameters set for sintering/HIP for materials and AM test samples	28	Complete	
D5.4	SLM and LMD-processed and sintered/HIP specimens for material integrity testing in WP3	32	Complete	

Milestones	Milestone Title	Delivery Date	Status	Comments
MS8	Delivery of ODS LMD, SLM and Hybrid samples for material analysis in WP3	32	Complete	Approx. 150 tensile samples and more than 50 additioonal samples for advanced characterisation were delivered

4.5.1 WP Summary Description and main objectives

The main objectives of WP5 are the development of processing procedures for Selective Laser Melting, Laser Metal Deposition and conventional sintering / HIP processes for all the materials addressed in the project (Task 5.1 to Task 5.3). The material development is thereby following a 2-step approach: In a first step, selected base-line Ni-alloys are processed and the consolidated materials are characterized. In a second step, one ODS variant of a Ni-alloy is developed and characterized, allowing a comparison of microstructure, mechanical performance and oxidation behaviour between base-line alloy and its ODS-variant (Task 5.4, 5.5). Testing itself is mostly conducted in WP 3.

4.5.2 Main Science and Technology Results

- Task 5.1

The main work of this task consisted in finding the fitting SLM process boundary conditions and parameter sets most materials developed during the course of the project. Nonetheless, the focus of the work was on the Nickel based ODS and non-ODS materials.

The key achievements are:

1. The results have shown that all materials chosen by the consortium (IN718, IN625, H230) can be successfully consolidated to a crack and nearly pore free microstructure without any post processing step. Nonetheless, the as-built microstructure shows strongly elongated grains along the build direction (z-direction), causing mechanical anisotropy between 10 % to 30 %, especially for the elastic modulus. This is also highly influenced by the processing parameters used and is therefore a critical point when parts are being used in the as built state.

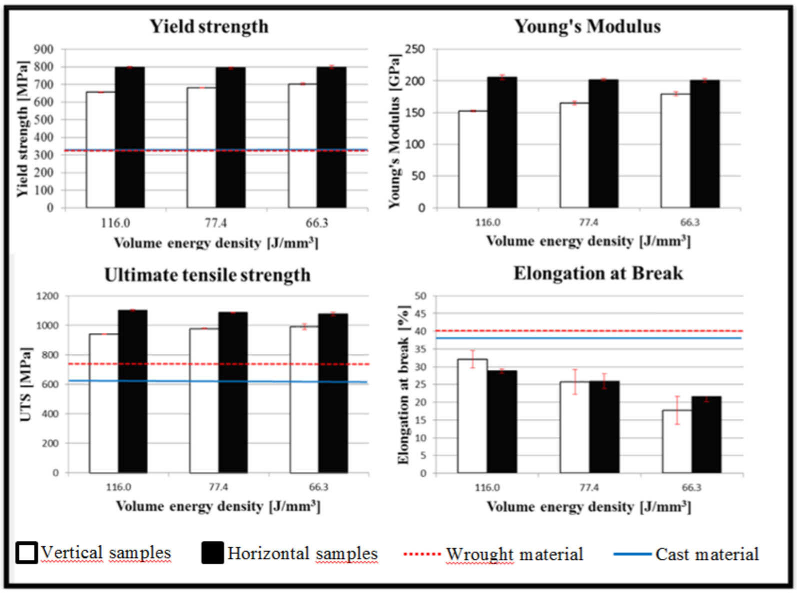

Figure 4.5a: Exemplary mechanical data of H230 non ODS

2. Due to the aforementioned issues, in practice the materials require sophisticated heat treatment cycles in order to lower the anisotropy as far as possible, or ideally to remove the texture through full recrystallization. Together ULIV, PMI and Inspire developed adjusted heat treatment cycles to achieve the goal of a reduced anisotropy for all materials inside the project focus.
3. The SLM-machine capabilities inside the OXIGEN consortium (INS, TWI, GE) were used in order to deepen the understanding of the influence of different machines on the processing behaviour of the materials used. This lead to a profound knowledge on how to transfer the results from Inspire to the SLM-systems of the project partners.
4. A parameter set for a pore and nearly slag free volume for SLM could be developed.

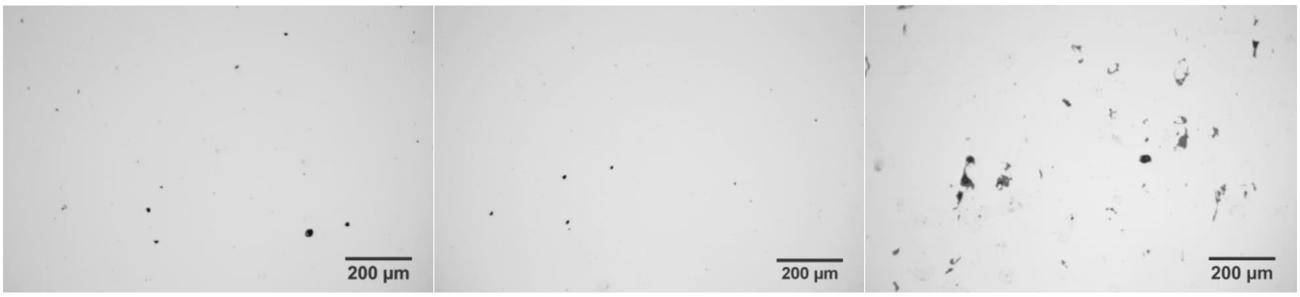

Figure 4.5b: Slag inclusion are emphasized by higher scan speeds and larger hatch distances, the pictures show increasing scan speeds

5. As a result of the very high volume energy density needed for the consolidation the surface parameter had to be carefully adjusted to allow for the production of complex parts with an acceptable surface quality for post-processing.
6. The technical obstacles of the mechanical alloyed powders were overcome by the blending process but with reducing the number of oxide particles remaining in the matrix.
7. Increasing the productivity and reliability for the production of SLM parts using blended material.

- Task 5.2 LMD

The tasks in this work package were focussed on the crack free processing of TiAl ODS variants and therefore the development of the appropriate processing equipment to allow for heating at elevated temperatures, controlled cooling as well as inert atmosphere shielding.

<u>The key achievements are:</u>

1. Crack free processing of TiAl and TiAl ODS variants.
2. Successful development of processing strategy including controlled cooling and heating cycles to limit the cracking during thermal cycling of the process.
3. Design of the required equipment (induction heater, gas shielding) to perform the required tasks.

Development and implementation of an induction-assisted laser metal deposition process to improve TiAl alloy processing and produce TiAl ODS and non-ODS samples. The developed process aimed to resolve the processability issues of oxidisation above 450°C and material solidification cracking upon rapid thermal cool down. The material is highly crack susceptible due to its ultra-low ductility (especially low at room temperature) and.

EMPA developed a new TiAl alloy and MBN produced it in powder form using HEBM for the LMD technology. The powder was delivered to TWI for the AM research to be carried out in the last 12 months of the project. The process conditions were developed to utilise a fine melt pool which minimised thermal stresses and reduced the thermal difference between substrate and deposition track. The downside to the material requirements is a restricted deposition rate at this point in the technology readiness. Parameter development and sample creation lead times were therefore increased substantially.

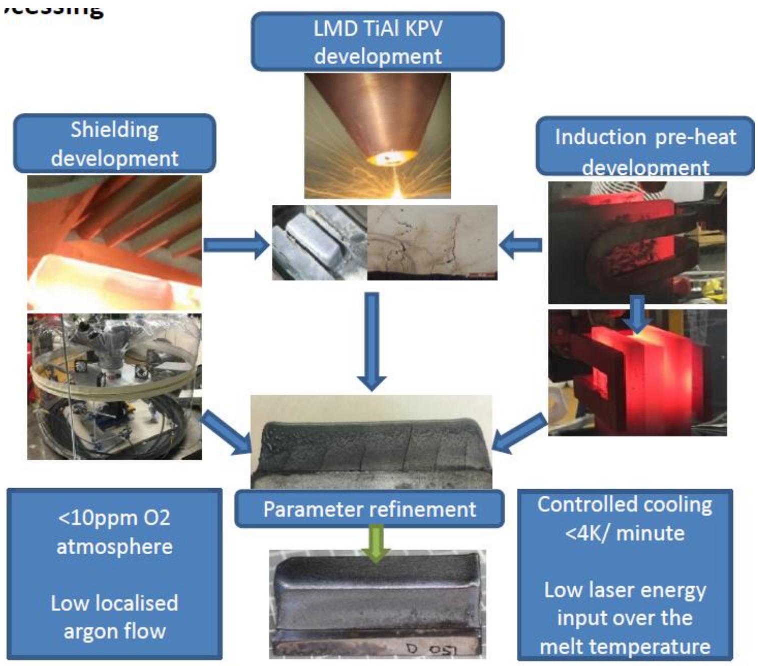

Figure 4.5c: Overview of steps need to reach a crack free build of TiAl using LMD

Limited tensile testing was performed on the more promising TiAl ODS specimens. The tests were performed at 800°C. The LMD processed TiAl ODS appeared to perform better than the SPS processed TiAl non-ODS material but worse than the SPS processed TiAl ODS.

- Task 5.3 Sintering

It was investigated a possibility of additional compacting of SLM samples using HIP and was determined, that under the SLM samples density more than 96,5%, it is observed an additional compacting up to state practically without pores. A maximum measured value of additional compacting is 3,5 relative percents. A density near to theoretic for all samples from industrial nickel alloys was obtained after HIP using standard modes.

It must be noted that after hot isostatic pressing of SLM samples, besides density, their structure and microhardness are changed. A specific layered structure and splat orientation disappear and a structure of alloy typical for used parameters of treatment is formed. What is more, this structure practically does not depend on structure of SLM samples before HIP, excepting the fact that in more porous samples some pores (disposed, as a rule, in the field of accumulation of some non-melted particles) remain unchanged.

As in the case of IN 625 ODS powder, a traditional technological scheme of powder metallurgy, including a pressing and subsequent vacuum sintering is not effective also for H 230 ODS powder. The density after vacuum sintering makes about 88% and during the sintering process a great number of fragile phase inclusions is formed which are crumbled during sections preparation.

The bests results of ODS powders consolidation are achieved with using of thermobaric sintering. Using this method, samples from IN 625 ODS powder were obtained having a density near to theoretical without a visible segregation of elements and oxide consolidation. In addition, investigations made on H230 powders have shown that an optimal temperature.

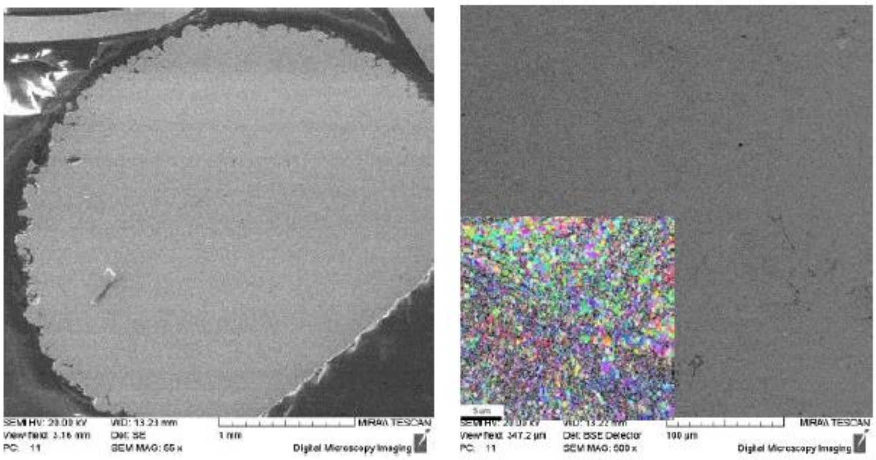

Figure 4.5d: Microstructure of thermobaric sintered Inconel 625 ODS (HEBM) displaying very fine grains in the micron scale

<u>The key achievements are:</u>

1. Close to full density can be achieved for thermobaric sintering
2. Processing windows for thermobaric sintering for HEBM ODS-Ni-superalloy powders, and for HIP-post-processing of SLM- samples from the conventional nickel superalloys powders are defined.
3. TBS of H230-ODS samples led to good compressive strength
4. Conventional sintering and vacuum sintering + HIP is leading to sufficient material density, due to coarse and brittle oxide formation.

4.5.3 Potential Impacts and Exploitation

The broad field of processes and ODS as well as non-ODS materials investigated during OXIGEN enables the research and industry partner to be at the frontier of their specific field.

In the first part of the project the understanding of the base alloys later used for ODS additions were significantly deepened for Titanium-Aluminides as well as Nickel-based alloys. The additional data created by the processing of the ODS materials enables a solid foundation for the development of high performance alloys using SLM, LMD as well as conventional sintering. Therefore TWI, INS and PMI already use the gained processing knowledge for other advanced materials.

INS together with MBN developed an alternative powder production route to blend particles or in-situ alloy different materials which has proven to be a very successful approach for not only Nickel-based alloys but a few different material classes (aluminium based alloys for example). Due to unknown long term behaviour of those blended powders it remains unclear whether this in-situ alloying remains a lab scale technology or can reliably be used in large scale manufacturing. So far the results of the ODS demonstrator production are looking promising.

Also work future cooperations between the project partners has been done.

4.6 Work Package 6: Component Demonstration

Start month: ***Month 36***
Schedule Completion: ***Month 48***
Status: Completed
Lead Beneficiary: Siemens

Task	Task Title	Start Month	Scheduled End Month	Status
6.1	Preparation Phase	36		Complete
6.2	SLM of demonstrator components	36		Complete
6.3	LMD of Demonstrator Components	36		Complete
6.4	Hybrid Manufacture of Demonstrator Components	36		Complete
6.5	Embedment of Fibre Optic Sensor	36		Complete
6.6	Post machining and Metrology	36		Complete
6.7	NDT Validation and Quality Assurance	36		Complete
6.8	Performance Benchmarking	36		Complete
6.9	Powder Production Capability	36		Complete
6.10	Demonstrator Life Cycle Impact	36		Complete

Deliverable	Deliverable Title	Delivery Date	Status	Comments
D6.1	Manufacture of one LMD demonstrator component	40	Complete	
D6.2	Manufacture of one SLM demonstrator component	44	Complete	
D6.3	Manufacture of one Hybrid demonstrator component	46	Complete	
D6.4	Benchmarking study of demonstrator performance	46	Complete	
D6.5	Life cycle Impact of 2 demonstrators	48	Complete	

Milestones	Milestone Title	Delivery Date	Status	Comments
MS11	First demonstrator for field testing produced to specification outlined in WP1	40	Complete	
MS12	100Kg of MA powder produced to demonstrate feasibility of production capability	44	Complete	

4.6.1 WP Summary Description and main objectives

The objective of WP6 is to manufacture of demonstrator components using combinations of AM and HIP using materials and powders developed and produced within OXIGEN. The next step is a validation of demonstrators by end users. Further initial objective was to embed, test and evaluate a temperature/strain sensor in SIEMENS demonstrator component. This task has been hand over to GE (former ALSTOM). The last objective of WP6 is to evaluate life cycle impact of Project implementation on one demonstrator.

4.6.2 Main Science and Technology Results

The manufacturing of a LMD demonstrator (Ivchenko)

10 Large (70x20x20mm) TiAl tensile specimens were produced by LMD and were sent to Ivchenko-Progress for carrying out machining trials and tensile tests. The blocks were produced using high

control over the processing conditions for thermal and shielding requirements. Thermal cool down was controlled by induction heat and oxidization was controlled by completely shrouding the build in an inert argon chamber.

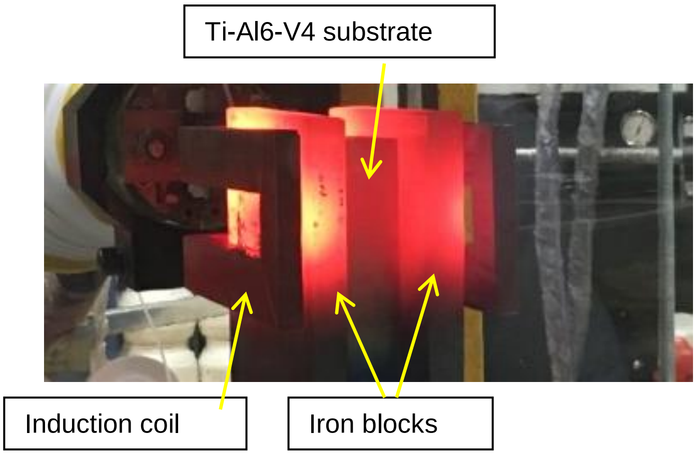

Figure 4.6b: shows the final pre –heating setup

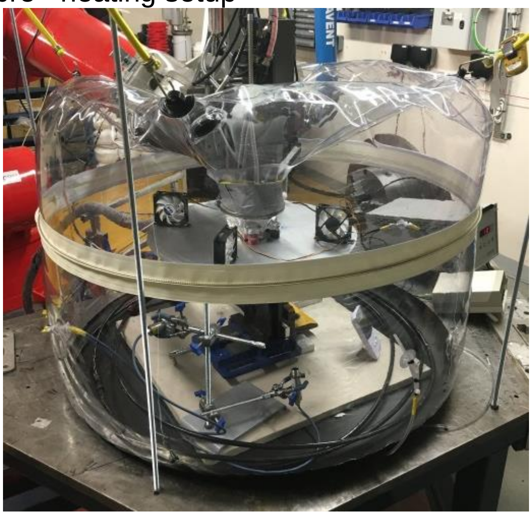

Figure 4.6c: Flexible purge chamber developed to seal around the robot head and still allow limited articulation.

The final blocks were built with relatively fixed parameters. Although it would have been beneficial to build some further blocks with varied conditions and build strategies.

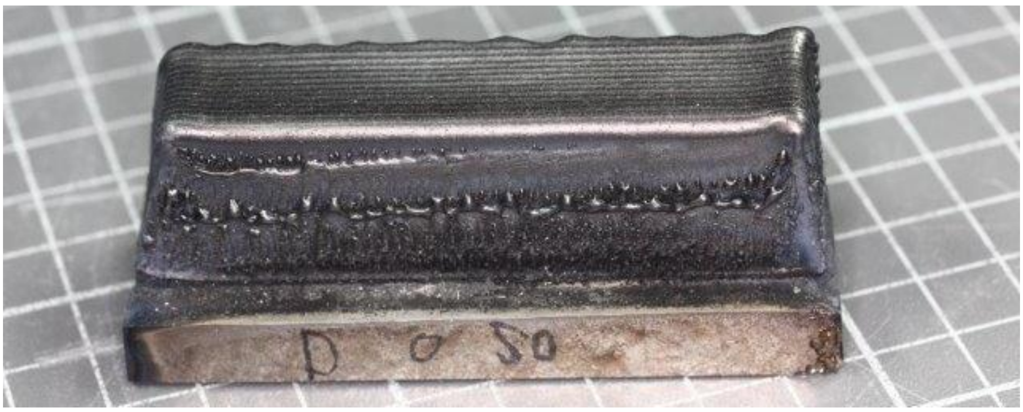

Figure 4.6d: one of the TiAl ODS tensile test specimens built by LMD

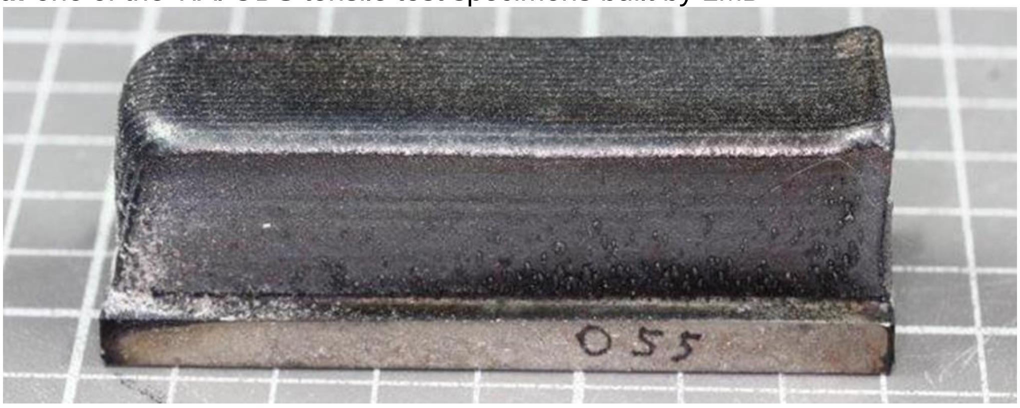

Figure 4.6e: one of TiAl non-ODS tensile test specimens build by LMD

The manufacturing of a SLM demonstrator's (Siemens)

The demonstrator provided by Siemens is a gas turbine heat shield. A quantity of three pieces was manufactured in the OXIGEN project from the newly developed ODS-variant of IN625. The Selective Laser Melting process required for the production step was provided by Inspire where other demonstrators and test specimens were manufactured in the same production batch as shown in the image below where on the left side two heat shields can be seen in the front. The right side shows how the heat shields can be tiled with a perfect fit just like inside the combustion chamber of a gas turbine.

Figure 4.6f Demonstrators and test specimens manufactured from Selective Laser Melting

University of Liverpool contributed the heat treatment and respective HIPing before Inspire did the cutting of the demonstrator parts from the build platform as well as machining of the surfaces supported with auxiliary structures only needed at build time.

The oxidation testing was not performed on the actual component as the results obtained from the oxidation testing specimens already suggested excellent reduction of the oxidation rate by a factor of approx. 20.

The manufacturing of a HYBRID demonstrator's – GE Demonstrator #1

The GE Demonstrator #1 – tripled Burner Segment has been manufactured using the new HYBRID approach utilising both SLM and LMD. In this case, LMD has been a secondary manufacturing method to build up additional material onto an SLM built substrate.
The manufacturing process chain used for the manufacturing the HYBRID demonstrator contain following processes and is visualized in Figure 4.6h:

1. Additive manufacturing of the tripled Burner Segment out of Haynes 230 alloy using SLM at INSPIRE. Sub-section 2 has been manufactured twice to guaranty two components with an embedded sensor are available for further testing on test rig.
2. Wire EDM to remove the components from the substrate plate at GE.
3. Hot Isostatic Pressing (HIP) of the tripled Burner Segment for reduction of porosity of the SLM generated material at GE.
4. Final machining for removal of support material and post-processing of functional surfaces like sealing slots using milling and grinding at GE.
5. LMD for attaching and coating of the SLM embedded high temperature sensor coupons (developed and manufactured in WP3) to the tripled Burner Segment at TWI.

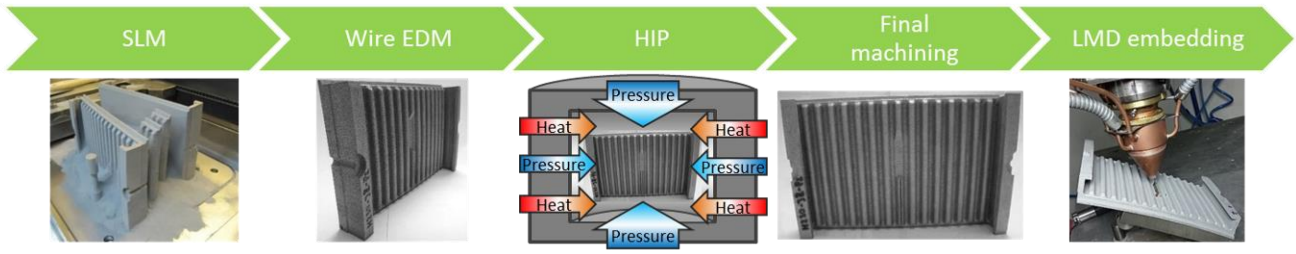

Figure 4.6h: Manufacturing process chain for the manufacturing of the HYBRID demonstrator

D6.4: Benchmarking study of demonstrator performance – GE Demonstrator #1

The GE Demonstrator #1 – tripled Burner Segment has been used for benchmarking of the performance of the HYBRID component. To allow the benchmarking of critical components and material properties following tests have been performed at GE.

1. Optical measurement to check the dimensions of the tripled Burner Segment in as build conditions and after HIP at GE.
2. Roughness measurement at critical surfaces in as build conditions and after HIP at GE.
3. Computer tomography (CT) to check the component for internal defects in as build conditions and after HIP at GE.
4. Mechanical testing of tensile specimens build in the same batch as the demonstrator component itself. Samples in as build conditions have been tested at room temperature (23°C). HIPped samples have been tested as well at room temperature (23°C) as at the elevated temperature of 850°C at GE.
5. Testing of sensors embedded in the HYBRID GE Demonstrator #1 in a test rig at temperatures up to 800°C at GE.

The achieved results are described in detail in the following text.

Benchmarking results – Optical measurement

The three Burner Segment sections had been optically measured in as build conditions and after HIP. In as build conditions the deviation of all critical measuring points fluctuate between -0.083 mm and 0.110 mm, see Figure 4.6b A, C and E. Apart from measuring point RPSPunkt_3_Y of sub-section 1 all measuring points are within the tolerance, see Figure 4.6i A).

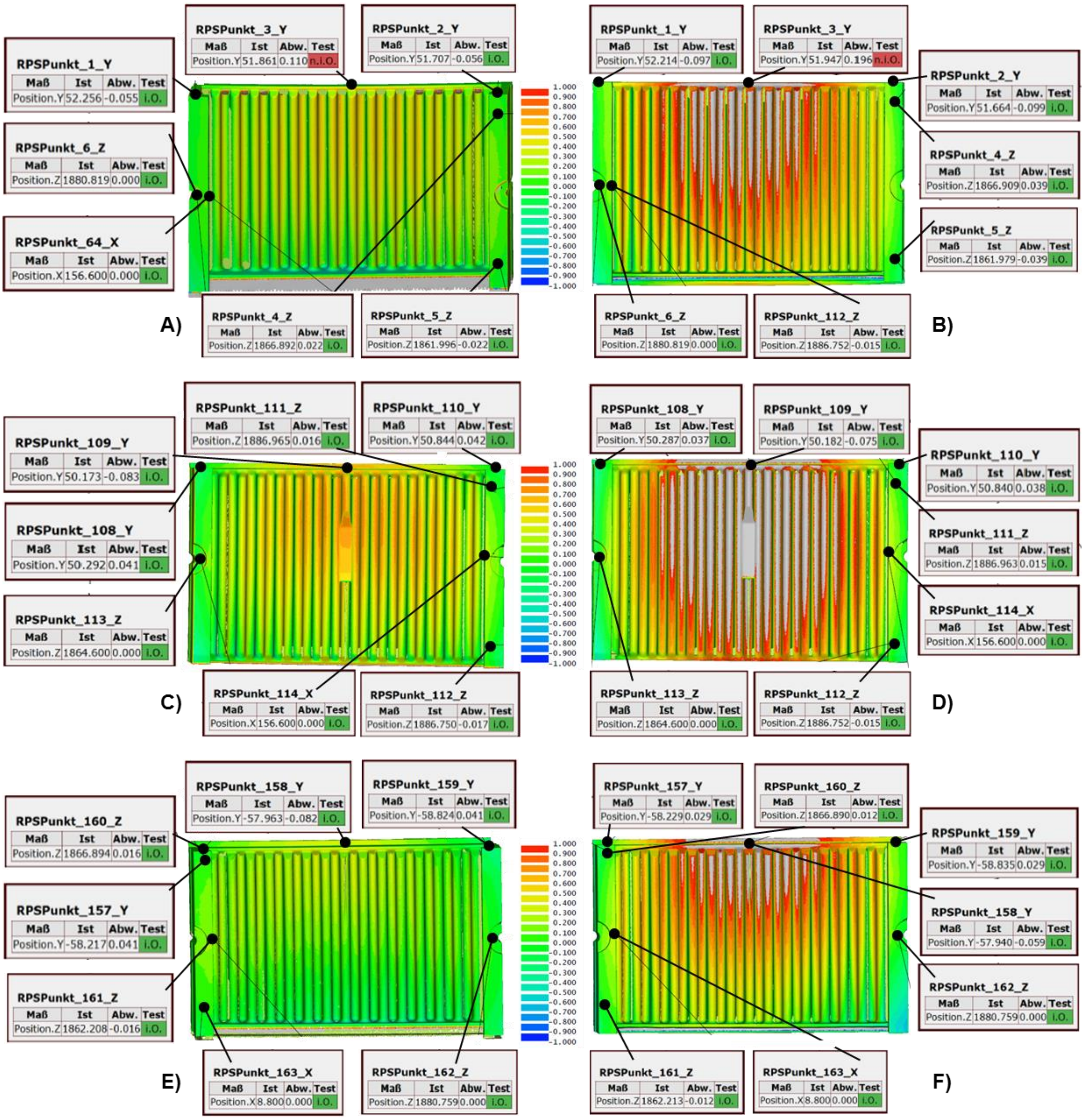

Figure 4.6i: Results of dimensional measurement of GE Demonstrator #1;
A) sub-section 1 in as build conditions; B) sub-section 1 after HIP;
C) sub-section 2 in as build conditions; D) sub-section 2 after HIP;
E) sub-section 3 in as build conditions; F) sub-section 3 after HIP

After applying the HIP process the deviation of all critical measuring points fluctuate between -0.099 mm and 0.196 mm. As already observed at the as build components measuring point RPSPunkt_3_Y of sub-section 1 in HIPped conditions is out of tolerance, see Figure 4.6i B).

All three sup-sections show in their central region a significant deformation after HIP. For the fundamental functions of the component these deformations are not critical. Further, these now known deformations, can be considered during the design phase of the component and hereby significantly reduced.

Benchmarking results – Roughness measurement

The roughness of the three Burner Segment sections has been tactile measured in as build conditions and after HIP. The measurement has been performed on 17 different positions, see Figure 4.6j. 3. Three measuring positions are allocated at the cold side of the GE Demonstrator #1, 10 are at the hot side, further four are on the left and the right rail.

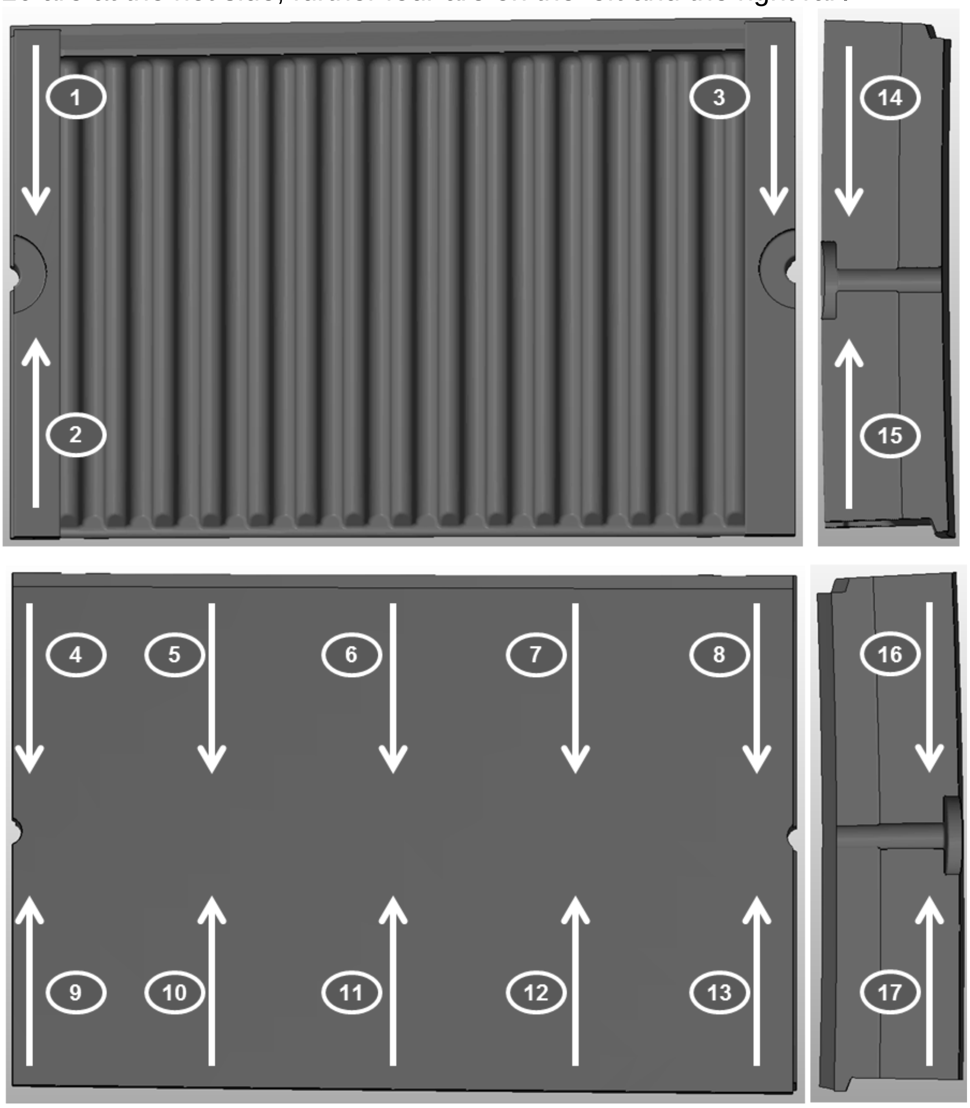

Figure 4.6j: Position of roughness measuring areas of the GE Demonstrator #1; 1-3) cold side; 4-13) hot side; 14-15) left rail; 16-17) right rail

The measurement results of the roughness in as build conditions can be found in Figure 4.6k. The average roughness Ra of the four faces of all four manufactured sub-segments is between 14 µm and 19 µm. Changes in the roughness of the four manufactured sub-segments result from their different orientation in the SLM process chamber during SLM manufacturing. After applying HIP further change of roughness have not been observed.

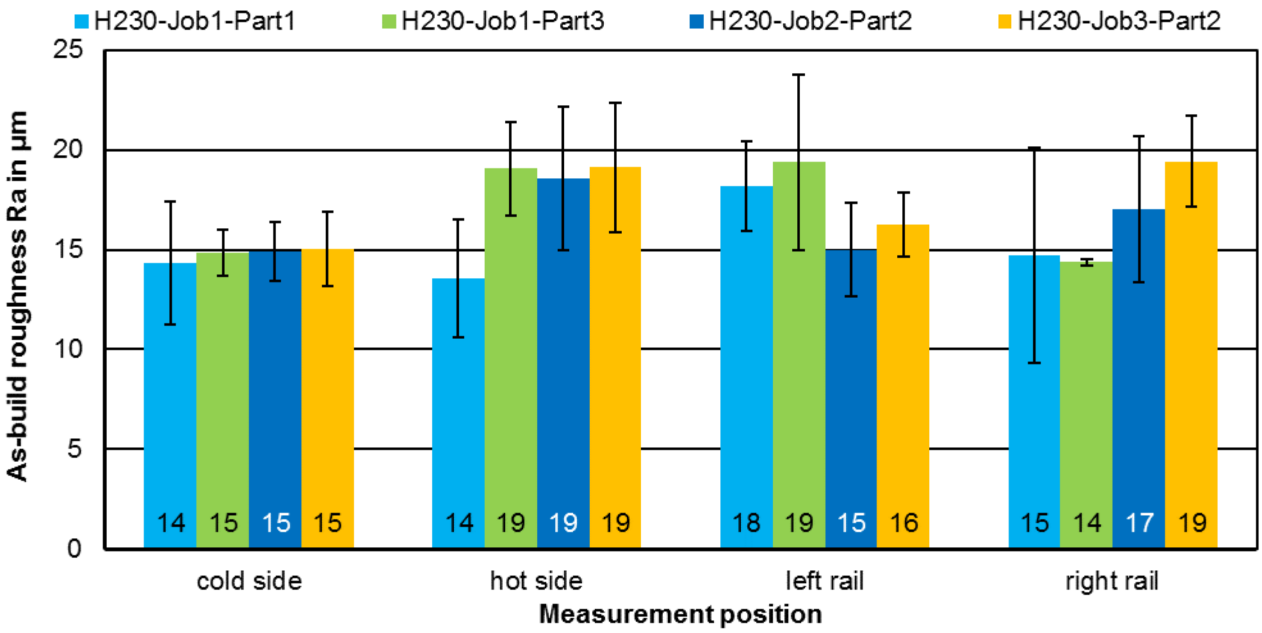

Figure 4.6k: Position of roughness measuring areas of the GE Demonstrator #1; 1-3) cold side; 4-13) hot side; 14-15) left rail; 16-17) right rail

Benchmarking results – Computer tomography (CT)

In as build conditions all three sub-sections show a significant amount of porosity. The maximal pore size in as build conditions is 2.0 mm. Sub-section 2 shows in addition several layer defect.
Sub-section 1 shows in as build conditions a large number of pores with a maximal pore size of 0.6 mm, see Figure 4.6l. After HIP, most of the pores disappear. Only one pore is still present after HIP, compare Figure 4.6m.

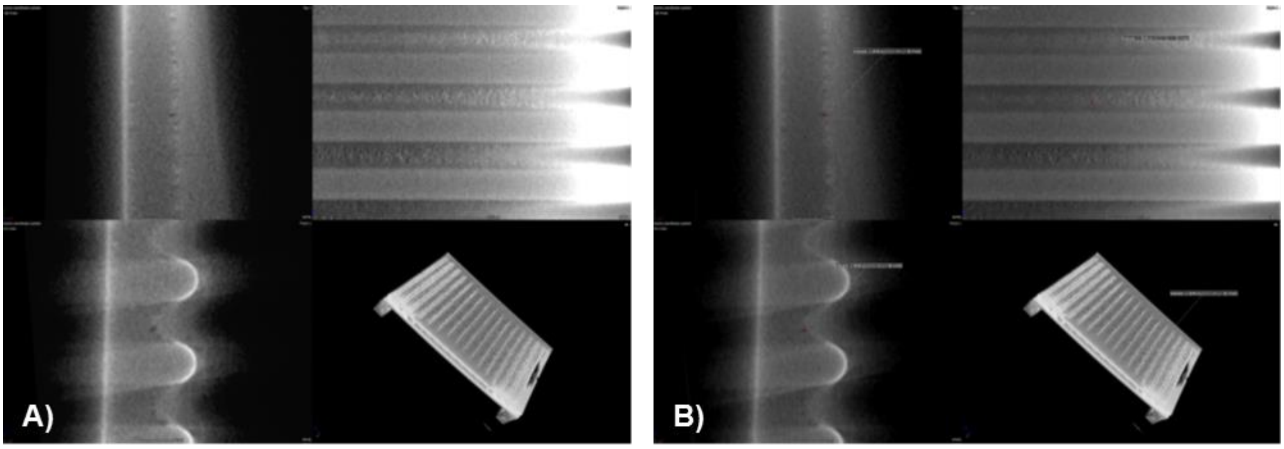

Figure 4.6l: GE Demonstrator #1, sub-section 1;
A) as build conditions; large number of pores, max size 0.6 mm
B) after HIP; Indication still present after HIP

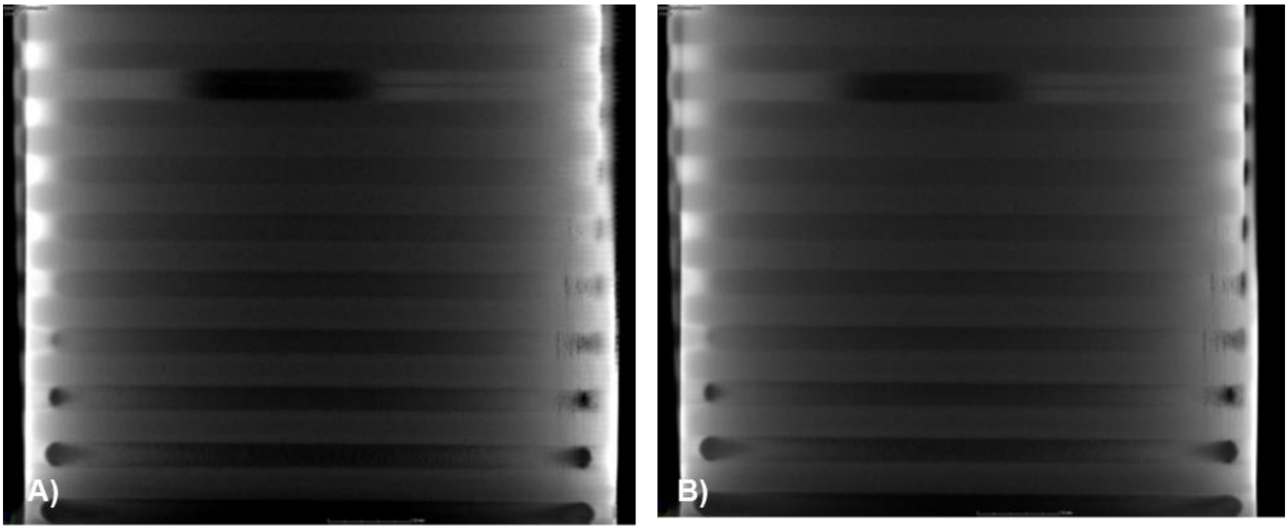

Figure 4.6m: GE Demonstrator #1, sub-section 2;
A) as build conditions; Several layer defects and porosity
B) after HIP; Layer defects still present, but pore disappeared

Figure 4.6n Left: represents a key outcome of the thermal cycling trial carried out at GE showing the phase response of an embedded Fabry-Perot type sensor installed on one of the two manufactured and tested heat shield components.

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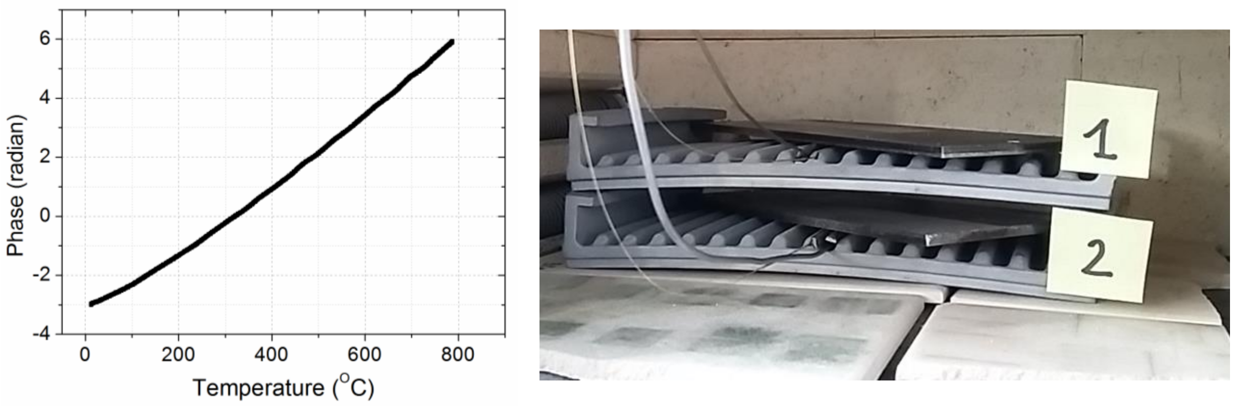

Figure 4.6n: Left Phase response of a F-P sensor embedded in a GE heat shield component under thermal cycling from ambient to 800 °C. The phase scales as a 2nd order polynomial with temperature. (Phase = -3.16 + $9.0 \cdot 10^{-3} \cdot T$ + $3.60 \cdot 10^{-6} \cdot T2$; confidence value R^2=0.9998); Right: Heat shields installed in furnace for temperature testing to 800°C

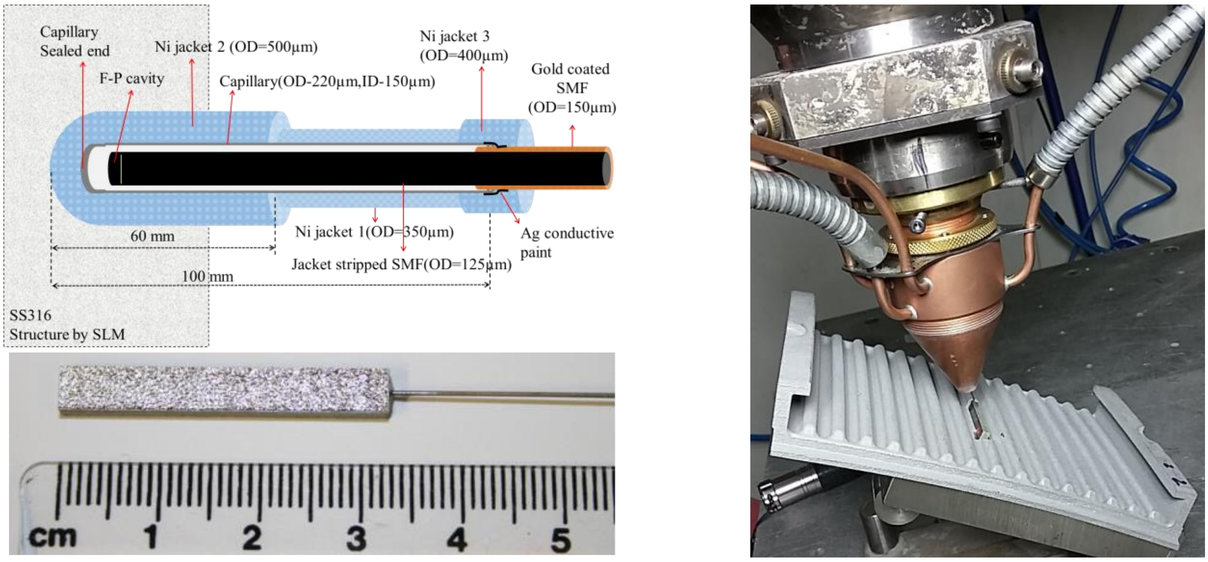

Figure 4.6o: Left top: Fabry-Perot sensor configuration; Left bottom: Completed SLM manufactured sensor coupon with integrated F-P sensor prior to installation onto the GE heat shield; Right: installation of sensor coupon onto heat shield by LMD at TWI

Figure 4.6o illustrates the Fabry-Perot sensor concept, the completed F-P sensor prior to installation and the installation process using the hybrid processing strategy developed in WP2 and employed here in WP6. The overall process leading to a completed sensor assembly is in its current manual, laboratory based assembly a highly complex procedure as outlined in the bulleted list **Error! Reference source not found.**, hence it was only possible to manufacture 2 sensors and install them on the supplied and design adapted heat shield in the described multi stage process.

4.6.3 Potential Impacts and Exploitation

From the perspective of the end users in the gas turbine industry the state-of-the-art was progressed immensely since OXIGEN resulted in providing a solution for using a material in the hot section of a gas turbine which was processed by means of Selective Laser Melting. Previously, this made the use of so-called gamma-prime hardening material mandatory such as IN738, IN939, etc. These materials, however, are not suitable for Selective Laser Melting since their respective properties depend fully on their gamma-prime precipitations which will only develop if the melted material is cooled down appropriately. In a Selective Laser Melting process the cooling rates are magnitudes higher than required.

However, using the ODS variant of IN625 as developed in OXIGEN now allows to produce hot section components with Selective Laser Melting as this variant reduces the oxidation rate by a factor of approx. 20 compared to conventional IN625.

In-situ condition (strain) monitoring in metals at elevated temperatures in the order of 700°C has the potential to disrupt metrology and process monitoring capabilities in many technological areas. For example in the case of super saturated steam systems for power generation, the technology could be used for the detection of stress corrosion cracking [SCC]. The ability to detect SCC during operation will enable the safe and effective operation of power generation systems at ever higher temperatures, thus enhancing the thermos dynamic efficiency of the plant, and mitigate against unexpected shutdowns.

The key element to commercial and technological exploitation for such applications is the patented process (applied for) enabling the embedment of fibre strain sensors in a compressed state. HWU will drive forward the exploitation of this technology by continuing to work on refinement and improvement of the technology in collaboration with relevant industry partners. Initial links with Doosan Babcock have been established. We will also continue to work with TWI, our partner in 'OXIGEN', to utilise dissemination to their network of members through workshops and industry relevant publications.

Within 'OXIGEN' the outcomes of WP2 have primarily been demonstrated in two end user components which were successfully tested at GE's facilities and future exploitation and use within GE is under review. HWU, the lead in WP2 will maintain links with GE and support their future in-house exploitation.

SLM manufacturing technology, used for the embedding of fibre optic sensors, by itself requires optimisation and more fundamental research to identify and mitigate processes leading to intrinsic stresses present in manufactured components. Research in WP2 has shown that embedded fibre strain sensors can provide an enhanced level of understanding of the SLM manufacturing process and HWU will develop the technology in collaboration with Inspire and TWI, 2 of our 'OXIGEN' partners and Renishaw, a strategic research partner to HWU and a leading manufacturer of SLM systems and metrology instrumentation.

5 Project Impact, wider social implications and exploitation and dissemination

5.1 Project Impact

Within the project duration 20 publications have been published:

- OXIGEN project website,
- 10 conferences,
- 1 conference poster,
- 2 abstract,
- Journals,
- 2 PhD Thesis,
- 1 IP application has been submitted → publication planned on or shortly after 29 June 2018.

Currently further 9 publications are planned for 2017ff.

5.1 Website

The project website is available at http://www.oxigen-project.eu/. It allows the public dissemination of the OXIGEN programme and the controlled exchange of documents between beneficiaries via a secure login page.

5.2 Journal articles

The publication of research articles is essential for academic beneficiaries and R&D departments of industrial beneficiaries as this primarily contributes to the impartial evaluation of the research work.

The list of the published and submitted papers sorted by release date in tabulated in appendix A1.

5.3 Conference participations

Conference participations included preferential oral presentations, poster presentations and proceedings. Many conference papers were reviewed according to conference regulations. All of the OXIGEN beneficiaries are main authors regarding the scientific and technical themes addressed in the conference.

The list of dissemination activities generated by the OXIGEN project is tabulated in appendix A2.

5.4 Eastern European Partnership Co-operation (SICA)

"Eastern European Partners involved in the Oxigen project have an enormous academic and industrial expertise in their field of technology. They deliver high quality results on time. Unfortunately due to customs it was not easy to send material to the Eastern European Partners". ***GE***

"PMI and IVCHECNO became integral to the development of OXIGEN technologies and were key to the successes of the project. Participation has also given new opportunity for collaboration in future projects" ***INSPIRE***

"The OXIGEN Project has given valuable collaboration and interest / appreciation of aligning materials testing to different standards and ways of thinking" ***TWI***

“We have acquired new knowledge and experience in the field of processing of composite powders IN 625, In 718, the Hay 230 (compaction (traditional pressing, explosive compaction), vacuum sintering, thermobaric sintering, HIP) and SLM samples using vacuum sintering and HIP (100% density). This will enable new collaboration with Ivchenko and other partners post project.“ ***PMI***

The inclusion of Eastern European partners allowed new insights on behalf of scientific and technical approach since many of their products, materials and methods were developed independently over the course of decades. This added good overall value to the project.” ***SIEMENS***

“The involvement of Ivchenko in the OXIGEN project has opened up new ideas and opportunity for the inclusion of AM technology in outrdesign and development division of our business” ***IVCHECNKO***

“The shipment of development samples and powders to/from Eastern partnership countries was very problematic due to local export/import restrictions. In some cases this resulted in shipments being returned to source. This add delays to project timelines.” This needs to be addressed in future opportunities for collaboration. ***All Partners***

Appendix A1

Title	Author(s)	Title of the periodical or the series	Number, date or frequency	Publisher	Date of publication	Relevant pages
Influence of Nb and Mo on microstructure formation of rapidly solidified ternary Ti–Al-(Nb, Mo) alloys	C. Kenel, C. Leinenbach	Intermetallics	Vol. 69	Elsevier Limited	01/02/2016	82-89
Temperature and Strain Measurements with Fibre Bragg Gratings Embedded in Stainless Steel 316	D. Havermann, J. Mathew, W.N. MacPherson, R. Maier, D. Hand	Journal of Lightwave Technology	33/12	Institute of Electrical and Electronics Engineers Inc.	02/01/2015	2474-2479
In-Fibre Fabry-Perot Cavity Sensor for High Temperature Applications	J. Mathew, O. Schneller, D.Polyzos, D. Havermann, R. Carter, W.N. MacPherson, D. Hand, R.R.J. Maier	Journal of Lightwave Technology	33/12	Institute of Electrical and Electronics Engineers Inc.	02/01/2015	2419-2425
3D printed metal parts with integrated fiber optic sensors for temperature monitoring up to 1000 °C	J. Mathew, D. Havermann, D. Polyzos, W. N. MacPherson, D. P. Hand, R. R. J. Maier	Smart Materials and Structures		Institute of Physics Publishing	05/10/2015	
Oxidation of ODS Nickel Based Alloys	K. Arnold	Materials at High Temperatures		Science Reviews Ltd	19/09/2015	
Influence of Nb and Mo on microstructure formation of rapidly solidified ternary Ti-Al-(Nb, Mo) alloys	C. Kenel, C. Leinenbach (EMPA)	Intermetallics		Elsevier Limited	27/08/2015	
A novel oxide dispersion strengthened β-solidifying γ-TiAl alloy for powder-based processing technologies	C. Kenel, (EMPA) K. Dawson, C. Hauser, G. Dasargyri, T. Bauer, A. Colella, A.B. Spierings, G. J. Tatlock, C. Leinenbach	Materials and Design		Elsevier BV	21/09/2015	
Microstructure and oxide particle stability in a novel ODS γ-TiAl alloy processed by spark plasma sintering and laser additive manufacturing	C. Kenel, (EMPA) K. Dawson, C. Hauser, G. Dasargyri, T. Bauer, A. Colella, A.B. Spierings, G. J. Tatlock, C. Leinenbach	Acta Materialia		Elsevier Limited	16/08/2016	
ODS γ-TiAl alloy processed by spark plasma sintering and laser additive manufacturing – Part II. Mechanical performance and oxidation resistance	C. Kenel (EMPA), A. Lis, K. Dawson, M. Stiefel, C. Pecnik, J. Barras, A. Colella, C. Hauser, G.J. Tatlock, C. Leinenbach, K. Wegener	Materials and Design		Elsevier BV	26/01/2017	
Selective laser melting of an oxide dispersion strengthened (ODS) γ-TiAl alloy towards production of complex structures	C. Kenel (EMPA), G. Dasargyri, T. Bauer, A. Colella, A.B. Spierings, C. Leinenbach, K. Wegener	Materials and Design		Elsevier BV	26/01/2017	

Appendix A2

Type of activities	Main leader	Title	Date	Place	Type of audience	Size of audience	Countries addressed
Oral presentation to a scientific event	TWI LIMITED	Project Overview	14/04/2014	AILU Workshop, Sheffield, UK	Scientific community (higher education, Research)		UK
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	Optical Fibre Sensing in Metals by Embedment in 3D Printed Matallic Structures	05/06/2014	Invited Paper, 23rd International Conference on Optical Fiber Sensors, Proc. of SPIE Vol 9157, 9157	Scientific community (higher education, Research) - Industry	450	International
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	High Temperature Sensor Based on an In-Fibre, Fabry-Perot Cavity	05/06/2014	23rd International Conference on Optical Fibre Sensors, Proc. of SPIE Vol. 9157, 91578L (2014)	Scientific community (higher education, Research) - Industry	450	International
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	In-situ Measurements with Fibre Bragg Gratings embedded in Stainless Steel?	05/06/2014	23rd International Conference on Optical Fibre Sensors, Proc. of SPIE Vol. 9157, 9157A1 (2014)	Scientific community (higher education, Research) - Industry	450	International
Oral presentation to a scientific event	THE UNIVERSITY OF LIVERPOOL	Microscopy of an ODS Ni based superalloy MA754 oxidised at 900°C	14/04/2014	9th International Microscopy of Oxidation conference	Scientific community (higher education, Research)	90	International
Oral presentation to a scientific event	INSPIRE AG FUR MECHATRONISCHE PRODUKTIONSSYSTEME UND FERTIGUNGSTECHNIK	Microstructure and mechanical behaviour of SLM processed H230	15/08/2015	Solid Freeform Fabrication Symposium	Scientific community (higher education, Research) - Industry	500	USA, International
Oral presentation to a scientific event	INSPIRE AG FUR MECHATRONISCHE PRODUKTIONSSYSTEME UND FERTIGUNGSTECHNIK	Presentation of the OXIGEN project	15/08/2015	Solid Freeform Fabrication Symposium	Scientific community (higher education, Research) - Industry	500	USA, International
Oral presentation to a scientific	HERIOT-WATT UNIVERSITY	SS316 structure fabricated by selective laser melting and integrated with strain	12/10/2015	24th Int Conf on Optical Fibre Sensors, Brazil	Scientific community (higher education, Research) - Industry	400	International

event		isolated optical fiber high temperature sensor					
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	Measuring Residual Stresses in metallic components manufactured with Fibre Bragg Gratings embedded by Selective Laser Melting	12/10/2015	24th Int Conf on Optical Fibre Sensors, Brazil	Scientific community (higher education, Research) - Industry	400	Internationa l
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	Stainless steel component with compressed fiber Bragg grating for high temperature sensing applications	28/01/2016	European Workshop on Optical Fiber Sensors, EWOFS2016	Scientific community (higher education, Research) - Industry		Internationa l
Oral presentation to a scientific event	EIDGENOSSISCHE MATERIALPRUFUNGS- UND FORSCHUNGSANSTALT	Design of an ODS-TiAl alloy for additive manufacturing technologies	27/10/2016	Materials Science & Technology 2016 in Salt Lake City	Scientific community (higher education, Research) - Industry		Internationa l
Oral presentation to a scientific event	THE UNIVERSITY OF LIVERPOOL	High Temperature Oxidation Behaviour of Oxide Dispersion Strengthened (ODS) Derivatives of two commercial nickel-based Superalloys	05/05/2016	9th International Symposium on High-temperature Corrosion and Protection of Materials in Les Embiez	Scientific community (higher education, Research) - Industry		Internationa l
Oral presentation to a scientific event	INSPIRE AG FUR MECHATRONISCHE PRODUKTIONSSYSTEME UND FERTIGUNGSTECHNIK	Embedding fibre optical sensors into SLM parts	05/08/2016	Solid Freeform Fabrication Conference, Texas	Scientific community (higher education, Research) - Industry		Internationa l
Websites/ Applications	TWI LIMITED	LMD of ODS TiAl Alloys for Power Generation Applications	15/11/2016	TWI website	Scientific community (higher education, Research) - Industry - Civil society - Policy makers - Medias		Internationa l
Oral presentation to a scientific event	HERIOT-WATT UNIVERSITY	Integrating fibre Fabry-Perot cavity sensor in to 3D printed metal components for extreme high temperature monitoring applications	09/01/2017	IEEE Sensors 2017	Scientific community (higher education, Research) - Industry		USA, Internationa l