# Publishable Executive Summary

## Background:

IMPRESS is an acronym for Intermetallic Materials Processing in Relation to Earth and Space Solidification and it is a 41M€ *Integrated Project* that involves **40 organisations from across Europe and Russia**. The 5-year project, under the coordination of the European Space Agency, was selected in 2004 under Priority 3 of the Sixth Framework Programme: Nanotechnologies and nanosciences, knowledge-based multifunctional materials, new production processes and devices - "NMP".

In view of the many scientific and technical facets of this project, **a multidisciplinary team of 150 researchers** has been assembled - with expertise spanning the fields of physical metallurgy, chemistry, metrology, fluid science, space experimentation, computer modelling, environmental engineering and industrial product development relevant to turbo-machinery, industrial catalysts and fuel cells.

***The geographic distribution of IMPRESS partners, across Europe and Russia.***

In contrast to normal alloys, intermetallics are defined as ordered chemical compounds of two or more metals. Within IMPRESS, two specific families of intermetallics are under study, namely titanium aluminides (TiAl) and nickel aluminides (NiAl).

TiAl intermetallics have remarkable mechanical and physical properties at temperatures of up to about 800ºC. The combination of high melting point, high-temperature strength and creep resistance and low density makes TiAl ideal for high-performance gas turbine blades.

NiAl intermetallics, on the other hand, have good catalytic properties making them particularly useful for numerous hydrogenation reactions in the chemical industry, as well as electro-catalysts in alkaline fuel cells.

## Summary of Objectives:

The primary scientific objective of IMPRESS is to understand the critical links between the solidification processing of intermetallic alloys, the structure of the material at the micro- and nanoscale, and the final mechanical, chemical and physical properties. In terms of industrial applications of intermetallics, the team decided to focus on (i) gas turbine blades, and (ii) catalytic devices. The technical objectives are thus to develop, produce and test novel intermetallic alloys for these two strategic applications:

- high-quality 40cm-long investment-cast and heat-treated $\gamma$-TiAl gas turbine blades for aero-engines and power generation turbines;
- improved catalytic sponge Ni powders, based on Ni-Al precursors, with a range of particle sizes (both nanometric and micrometric) for use in industrial hydrogenation reactions and alkaline fuel cell electrodes.

**Work Performed and Results:**

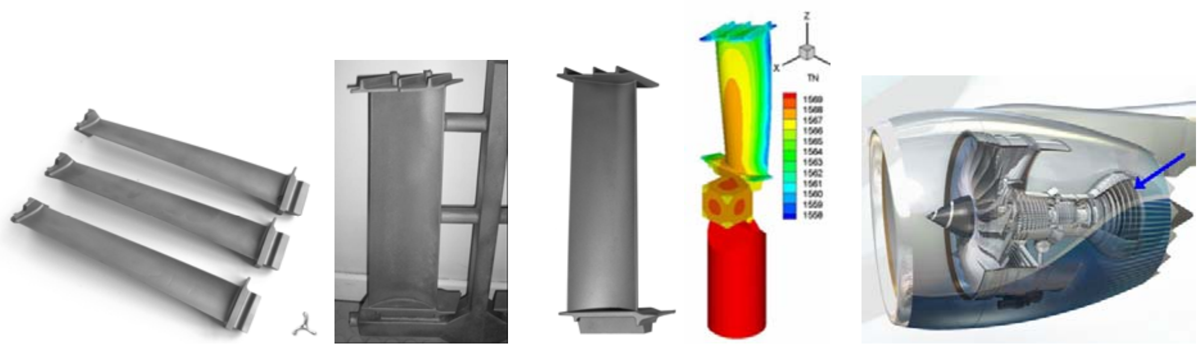
***(1) γ-TiAl Turbine Blades***

At the start of the IMPRESS Project in 2004, most turbine and aero-engine producers had attempted to produce and test γ-TiAl intermetallic blades. Some efforts were devoted to forging and others to casting processes. However, in all cases, quality was poor, process yield was low and any blades made were very expensive. In addition, most attention was given to first-generation lower-strength alloys, like Ti-48Al-2Cr-2Nb.

Within this rather unsatisfactory backdrop, the decision was made at the outset of IMPRESS that turbine blades must be produced by **cost-effective investment casting**, as opposed to the more costly forging approach. However, due to the reactivity of liquid TiAl at 1700°C, numerous problems in casting were highlighted, such as melt/crucible reactions, poor surface finish and porosity. Furthermore, the ductility of cast γ-TiAl components was shown to be intrinsically low, thus highlighting the need for further alloy development. The work within IMPRESS has thus been targeted in these **two linked but separate areas**; firstly the development of casting technology and secondly the development of cast blades that would meet the end-user specifications, including a minimum room-temperature ductility of 1%.

The experimental work within these two areas has been supplemented by comprehensive **multi-scale modelling** which has required basic properties to be obtained. Accurate **thermodynamic phase diagrams** are now in place for all alloys studied including the quaternary system Ti-Al-Nb-Ta, and relevant binary and ternary subsystems. In addition, accurate **thermophysical property databases** for liquid intermetallics have been created, for the first time, with the help of benchmark **electromagnetic levitation experiments carried out in space**. These data are essential for understanding, optimising and modelling the different blade production steps; such as melting, mould-filling, heat transfer, solidification and subsequent heat treatment processes. The success of the experimental programme has also been assured by comprehensive **measurements of the mechanical and oxidation properties** of test-pieces produced from castings of the selected TiAl intermetallic materials.

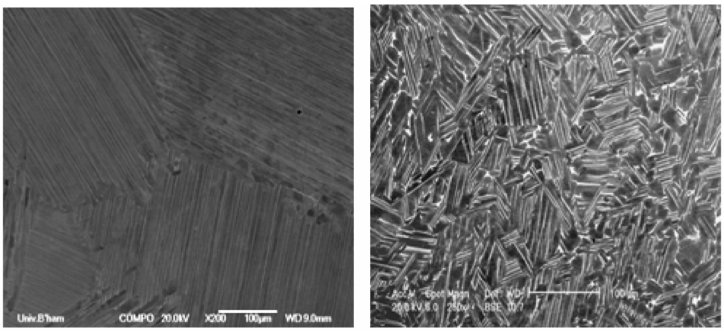
Investment casting of low-pressure γ-TiAl turbine blades up to 60cm in length, has been successfully carried out using **centrifugal casting,** and up to 40cm using **tilt casting techniques**. Examples of blades produced during IMPRESS are shown in Figure 1a, b and c. Several hundred γ-TiAl test-samples have been used to assess the tensile, creep, fatigue and fracture toughness properties and oxidation resistance of the cast and heat-treated samples. From a detailed assessment, it appears that the manufacturing technology developed within IMPRESS, supplemented by studies of the solidification fundamentals, have led to a **major improvement in the quality and yield of net-shape γ-TiAl castings.**



(a) (b) (c) (d) (e)

**Figure 1: γ-TiAl-based low-pressure turbine blades centrifugally-cast at (a) ACCESS and (b) SETTAS-Doncasters, and tilt-cast at (c) IRC-Birmingham. (d) shows an example of heat and flow modelling applied to blade production (Greenwich University). (e) shows the end-user aero-engine turbine application (Rolls-Royce).**

In terms of materials, the two intermetallic compositions selected after an extensive alloy development programme are Ti-46Al-8Nb and Ti-46Al-8Ta (at%). Addition of the niobium or tantalum permits the **heat treatment**, developed within IMPRESS, to be carried out on samples up to at least 20mm in diameter. The heat treatment leads to the cast samples being transformed massively through thickness, after which the material is HIPped (hot isostatically pressed). In turn, this produces a complex **“convoluted” microstructure,** which is illustrated together with the coarse as-cast microstructure in Figure 2. It is this special convoluted microstructure and its grain refinement that confers the excellent balance of mechanical properties to the turbine blade.




(a) (b)

**Figure 2: Back scattered scanning electron micrographs of samples of Ti46Al8Ta (a) as-cast and (b) cast and air cooled from 1360°C and subsequently HIPped at 1260°C for 2h illustrating the microstructural refinement that is achieved via this heat treatment** [Courtesy: IRC-Birmingham].

As an example of mechanical properties at room temperature, the Ti-46Al-8Ta alloy has: 0.2% proof stress of over 620 MPa, tensile ductility of over 1%, fracture toughness of about 16 $MPam^{1/2}$ and fatigue testing shows lifetimes of more than 100,000 cycles for stresses up to 660 MPa. Creep life at a stress of 350 MPa at 700°C is found to be over 4000 hours. Oxidation resistance is also satisfactory for these alloys. Data so far indicates that the **selected γ–TiAl intermetallics meet all specifications**, defined by the end-user.

In view of the success of this work, it is now becoming apparent that there are numerous other areas that could also benefit from lightweight high-strength intermetallics. Although, this project investigated LP turbine blades, it is conceivable to use these lightweight **γ–TiAl** in other parts of a gas turbine including high-pressure compressors, stator vanes, centrifugal impellers, as well as large turbochargers.

With this wide variety of applications in mind, it was deemed necessary to develop a **commercially-viable recycling route** for scrap material and out-of-service components. In this regard, a new melting and de-oxidation approach has been developed that produces large recycled γ–TiAl ingots (example shown in Figure 3) with very low oxygen and inclusion levels. This recycling route is the only one of its kind in the world, and is now ready for industrial scale-up. In addition, a **new low-cost alternative for ingot production**, based on consolidated elemental blocks (CEB), has been developed from scratch.

**Figure 3: Recycled ingot of γ–TiAl produced in IMPRESS.**
[Courtesy: IME-RWTH]

### *Top Ten Results for γ-TiAl Turbine Blades*

In summary, IMPRESS has resulted in:

- (i) high-yield cost-effective net-shape casting processes (both centrifugal and tilt),
- (ii) new high-temperature capable γ-TiAl alloys (viz. Ti46Al8Nb and Ti46Al8Ta),
- (iii) patented heat treatment process for grain refinement of cast γ-TiAl material,
- (iv) longer lasting yttria slurries and lower cost zirconia moulds,
- (v) multiple VAR ingots for large-scale industrial melting up to 1 tonne,
- (vi) novel recycling process for γ-TiAl casting scrap and out-of-service blades,
- (vii) multi-scale modelling capabilities for all turbine blade manufacturing steps, leading to a commercial software package developed by Calcom-ESI,
- (viii) thermodynamic and thermophysical property databases for Ti-Al-(Nb,Ta), aided by world-unique experiments in microgravity and using synchrotron X-rays,
- (ix) first-ever mechanical property databases for both Ti46Al8Nb and Ti46Al8Ta,
- (x) completion of an industrial life-cycle, cost-benefit and supply-chain analysis for γ-TiAl turbine blade production and in-service use.

With this extraordinary combination of know-how, this class of TiAl alloy can now result in a **40-50% weight reduction for low-pressure turbine stages,** compared with conventional nickel superalloys. If successfully implemented by industry, such a significant weight reduction would ultimately lead to improved thrust-to-weight ratios of aero-engines, higher efficiency, reduced fuel consumption and lower exhaust emissions. Industrial gas turbines also stand to benefit with increased disc lives. The next stage beyond IMPRESS will be focused on industrial scale-up, validation testing and supply chain management, to ensure that **European industry can benefit from these major technological and environmental advances.**

## *(2) Sponge Ni Catalysts*

Sponge nickel catalysts, based on NiAl precursors, have been in industrial use for over 80 years [M. Raney patents: US1563587 from 1925, and US1628190 from 1927] for many industrial reactions, such as hydrogenation of butyraldehyde, acetone, nitrobenzene, adiponitrile, ethylbenzene and citral - to name just a few. Despite this long history of industrial use, there are limitations to the traditional process. Normally these catalysts are prepared by casting and crushing ingots of Ni-50wt%Al intermetallic, followed by caustic leaching in sodium hydroxide (NaOH) to remove aluminium atoms and create a large and catalytically-active surface area, typically 50-80 $m^2$ per gram. In the case of cast-and-crushed material, the microstructure of the precursor material is an equilibrium one, which does not lend itself to further modification. However, by adopting **gas atomisation** instead, one can produce rapidly-solidified spherical NiAl powder with non-equilibrium structures, an example of which is shown in Figure 4. In IMPRESS, well over **600 NiAl gas-atomised samples** were produced, tested and characterised by various groups.

As a result of many hundreds of catalytic trials in the IMPRESS programme, it has been realised that the **microstructure** of gas atomised powder is very important and can be tailored to give better catalytic performance after NaOH activation. Important powder features include: chemical composition, particle size, volume fraction of different phases, ratio of $NiAl_3$ / $Ni_2Al_3$ phases, fineness and tortuosity of the dendritic network, porosity, surface area, atomic roughness and the location of dopant elements at the catalytic surface. These features are related to the starting composition, the parameters of the gas atomisation process and the parameters of the caustic leaching process. For the first time in this field, **a selection map has emerged linking process, structure and final catalytic properties**.

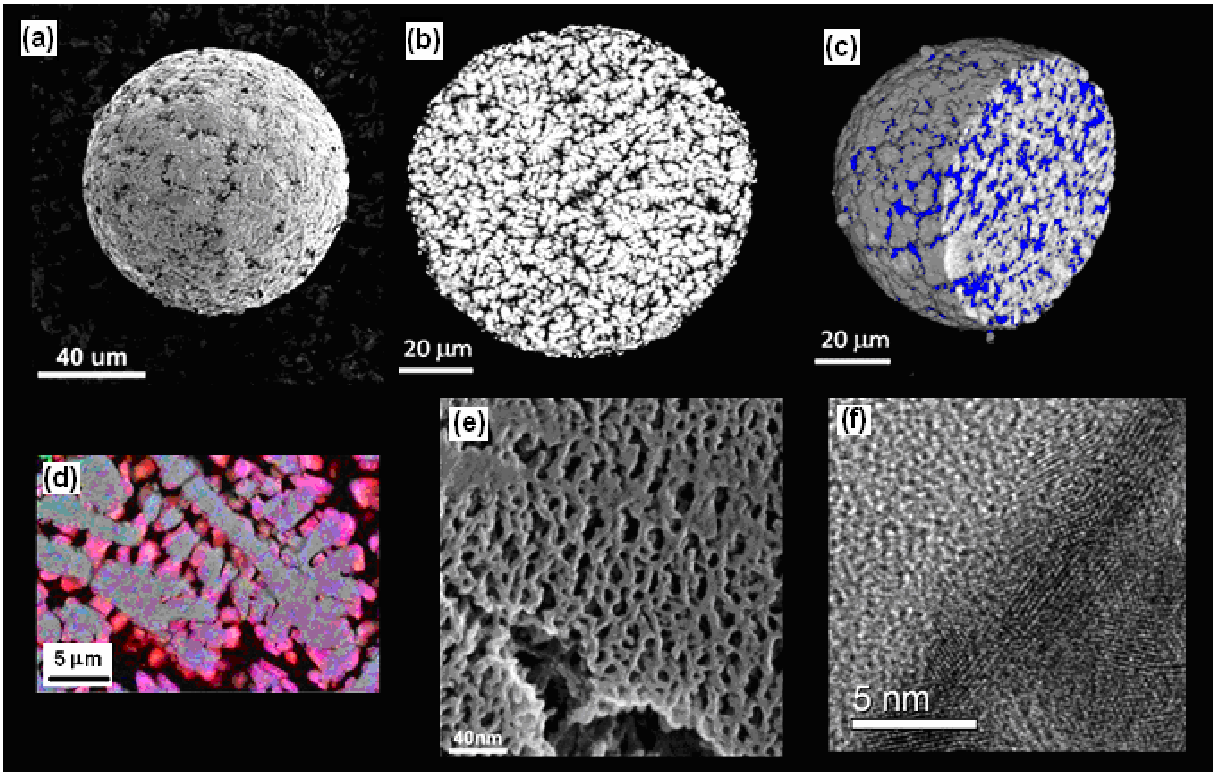



**Figure 4: Structural hierarchy across 6 orders of magnitude: (a) a single gas-atomised sponge Ni powder particle *after* caustic leaching, (b) a cross-sectional micrograph showing the internal dendritic microstructure, (c) a 3D X-ray microtomograph from ESRF showing the internal porosity and connectivity in blue, (d) a compositional map of a leached dendrite, (e) the nanoporous sponge comprising ≈5-10nm Ni crystallites, and (f) the nano-steps at the outer surface of the catalyst.** [Courtesy: University of Ulm, Zeiss, CERAM Ltd. and ESRF/ESA].

In terms of structure after leaching, the **complete hierarchy** has been elucidated and can be described as follows:

a) spherical gas-atomised particle (typically with a diameter of 100 μm);
b) fine 3D interconnected dendritic microstructure (with dendrites of the order 10 μm);
c) individual phases containing capillary-sized microscopic pores (1-5 μm wide);
d) mesoporous foam-like structure containing agglomerates of nano-Ni crystallites (typically with a diameter of 5-10 nm);
e) monolayer atomic steps (0.2 nm in size) on the surface of the nano-Ni crystallites.

Thanks to the efforts of computer modellers, many of these multi-scale structures are now being predicted too. Figure 5 shows an example of Metropolis Monte Carlo modelling of the leaching process generating a nanoporous structure of spongy Ni. The properties of the material, namely pore size, tortuosity, surface area and atomic composition at the surface are all in good agreement with the experimental data (see again Fig. 4(e)).

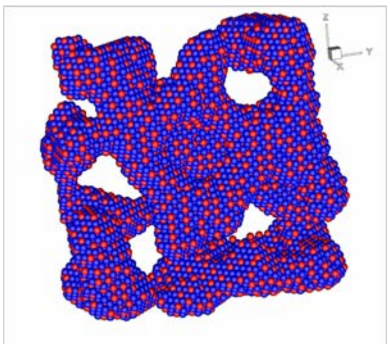


**Figure 5: Computer simulation of the Ni-Al leaching process to produce nanoporous sponge Ni (Al-red Ni-blue).**
[Courtesy: Swansea University]

Similar to the turbine side of the project, a unique database of phase diagrams and thermophysical properties has also been obtained for the Ni-Al-X systems of interest. Again, the contribution of **space experiments** has been very helpful in understanding the **solidification fundamentals of Ni-Al** as a function of undercooling and measuring its **thermophysical properties** as a function of temperature – all of great benefit to modellers.

In terms of enhanced performance, the IMPRESS team has identified a number of atomised NiAl precursors with extremely high catalytic activity. The **best promoted Ni catalysts are an order of magnitude better than the standard Raney Ni composition**, for various industrially important chemical reactions. Encouragingly, some useful improvements in selectivity and stability are also observed. It is the novel ability to tailor the micro/nanostructure that clearly differentiates the gas-atomised product from traditional cast-and-crushed catalysts.

In addition to the hydrogenation testing (Figure 6a), IMPRESS-made sponge nickel has been extensively tested in alkaline fuel cells (Figure 6b). Very promising results have been found in this application area too, since sponge nickel provides excellent catalytic performance as part of the anodic electrode. In fact, this Ni powder has **performance that is better than conventional platinum/palladium catalysts**, at a fraction of the cost, and for well over 2000 hrs of operation. Commercially, this offers the opportunity to bring alkaline fuel cells to the market at a more affordable and reliable level than ever before.

**(a)** **(b)**

**Figure 6: (a) Slurry-phase hydrogenation reactors, and (b) commercial alkaline fuel cell.**
[Courtesy: Johnson Matthey and Hydrocell Oy]

### *Top Ten Results for Sponge Ni Catalysts*

In summary, IMPRESS has resulted in:

- (i) a cost-effective gas atomisation and leaching process for commercial sponge nickel production,
- (ii) new precursor Ni-Al-X alloy compositions with specialised dopants,
- (iii) doped sponge Ni catalysts with 10x better catalytic activity c.f. standard commercial Raney-type nickel, as well as selectivity/stability improvements,
- (iv) performance in alkaline fuel cells equivalent or better than Pt/Pd electrocatalysts,
- (v) extensive characterisation of sponge nickel using techniques such as atomic-resolution He-ion and electron microscopy, XPS, X-ray nanotomography, neutron diffraction, Hg intrusion and $N_2$ adsorption,
- (vi) a centralised database of all project results, quantitatively linking process, composition, structure and catalytic performance,
- (vii) a totally new approach for producing long and aligned nanostructured Ni chains using inert gas condensation, made on ground and in *microgravity*,
- (viii) multi-scale modelling capabilities for all process steps, ranging from the macroscopic process level all the way down to the atomic level.
- (ix) thermodynamic, kinetic and thermophysical property databases of Ni-Al-X, aided by world-unique experiments in microgravity and at X-ray synchrotron sources,
- (x) completion of an industrial life-cycle, cost-benefit and supply-chain analysis for Ni-Al powder production and in-service use.

With this wealth of scientific and technical information, the team has been able to gas-atomise a large 200 kg batch of Ni-Al powder for scale-up trials in an industrial hydrogenation reactor and fuel cells. By using such enhanced lower-cost catalysts, it is expected that there will be **significant savings for the chemical and energy sectors in terms of cost, time, energy and $CO_2$ emissions.** In this regard, IMPRESS has made a good scientific and technical contribution towards **"sustainable chemistry"** in Europe.

**Impact of IMPRESS after 5 Years:**

As a final conclusion, IMPRESS has made substantial progress on all fronts. The wealth and overall consistency of data has led to some profound insights into the various TiAl and NiAl intermetallics under study. The scientific knowledge has been well transferred to the technical developments, and this brings confidence that the new industrial prototypes will eventually be brought to the market by the suppliers and end-users. The EU impact of **lightweight and energy-saving materials** will of course be felt once the prototypes are in service and are able to reduce greenhouse gas emissions, principally $CO_2$.

The basic scientific knowledge generated from the project will have a **lasting impact on solidification processing and physical metallurgy**, mainly related to intermetallics, but also transferable to many other alloy systems (e.g. Ti, Ni, Cu, Fe, Zr etc). The demands of industry for better understanding, control and modelling of solidification processes have also been very well addressed by this project, such that commercial software is now in place.

**Technical prototypes have been successfully made and delivered to industry**, as promised in the original proposal. European industry now has the capability to produce 40cm low-pressure TiAl(Nb,Ta) turbine blades, via a cost-competitive manufacturing chain. These large net-shape cast aero-engine and IGT blades are the first of their kind in Europe, and probably also world-wide. Enhanced catalytic devices such as Ni-based hydrogenation slurries and hydrogen fuel cell electrodes have been produced and are now set for future testing and market exploitation.

**Further prototype development and testing** will be continued after the end of IMPRESS in 2010 by the industrial manufacturers and end-users, with the possible financial support of market-oriented funding schemes, for example EUREKA or regional agencies.

When referring to the state-of-the-art in 2004, Europe had a very fragmented and weak foothold in the field of intermetallics. However, it is fair to say that IMPRESS has completely reversed this situation by structuring and integrating the academic and industrial expertise across 16 countries. After 5 years, IMPRESS has now become the **world-leading "intermetallics" project**, unparalleled in the US, Japan or China.

Another positive outcome of the project has been the development of **spin-off projects**. Three new metallurgy projects have been conceived, as a result of IMPRESS research, and these are now being proposed to the EC for implementation in the coming years.

Finally, the success of the IMPRESS project has led to the establishment of a new R&D centre at the European Space Agency. The **ESA Creative Metals Centre** (CMC) will have the mandate to coordinate and carry out world-class metallurgical R&D of relevance to the Agency and other adjacent industrial sectors (e.g. aeronautic, chemical, energy, automotive, electrical, nuclear). In view of the growing requirement to reduce energy needs and the impact of climate change, a strong focus of the CMC will be placed on new and improved metals that can contribute to **low-emissions energy technology and lightweighting**, as promoted in the new EC SET-Plan (October 2009).

**Intentions for Dissemination and Use:**

The final year has witnessed a continued enthusiasm to disseminate interesting (non-confidential) information about the project; while at the same time protecting and exploiting any commercially-relevant IP, whenever necessary.

In terms of scientific dissemination, a **large body of original research** has come out of IMPRESS. This has been published mainly in the form of academic papers in international conference proceedings and high-level journals, including Intermetallics, Acta Materialia, Physical Review Letters, Nature Materials, Journal of Catalysis, MRS Bulletin, Surface Science etc. So far, over 140 IMPRESS-derived papers have been written. On average, this represents a published paper every 10 working days. The weighted impact factor of the IMPRESS-targeted journals is ≈2.7, which is higher the average for materials science (≈1). A number of best-paper awards have also been bestowed on the IMPRESS team at various international conferences.

Other prominent **dissemination activities** include: a permanent IMPRESS exhibition at the London Science Museum, web-based news stories e.g. “BBC World”, additional on-line scientific lectures, keynote lectures at conferences, special magazine articles in “Materials World” and “Scientific American”, presentations in local schools and an online IMPRESS Education Kit.

The types of audience that have been targeted in the dissemination plan range from school students to university students, fellow scientists, industry practitioners, and of course interested members of the general public.

Regarding IPR, **3 patent applications** have been registered related to alloy development, heat-treatment and ingot production. Another 2 patents are now under consideration in the final year. Such patents have commercial applications in the aerospace, power-generation and chemical sectors, and will be exploited by the patent applicants.

**The IMPRESS Project Website:** www.spaceflight.esa.int/impress

**Partners Involved:**

The following table lists the 40 academic and industrial partners that are involved in the IMPRESS Project. For clarity the 4-letter short codes, indicated in the table here, are used throughout this document.

| ***Full Name of Partner Organisation*** | ***Code*** |
|---|---|
| European Space Agency (Project Coordinator) | ESAE |
| Max-Planck Institut für Eisenforschung GmbH (D) | MPIE |
| University of Birmingham (UK) | IRCB |
| Institut National Polytechnique de Toulouse (F) | INPT |
| Helsinki University of Technology (FIN) | HUTF |
| Kungl Tekniska Högskolan (S) | KTHS |
| Slovak Academy of Sciences (SK) | IMSA |
| Research Institute for Solid State Physics and Optics (HUN) | SZFK |
| Deutsches Zentrum für Luft- und Raumfahrt (D) | DLRZ |
| British Ceramic Research Ltd. (UK) | CERA |
| University of Swansea (UK) | SWAN |
| University of Leeds (UK) | LEED |
| University of Greenwich (UK) | GREE |
| Calcom ESI S.A. (CH) | CALC |
| National University of Ireland (IRL) | NUID |
| ACCESS e.V. (D) | ACCE |
| Leibniz-Institut für Festkörper- und Werkstoffforschung (D) | IFWD |
| Ecole Polytechnique Fédéral de Lausanne (CH) | EPFL |
| Centre National de la Recherche Scientifique – Grenoble (F) | CNRS |
| Institute of Structural and Macrokinetics & Materials Science (RUS) | ISMA |
| Hydrocell Ltd. (FIN) | HYDR |

| | |
|---|---|
| Krakow University of Mining and Metallurgy (POL) | UMMK |
| Intrinsiq Materials Ltd. (UK) | QINL |
| Ufa State Aviation Technical University (RUS) | USAT |
| Fraunhofer Gesellschaft e.V. (D) | IFAM |
| Institut National Polytechnique de Lorraine (F) | INPL |
| Katholieke Universiteit Leuven (B) | KULB |
| CNR-IENI Milan & Genoa (I) | IENI |
| INASMET Foundation Ltd. (E) | INAS |
| NPL Management Ltd. (UK) | NPLM |
| Universiteit Leiden (NL) | LEID |
| Universität Ulm (D) | UULM |
| Institute of Chemical Problems for Microelectronics (RUS) | ICHP |
| Rolls-Royce Plc. (UK) | ROLL |
| ALD - Vacuum Technologies AG (D) | ALDV |
| CEMEF-Armines (F) | CEME |
| Université de Rouen (F) | ROUE |
| Doncasters Group Ltd. (B, UK, D) | DONC |
| Johnson-Matthey Plc. (UK) | JOHN |
| IME RWTH Aachen (D) | RWTH |

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- Last but not least, thanks are given to the independent reviewers that have taken the time to evaluate the IMPRESS project on an annual basis, including Prof. Rüdiger Bormann, Prof. Jacob Moulijn and Prof. Jeffery Brooks.