innovaTiAl

"Innovative processes and materials to synthesise knowledge-based ultra performance nanostructured PVD thin films on gamma titanium aluminides"

FP6 project: NMP3-CT-2005-515844

Final Publishable Report

- **Period covered**: from May 1, 2005 to October 31, 2009
- **Date of preparation**: 22/12/09
- **Start date of project**: May 1, 2005 Duration: 54 months
- **Project Coordinator Name**: Prof. Christoph Leyens
- **Revision**: Final version

Document Information

Document Name: Final Publishable Report, Month 54
ID: Publishable Report
Revision: Final Version
Revision Date: 091222
Author: Etienne Macron
Dissemination level: Confidential

Diffusion List

Consortium by PRODIGE

Approvals

	Name	Company	Date	Visa
Project Coordinator	Christoph Leyens	DLR	22/12/09	Approved
Quality Manager	Etienne Macron	ALMA	22/12/09	Approved

Document history

Revision	Date	Modification	Author
Version 0	11/12/2009	First version of the final publishable report	Etienne Macron
Version 1	22/12/2009	Final version of the report including the wording and layout revisions from DLR	Etienne Macron

Content

Section 1. Reminder of the Innovatial objectives

Innovative processes and materials to synthesise knowledge-based ultra-performance nanostructured PVD thin films on gamma tinanium aluminides

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1.1 Background and project objectives

Intermetallic γ-TiAl alloys are novel, promising materials for automotive, energy and aerospace applications. Their specific stiffness and strength, as compared to their low weight, potentially lead to large weight savings (50%) and therefore lower mechanical loads on components like turbines or engine parts. However, the major obstacles for a wider use of these materials are their sustainability to severe environmental attack in oxidising, sulphidising, and hot corrosion environments as well as insufficient wear and erosion resistance at elevated temperature (>650 °C). Protective coatings must therefore be developed to overcome these problems.

Similar concerns in terms of coating development have been observed on traditional substrates like cemented carbides, steels, iron cast) in several other manufacturing industries (cutting tools, machining industry, automation industry). Here, novel materials such as composites have to be machined with outstanding surface quality at high speed which require advanced tools with long lifetime and advanced performance.

While for γ–TiAl there is not one single coating system available today, coatings for machining tools have been widely used already. However, the current coatings provide only short term protection, typically not longer than 1 hour. The lifetime of the coatings will be substantially extended by the work performed within this project. The major emphasis of INNOVATIAL, however, is on the development and introduction of new coating systems for γ–TiAl which availability is believed to contribute to a quantum leap in automotive, energy and aerospace applications. In these sectors, there is a strong demand for coatings systems with lifetimes of several thousand hours at elevated temperatures.

The INNOVATIAL consortium is hence tackling the demand for coatings that can withstand attack up to 1000°C by complex environments and that can provide immunity against damage due to wear and erosion at high temperature for long term.

Technical and scientific objectives and innovative aspects of the INNOVATIAL project are to answer the strong demand from the industry for thin films ensuring long term protection of components (made of γ–TiAl and of additional substrates). This European project gathers all the necessary tools and personnel with a strong track-record to develop a robust structural design of unsurpassed thin films. The technical objectives of this project are to synthesise ultra-performance nanoscale-structured PVD thin films, which can provide environmental protection at high temperatures in the range from 650 up to 1000 °C: this will greatly boost the application of γ-TiAl to high service temperatures and long dwell times and this will improve lifetime of additional coated substrates. To reach this technical objective, this project will address the fundamental problems in the three main regions of the thin film/substrate system: the interface, the bulk, and the surface of the coating, in a coordinated manner. This project proposes to bring groups at the highest scientific level together to work on the entire system in contrast to the current narrower and discriminating approach.

Since the beginning of the project, among others, the following important technical and scientific achievements were made:

- Substantial improvement of interface engineering by Cr- and Y-containing interfaces;
- Etching conditions for hindered coating – substrate cross – diffusion found;
- Major progress in the development of Ti-free CrAlYCN/CrCN coatings for medium (650°C) and high (650-850°C) temperatures achieved;
- CrAlYN/CrN+CrAlYON coating systems show excellent performance in high temperature oxidation and tribological conditions;
- TiAlYN/CrN+ALOx coatings were successfully tested at 650°C for 2500 h without any sign of degradation;
- Intermetallic coatings showed good oxidation resistance up to 900°C;
- TBC lifetimes larger than 2000 h at 850°C on gamma TiAl achieved;
- Major progress in the understanding of coating microstructures, properties and degradation mechanisms achieved;
- Fatigue tests of reference and aged TiAl samples were successfully performed;
- Baseline creep tests at various temperatures and stress levels were carried out;
- First time observation of a reduction in friction at higher temperatures in a coating that does not contain vanadium but an oxy-nitride layer instead;
- Provision of a room temperature tribological test setup for "industrial conditions";
- Extensive thermodynamic modeling on INNOVATIAL coating compositions was performed;
- Training activities included a tutorial at international conference (ICMCTF 2007, San Diego), HIPIMS-ABS Days held at Sheffield Hallam University an undergraduate course on coatings and surface science as well as workshops on surface and structural characterization;
- Several papers have been published in journal and/or at conferences;
- Integration of MTU Aero Engines GmBH, a new aerospace partner was made so as to complete the panel of end-users' applications on which the developed materials and coatings will be tested in SP7 and SP8.
- Transfer of the R&D work towards real industrial applications through a well established collaboration between SP1 and SP7/SP8 (eg. NP gas turbine buckets and OSVAT engine valves, coated with CrAlYN/CrN nanoscale multilayer coating or Wolframcarb rolling dies and Sapes pushing rods for diesel engines coated with CrAlYN/CrN nanoscale multilayer coating)

1.2 General project objectives and relation to the State of the Art

Intermetallic γ-TiAl alloys are novel, promising materials for automotive, energy and aerospace applications. Their specific stiffness and strength, as compared to their low weight, potentially lead to large weight savings (50%) and therefore lower mechanical loads on components like turbines or engine parts. However, the major obstacles for a wider use of these materials are their sustainability to severe environmental attack in oxidising, sulphidising, and hot corrosion environments as well as insufficient wear and erosion resistance at elevated temperature (>650 °C). Protective coatings must therefore be developed to overcome these problems.

Similar concerns in terms of coating development have been observed on traditional substrates like cemented carbides, steels, iron cast) in several other manufacturing industries (cutting tools, machining industry, automation industry). Here, novel materials such as composites have to be machined with outstanding surface quality at high speed which require advanced tools with long lifetime and advanced performance.

While for γ–TiAl there is not one single coating system available today, coatings for machining tools have been widely used already. However, the current coatings provide only short term protection, typically not longer than 1 hour. The lifetime of the coatings will be substantially extended by the work performed within this project. The major emphasis of INNOVATIAL, however, is on the development and introduction of new coating systems for γ–TiAl which availability is believed to contribute to a quantum leap in automotive, energy and aerospace applications. In these sectors, there is a strong demand for coatings systems with lifetimes of several thousand hours at elevated temperatures.

The INNOVATIAL consortium is hence tackling the demand for coatings that can withstand attack up to 1000 °C by complex environments and that can provide immunity against damage due to wear and erosion at high temperature for long term.

The INNOVATIAL clearly goes far beyond the international state-of-the-art of coating development and provides leading-edge research in the following areas:

New knowledge and scientific understanding of thin films for various substrates including Ti-Al materials. The optimisation of thin film properties can be realised only with the development of the scientific understanding. This European project provides answers to the lack of understanding and control for both the pathways of the structure evolution in multi-component nitride films, the operation of minority additives and trace elements (e.g. Y), the intrinsic stress formation in nanoscale multilayers, the inter-diffusion (threatening the very constitution of the coating and the adhesion to the component), the origin of super-hardness, toughness and the deformation mechanisms that occur on nano-structured coatings, the oxidation mechanisms (that obviously consumes the coating), the process parameters as well as the new regime induced by the new HIPIMS process.

Innovative coatings: this project aims at making for the first time the successful application of three candidate coating types for high temperature oxidation protection. The coatings have their origin within the partnership. To reach this objective, INNOVATIAL partners will:

- develop an interface layer with an enhanced adhesion and with no cross diffusion and crack propagation to retain the fatigue properties of the base material.
- make robust Cr-Al-N and Ti-Cr-Al-N nanoscale multilayer systems or nanocomposites of Ti-Al-N system with engineered properties to suit Ti-Al components and their applications, develop superlattices (nanoscale multilayer) and nanocomposite coatings with self-healing, self-adapting mechanisms and ultra-performance at high temperatures (900 - 1000 °C), and explore intermetallic coatings based on TiAl-(X, Y) (X=Cr, Ag; Y=Y, Hf, Zr, W, Si). It is a further originality that partners will integrate computational material science (including Ab Initio calculations) to design the above new coating types, not previously available at companies.
- develop surface layer with combined enhanced resistance against environmental attack and excellent tribological properties (coeff. of friction $\mu < 0.8$). These surface layers will be based on innovative Me-oxy-nitride glazes or Thermal Barrier Coatings.

New Processes used for thin film production: The INNOVATIAL project will benefit from the utilisation of a novel PVD method – High Power Impulse Magnetron Sputtering (HIPIMS) to replace the arc discharge in ABS (Arc Bond Sputtering). This new process will enable to deposit the protective nanostructured coatings with the highest adhesion and full density, which in turn will improve protection against oxidation and preserve the mechanical properties of the substrate (γ-TiAl system or additional substrates).

To use new characterisation techniques: these new techniques, which enable for the first time a complete microstructural characterisation at the nanometre (atomistic) scale, will be indispensable to improve the scientific understanding of the thin film development. These innovative techniques are composed of:

- in-situ XRD for studies of the thermal stability
- Low energy ion beam milling and focused ion beam (FIB) for an optimised preparation of TEM(XTEM) specimens to enable analysis of the relevant problems

Application of the state-of-the-art techniques not commonly used in the field of protective coatings: Differential scanning calorimetry (DSC), Scanning tunnelling microscopy/atomic force microscope (STM-AFM), scanning tunnelling spectroscopy (STS), current image tunnelling spectroscopy (CITS), x-ray photoelectron spectroscopy (XPS), high angle annular dark field (HAADF), spectrum imaging for energy filtered TEM (EFTEM), high resolution electron energy loss near edge structure (ELNES), Methods for the above will be developed for sub-nm analysis of interface structure and composition throughout the coating.

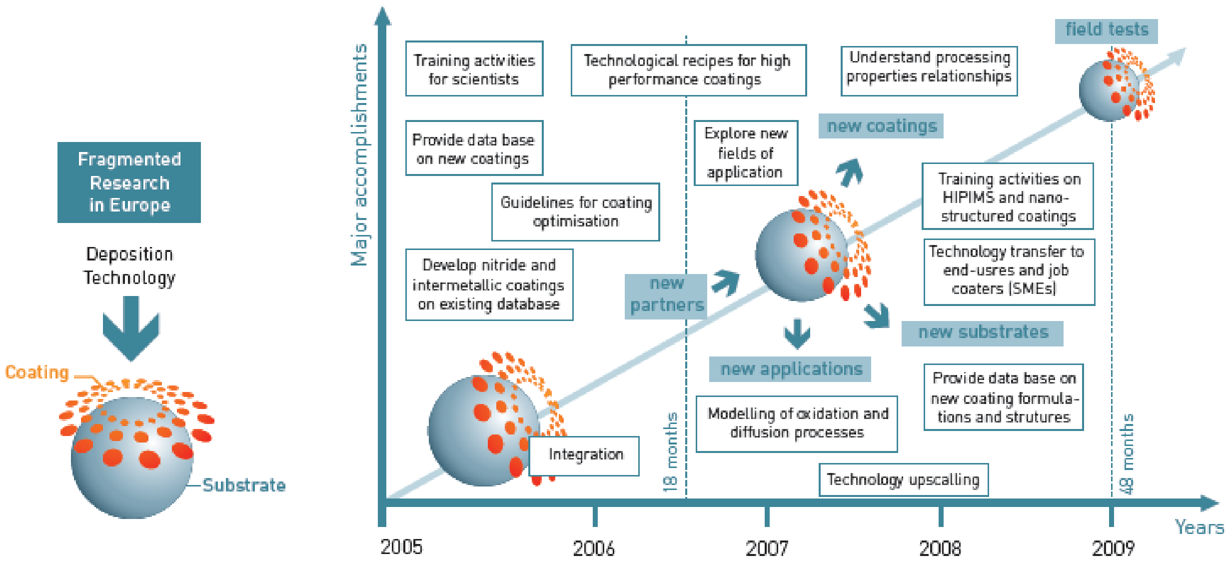

Figure 1: Innovatial roadmap

Section 2. Detailed objectives and results achieved per period

2.1 Year 1

Objectives	Results
The major objectives of the first period of the project (first 12 months) can be summarized as follows: ❒ Technical specifications in terms of processes, coating performance, final products, testing procedures (SP0) ❒ Materials procurement and sample supply to the partners (SP0) ❒ Produce new generation ultra-performance PVD coatings designed to protect gamma TiAl against oxidation and wear in order to allow utilization of these novel materials in high temperature applications (SP1) ❒ Address fundamental problems in the three main regions of the coating/substrate system: the interface, the bulk, and the surface of the coating (SP1) ❒ Launch investigations that will lead to understand the processes involved in the formation of the scales, their development and	Within the reporting period already important technical and scientific achievements were made which are in summary: ❒ Major specifications for expected coating performance in different fields of application were provided by industrials; ❒ A total of more than 650 difficult to machine TiAl samples were provided to the partners; ❒ TiAlYN/CrN coatings (medium temperature strategy 1 (450°C-650°C)) and aluminium oxide top layers were successfully produced in an industrial machine; ❒ Superior adhesion caused by HIPIMS etching over arc etching was successfully demonstrated. Detailed interface investigation provided first ideas on the cause for improved adhesion due to very smooth and clean HIPIMS treated interfaces. ❒ CrAlYN/CrN superlattice coatings (high temperature strategy 2 (650° to 850°C)) were developed, carefully characterised and tested. Outstanding properties such as smooth surfaces, low friction coefficient, high wear resistance and good oxidation resistance were demonstrated. ❒ Intermetallic TiAlCr coatings showed excellent oxidation resistance at 750°C up to 1000 hours. ❒ Thermal Barrier Coatings on TiAl were successfully tested at 900°C up to 1000 hours.

ability to prevent degradation and finally the failure of such scales (SP2). ❒ Study thermal stability of the developed coatings (SP3). ❒ Perform a thorough characterisation of developed coatings (SP4) ❒ Determine the properties of the HIPIMS process (SP5) ❒ Model plasma, oxidation processes, microstructure, microscopic/macroscopic performance, and carry out ab initio modelling (SP6) ❒ Launch first actions towards the elaboration of a training programme and initiate training activities (SP9) ❒ Project management and dissemination of results (SP10)	❒ DSC measurements on TiAlYN nanocomposite coatings have provided initial results on the thermal stability of these systems. ❒ Extensive micro-/nano-structure characterisation of the coatings before and after testing provided first outstanding insights related to the processing-properties-relationships. ❒ Monte Carlo simulation has been successfully applied to binary systems relevant to the more complex coating systems focused on in this project. ❒ Hardware and software preparation for modelling of microscopic structure to macroscopic performance is up and running. Finally, during year 1, 30 deliverables have been produced and 3 important technical milestones related to Thin Film Development have been successfully passed (an overview of deliverables and milestones is provided in section 3).

2.2 Year 2

Objectives	Results
The major objectives of the second period of the project (M13-M24) can be summarized as follows: ❒ Medium temperature range (450-650°C) nanoscale multilayer coatings, t>2000 hours; ❒ High temperature range (650-850°C) Ti–free nanoscale multilayer coatings, t>1000 hours; ❒ High temperature range (650-850°C) low friction nanoscale multilayer coating with at least 70% longer lifetimes than standard coatings; ❒ Improved wear protection of parts by the corresponding coating measured in terms of hardness (>35 GPa) and wear rate (20% improvement compared to as-deposited alloy coatings); ❒ Oxidation protection of ☐-TiAl by coating (>1000 hours at 1000°C); ❒ TBC lifetimes > 2000 h at 850°C; ❒ Understanding degradation mechanisms such as oxidation, sulfidation, microstructure thermal stability and interdiffusion of the coatings.	The consortium has strongly improved interaction between coating development, testing and characterization and has fostered communication to end-users and technology providers. The major challenge during the month 13-24 reporting period was the high number of deliverables to be finished in time. The consortium has worked hard to deliver a maximum number of deliverables planned and actually could submit most deliverables until M24. Some deliverables were postponed by mostly 6 months, and their delay was justified. In addition to the high number of deliverables submitted, most milestones were passed, while some were delayed. A detailed description of the technical and scientific achievements is given in the body of the following report. In the reporting period, the consortium has extensively worked on eliminating some of the weaker points of the project discovered by the 12M review. For example, a simple sample tracking system has been introduced which is maintained on a regular basis by **DLR**. A successful training event was held in July at **SHU** (HIPIMS-ABS Days) which attracted about 80 researchers from the international community. The event was accompanied by a small equipment exhibition. More than 25 presentations and several publications have been published in the reporting period, at least 7 further publications are currently in preparation or pending in review process. In summary, the project is on schedule in most areas, some delays are still obvious but have been justified. The work plan for M25-M42 has already taken these delays into account and re-planned the activities.

2.3 Year 3

Objectives	**Results**
The objectives that were set for the third year of the project were to: ❒ Produce new generation ultra-performance PVD coatings on demonstration parts designed to protect gamma TiAl against oxidation and wear in order to allow utilization of these novel materials in high temperature applications (SP1). ❒ Address fundamental problems in the three main regions of the coating/substrate system: the interface, the bulk, and the surface of the coating (SP1). ❒ Improve understanding of the processes involved in the formation of protective scales during exposure, their development and ability to prevent degradation and finally the failure of such scales (SP2). ❒ Study thermal stability of the developed coatings (SP3). ❒ Perform a thorough characterisation of developed coatings (SP4)	On this basis the major results achieved at Month 36 can be summarized as follow: ❒ Substantial improvement of interface engineering by Cr- and Y-containing interfaces; ❒ Etching conditions for hindered coating – substrate cross – diffusion found; ❒ Major progress in the development of Ti-free CrAlYCN/CrCN coatings for medium (650°C) and high (650-850°C) temperatures achieved; ❒ CrAlYN/CrN+CrAlYON coating systems show excellent performance in high temperature oxidation and tribological conditions; ❒ TiAlYN/CrN+AlOx coatings were successfully tested at 650°C for 2500 h without any sign of degradation; ❒ Intermetallic coatings showed good oxidation resistance up to 900°C; ❒ TBC lifetimes larger than 2000 h at 850°C on gamma TiAl achieved; ❒ Major progress in the understanding of coating microstructures, properties and degradation mechanisms achieved; ❒ Fatigue tests of reference and aged TiAl samples were successfully performed; ❒ Baseline creep tests at various temperatures and stress levels were carried out; ❒ First time observation of a reduction in friction at higher temperatures in a coating that does not contain vanadium but an oxy-nitride layer instead; ❒ Provision of a room temperature tribological test setup for "industrial conditions"; ❒ Extensive thermodynamic modeling on INNOVATIAL coating compositions was performed;

Objectives	Results
❒ Determine the properties of the HIPIMS process (SP5) ❒ Model plasma, oxidation processes, microstructure, microscopic/macroscopic performance, and carry out ab initio modelling (SP6). ❒ Launch a training programme and initiate training activities (SP9). ❒ Project management and dissemination of results (SP10).	❒ Training activities included a tutorial at international conference (ICMCTF 2008, San Diego), HIPIMS sessions at SVC 2008 (Chicago) and ICMCTF 2008, San Diego), HIPIMS-ABS Days held at Sheffield Hallam University, HIPIMS-Workshop in Sweden, an undergraduate course on coatings and surface science as well as workshops on surface and structural characterization; ❒ Several papers have been published in journal and/or at conferences.

2.4 Year 4

Objectives	Results
Focus of coating development ❒ Optimisation of the coating technologies (HIPIMS/HIPIMS coating technique) ❒ Development of low friction high temperature nanoscale multilayer coatings initiated to address other applications than high temperature oxidation resistance such as cutting tools. ❒ Combination of coatings such as intermetallics with TBC or nanoscale multilayers with Al2O3 to achieve better oxidation resistance at high temperatures.	First of all, the coating development was focused on: ❒ The optimisation of the coating technologies (HIPIMS/HIPIMS coating technique) ❒ The development of low friction high temperature nanoscale multilayer coatings initiated to address other applications than high temperature oxidation resistance such as cutting tools. ❒ The combination of coatings such as intermetallics with TBC or nanoscale multilayers with Al2O3 to achieve better oxidation resistance at high temperatures. ❒ Advanced surface analysis techniques used for structure, high temperature oxidation and tribological characterisation of the coatings. ❒ The assessment of the environmental protection capabilities of CrAlYN/CrN

- ❒ Advanced surface analysis techniques used for structure, high temperature oxidation and tribological characterisation of the coatings.
- ❒ Assessment of the environmental protection capabilities of CrAlYN/CrN nanoscale multilayer coatings produced by a combined UBM/HIPIMS deposition technique and Ti-Al-Cr based intermetallic layers with additions of quaternary alloying elements.
- ❒ Assessment of the different routes of thin film development in their effectiveness by detailed investigation of their thermal stability.
- ❒ Study of the thermal stability of superlattice and nanocomposite hard coatings (grain growth, decomposition of meta-stable phases).

Focus of end-users

- ❒ Preparation of the demonstration equipments, specimens and procedures.
- ❒ Production of different components
- ❒ Continuation of the tests campaigns in real conditions in order to assess the performances of the new coatings on additional substrates and to verify their potential industrial exploitation.

nanoscale multilayer coatings produced by a combined UBM/HIPIMS deposition technique and Ti-Al-Cr based intermetallic layers with additions of quaternary alloying elements.

- ❒ The assessment of the different routes of thin film development in their effectiveness by detailed investigation of their thermal stability.
- ❒ The study of the thermal stability of superlattice and nanocomposite hard coatings (grain growth, decomposition of meta-stable phases).

From the end-users partners' side, the focus has been maintained on:

- ❒ The preparation of the demonstration equipments, specimens and procedures.
- ❒ The production of different components (for transport and energy sectors such as:
  - o inserts, cutting tools and dies produced in hard metal and special steels
  - o turbine/compressor blades and gasifier components made of stainless steels and Ni-based super alloys for Energy sector Stainless steels and Ni-based super alloys for Energy sector
  - o valves made of special steels for transport industry applications
  - o blades and turbines for the aeronautic sector
- ❒ The completion of the tests campaigns in real conditions in order to assess the performances of the new coatings on additional substrates and to verify their potential industrial exploitation. One can especially mention the on-going test of the new coatings developed in INNOVATIAL on TiAl substrates for aeronautic and automotive applications.

Otherwise, the support from the characterisation and modelling activities to the coating development and the validation phase kept on going over this period.

Finally, the partners put all efforts in the last months of the project to finalise the planned activities and to ensure that all the foreseen targets are met.

Section 3. Outstanding results

3.1 Coatings Development and Fabrication

To meet the challenges of the demanding applications in the INNOVATIAL project several innovative strategies were implemented in the coating development process as well as utilisation of the novel HIPIMS technology. Four different coating strategies were followed to develop coatings to protect γ-TiAl against environmental attack involving nanoscale multilayers, nanocomposites, intermetallic coatings and thermal barrier coatings (TBCs).

Coating	Deposition technique	Coater	Thickness [µm]	Oxidation performance
Nanoscale multilayer coatings				
TiAlYN/CrN + Al_2O_3	UBM	HAUZER	3.5	2000h / 750°C
CrAlYN/CrN	UBM	SHU	5	2000h / 850°C
CrAlYN/CrN	HIPIMS	SHU	4.5	1000h / 900°C
CrAlYN/CrN + Al_2O_3	HIPIMS / UBM	SHU / HAUZER	5.5	2500h / 850°C
Nanocomposites				
TiAlYN	UBM	MUL	4	1000h / 750°C
CrAlYN	UBM	MUL	4	1000h / 900°C
Intermetallic layers				
Al_2Au	UBM	MUL	4	1200h / 850°C
Ti-Al-Cr	UBM	DLR	10	1000h / 900°C
Ti-Al-Cr-Hf	UBM	DLR	10	1000h / 900°C
Ti-Al-Cr-Y	UBM	DLR	20	1000h / 950°C
Ti-Al-Cr-Zr-Y	HIPIMS	SHU	11	1000h / 1000°C
TBC systems				
CrAlYN/CrN +YSZ	HIPIMS / EB-PVD	SHU / DLR	4.5 / 150	1000h / 900°C
CrAlYN + YSZ	UBM / EB-PVD	MUL / DLR	4 / 150	1000h / 900°C
Ti-Al-Cr + YSZ	UBM / EB-PVD	DLR / DLR	10 / 150	1000h / 900°C
Ti-Al-Cr-Y + YSZ	UBM / EB-PVD	DLR / DLR	20 / 150	1000h / 950°C

Figure 2: Innovatial coatings properties

Ti-free CrAlYN based nanoscale multilayer coatings were introduced by Sheffield Hallam University (SHU) where Y was used in all critical areas of the coating such as the interface, the base layer and the coating. Various top coats such as CrAlYON (SHU) and Al_2O_3 (Hauzer) were developed for further enhancement of the tribological and high temperature performance of the coating. Due to the HIPIMS etching, CrAlYN/CrN showed excellent adhesion to γ-TiAl and various other substrates like stainless steel and cemented carbide. Unlike many nitride coatings, CrAlYN/CrN reduces its friction coefficient with increasing the temperature and outperforms many intermetallic coatings in aggressive $H_2/H_2S/H_2O$ environment. Most importantly it was demonstrated that HIPIMS deposited CrAlYN based multilayers can preserve the mechanical properties and realise less than 10% fatigue deficit of the base γ-TiAl material.

Ti-55Al-15Cr-0.3Zr intermetallic coatings deposited by standard magnetron sputtering technique produced by the German Aerospace Center (DLR) were further adjusted in chemical composition and densified by HIPIMS/HIPIMS technology at SHU. The approximately 11 µm thick coating exhibited a dense layered structure and established the formation of a thin continuous alumina scale during high temperature exposure in air. The protective scale was still present after dwell time periods at 1000°C exceeding 1000 h, providing effective oxidation protection to γ-TiAl based alloys. The intermetallic coatings are free of nitrogen and therefore formation of TiN due to interdiffusion of nitrogen into gamma TiAl alloy substrate is avoided. The HIPIMS/HIPIMS intermetallic Ti-Al-Cr-Zr-Y layer is also a suitable bondcoat for thermal barrier coatings due to the excellent adherence of zirconia topcoats to alumina scales and represents the most oxidation resistant coating in INNOVATIAL.

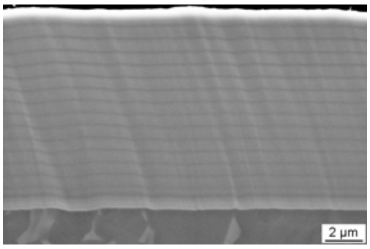

Figure 3: Cross section SEM of a γ-TiAl sample coated with HIPIMS intermetallic Ti-Al-Cr-Zr-Y

The successful application of protective coating systems to allow for the large scale industrial use of γ-TiAl based alloys as light weight components in high temperature applications such as turbine blades or valves heavily depends on the understanding of the relevant physical and chemical processes occurring during service, as well as the interrelation-ship between environment, protective coating and substrate. Laboratory-scale research at Montan Universität Leoben (MUL) proposed CrAlYN coatings with 1 and 4 at.% of yttrium being oxidation resistant and showing low interdiffusion.

Based on a combination of experimental and computational materials science, employing density functional theory, it is clarified that Cr, Al, and Y share and fully occupy metal sub-lattice sites of a face centered cubic structure. The calculations furthermore proposed the stability range and development of the elastic constants with the chemical composition of the cubic structure, assisting the coating development.

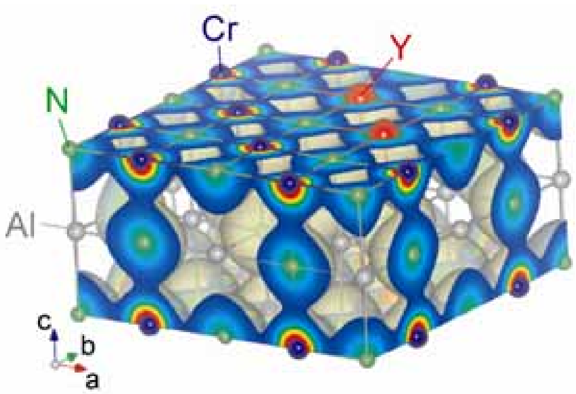

Figure 4: Face centered cubic structure of CrAlYN including the electron density distribution

In the INNOVATIAL project a successful development has taken place of PVD technology in general and specifically HIPIMS technology.

The development made fully automatic process cycles integrating HIPIMS available on the machine installed at SHU. Part of this task was to find a solution for the bias peak current problem, which has lead to a patent application for a special bias power supply or addition of a module to an existing bias power supply. The addition utilizes a very fast arc suppression unit, able to stabilize the bias voltage. This solution is patent pending (patent nr. GB 2437080A).

Within the framework of the INNOVATIAL project, Hauzer has introduced HIPIMS technology in house and is actively marketing this technology since a few years. Together with SHU has Hauzer developed the coating TiAlCrYN/CrN with an Al_2O_3 top coating. The Al_2O_3 top coating was initially produced with pulsed-DC magnetron technology, but this has the disadvantage of a limitation to the coating thickness.

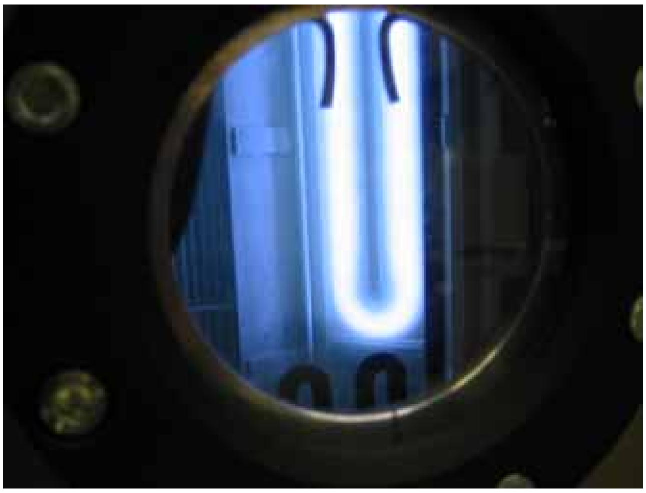

Figure 5: View through a viewport on the plasma of an active HIPIMS cathode (HAUZER).

Hauzer worked on the development with dual magnetron technology which enabled thicker coatings, but could also give better coating properties. With this technology it became also possible to improve the properties of the Al_2O_3 top coatings (harder and denser coatings). For this dual magnetron sputtering solution a patent application has been filed.

Using HIPIMS technology enables to perform metal ion etching with rare earth elements: the HIPIMS discharge is particularly suitable to be used for etching of rare metal earth ions. This allows producing hard protective coatings on a conductive substrate that is used at high temperatures. The solution is patent pending (GB0713671.6, EP 08012598.2). Examples for applications are coated tool or components.

In parallel a wide range of aluminide coatings deposited by CVD and pack aluminizing have been studied and developed by Turbocoating on different substrates for application on gas turbine components.

3.2 Coatings characterization, testing and modelling

In addition to coatings development and fabrication a major emphasis was placed on the characterization and testing of the coatings developed within the INNOVATIAL project.

INNOVATIAL coatings have one thing in common: they have ultra-fine scale structures and the bonding of the coating with the substrate depends strongly on the structure of the interface between coating and substrate. To understand the relationship between the microstructure, the processing and the properties, we have had to apply the very latest atomic resolution electron microscopy. In particular, we have used spherical aberration corrected transmission electron microscopy to determine structure and chemical bonding using electron beams that are smaller than the atoms we examine! Thus, remarkable details of the interface between the HIPIMS γ-TiAl treated substrate and the CrAlN base layer for a CrAlYN/CrN coating could be visualized. The HIPIMS treatment has promoted a thin (2-3nm) depth of Cr ion implantation (compared to 5-8nm for a Cr ion arc discharge (-1200V bias)), and excellent atomic bonding, as shown by the clear atomic image.

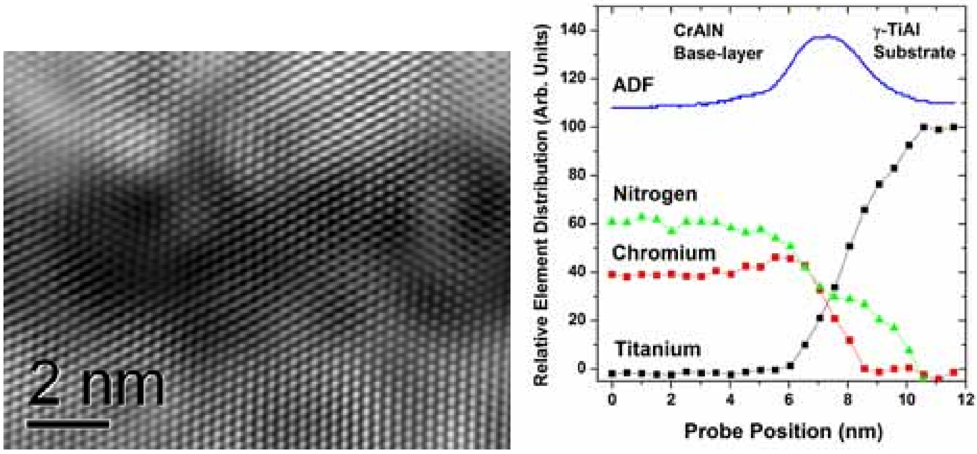

Figure 6: Ultra-high resolution characterisation of the interface between CrAlYN/CrN coating, the CrAlN base layer and the γ-TiAl substrate grown using HIPIMS technology, all viewed in cross-section. High resolution atomic image across the image showing the intimate atomic bonding at the interface (top). Atomic resolution chemical composition across the interface (bottom).

The INNOVATIAL coatings were extensively tested under various conditions such as oxidation hot corrosion and wear. Quite obviously, the different coatings behaved differently under certain testing conditions. Therefore, a major conclusion is that the coatings developed offer not a "one-fits-all" solution but have to be carefully selected according to the service conditions exposed.

It is well known that hard PVD coatings offer greatly improved wear resistance compared to conventional materials. However, one key objective is to develop coatings that offer both high wear resistance and also low friction. This can be achieved at low temperature through coatings such as DLC. However, it is a completely different issue at high temperature, where friction typically increases as temperature increases.

Moreover, high temperature wear requires that the structure of the coating does not degrade during prolonged exposure. The newly developed CrAlYN/CrN coating using HIPIMS technology offers, *for the first time*, both high wear resistance and low friction coefficient at high temperature, coupled with excellent coating thermal stability. The friction coefficient shows a remarkable reduction at high temperature. The wear resistance of the coating clearly depends on the manufacturing process, with the HIPIMS/HIPIMS technology producing up to a 15 fold reduction in wear rate.

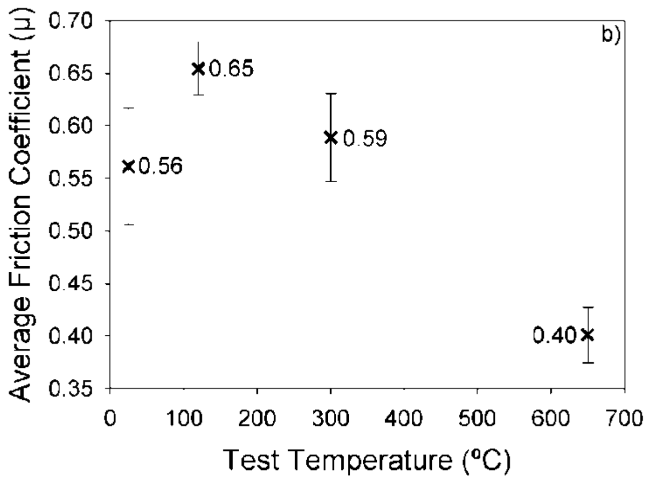

Figure 7: Wear behaviour of CrAlYN/CrN as a function of temperature; note the remarkably low friction at 630°C

Due to the great importance of high temperature capability of the coatings, interdiffusion between the coatings and the substrate was studied experimentally and by modeling. The Generalized Darked Method as well as the Genetic Algorithm Method were used in order to model the complex multi-component systems. Using these two methods, interdiffusion between TiAlCrY and CrAl-2%YN coatings and a γ-TiAl alloy was successfully modeled. The Genetic Algorithm method was used to optimize the diffusion coefficients in multi-component systems. The optimized diffusion coefficients were then used to compute composition profiles.

3.3 Industrial applications

INNOVATIAL coatings were applied to various components such as engine valves, gasifier components, aeroengine airfoils, gas turbine buckets, rollers and dies. The components were supplied by the partners Nuovo Pignone, Ansaldo, MTU Aeroengines, Wolframcarb, Osvat and Fiat.

Figure 8: Industrial applications of the Innovatial coatings