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CONFIDENTIAL

Contract n° : BRPR-CT-98-0795

Project n°: BE97 5021

Title:

DEVELOPMENT OF ENGINEERING SURFACE COATINGS OBTAINED BY ADVANCED, COST EFFECTIVE AND ENVIRONMENTALLY FRIENDLY TECHNOLOGIES

Project Coordinator: Flametal

Partners:

Flametal SpA (Project Coordinator), Italy
ACOM, Italy
Rubig, Austria
Teer Coating Limited, GB
IVECO, Italy
Imperial College, GB
Institute for Advanced Materials, EC
National University of Ireland, IE
ICMAB, Spain
INSTM, Italy

Reference period: from the 15/11/1998 to the 15/11/2001

Starting date: 15/11/1998 **Duration: 36 months**

Project funded by the European Community under the Industrial & Materials Technologies Programme (Brite-Euram III)

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1 SUMMARY

Keywords on the contents of the project

PVD

PACVD

Multilayer coatings

Plasma Spray

SHS powders

Abstract of the results and benefits of the project

TASK 1 was a real and pure research task with a very high innovative content, separated in 3 main subtasks. The main achievements to date are:

i) a new industrial technology and metodology for the spraying of synchroniser rings using unconventional 'semi-axial' torch and, a precious know-how concerning the design/assemblage of components to produce a full axial torch prototype, an open road for the usage of plasma sprayed coating for synchroniser rings (instead of the old and unique one process in this field: the wire metallization).

ii) A new plasma spray powder production process (SHS) where our feasibility study permitted us to demonstrate that this new powder technology can be easily applied in the field of plasma spraying with considerable perspective and promises at both the technical and costs levels.

iii) An assessment of both APS and RPS technologies for the spraying of MMC materials.

TASK 2 aim's was the production of high performant multilayer systems by PVD and CVD, using new engineered facilities. Technical achievements are the production of extremely hard nitride multilayer systems and oxide multilayer systems by PVD and several ultra hard multilayer systems(phases of TiN, TiAlN, TiB2, TiCN) by PACVD.

TASK 3

i) Development and Optimization of high performance steels for application of hot-forming.

ii) Development of a suitable technology to produce, via plasma spray, near-net-shape shells for composite dies

iii) Preparation and coating of industrials parts, using the outcomes of task 1 and task 2, these parts have been tested in task 4.

TASK 4 main achievements are:

i) The constitution of a consequent databases concerning the behaviour of engineered Plasma Spray coatings applied on synchronizer rings.

ii) A much better understandings of the problematic of the synchronizer rings that make PRAXAIR deciding to build-up an own testing bench

iv) Development of high performance steels and combined CVD treatments that lead to increase the life duration of industrial hot forming dies. Assessment of in-service wear performance.

v) A set of experimental datas, results and in service wear performance of new PS, PVD and PACVD coatings for truck chassis stamping and synchronizer rings.

2 THE CONSORTIUM

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The company started its activity in coatings in 1973 and today it is a leading company in Italy with about 120 people employed with dedicated equipment and facilities, while several external laboratories and universities co-operate in various fields of the company's activity. PRAXAIR SURFACE TECHNOLOGIES, div Flametal specializes in high quality metallic and ceramic coatings, produced by means of thermal spraying technologies (High Velocity Oxy-Fuel, Plasma Spray, Wire Spray etc.). PRAXAIR SURFACE TECHNOLOGIES, div Flametal has coordinated the project and was a task leader. In the project, the Company develpped new thick coatings, using their thermal and plasma spray facilties, for automotive applications.

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ACOM is a SME with 30 employees specialised in precision mechanical manufacturing especially for the aerospace industry, mathematical modeling (CAD/CAM), prototyping, dies and modelling. ACOM resources are suitable for the realization of prototypes in batches and small series using materials such as resins, aluminium, alloy and stainless steel, Cr, Ni, Co (Inconel 718), titanium alloys and superalloys. In the present project ACOM has been involved mainly with the development of Hot-forming-high-performance steels and the realization of stamping dies for the manufacturing of automotive synchronizers and gearbox parts.

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IC - Partner 3, Imperial College of Science, Technology and Medicine is part of the University of London and one of the foremost academic and research institutions in the United Kingdom. Its Department of Mechanical Engineering in particular, plays a leading role in the international scene in developing novel analytical, computational and experimental techniques for investigating the deformation and fracture behavior of metals, polymers and composite materials. The Department has also been instrumental in establishing novel fracture mechanics methodologies to describe low and high temperature fracture processes. Most of the Department's research activities are carried out in collaboration with industry and other research institutions, both nationally and internationally. Research funding comes partly from industry, some is provided by the UK's EPSRC (Engineering and Physical Science Research Council) in collaboration with industry, and some by the EU through

programs such as COST, Brite-Euram, INTAS, and VAMAS. Sponsors in the UK include Rolls-Royce, European Gas Turbines, Nuclear Electric, National Power, MoD, Powergen, Ford and ICI. In the project IC has been responsible mainly for the development of a computer code based on a microfracture model and applied on automotive longerons dies.

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The Institute for Advanced Materials (IAM) forms part of the Joint Research Center, a European scientific research center established by the Commission of the European Communities with headquarters in Brussels. The Institute for advanced Materials (IAM), with sites both in Petten(NL) and Ispra(I) executes the specific research program on advanced materials and comprises a total of approximately 210 permanent staff of which 110 staff including 30 graduate level researchers are based within the Ispra site in Italy.
Facilities available for surface modification include a high current ion implanter, laser treatment by high power CO2 and Nd-YAG lasers, thin film deposition by PVD, CVD, PACVD and thick coating deposition by atmospheric and reduced pressure plasma spray methods. These facilities are supported by a wide range of advanced chemical and structural characterization techniques as well as testing facilities for assessing biocompatibility, wear resistance properties and high temperature corrosion resistance. In the project they have mainly been involved in developing and testing of MMC coatings from SHS powders (task 1).

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The National University of Ireland, Galway, is a constituent college of the national university of Ireland. The Department of Mechanical Engineering has over a short number of years established itself as a significant industrial research department which has produced 5 research units. They have interacted with industry in terms of process development and product design and have contributed significantly to the economic growth of the west of Ireland. The Micromechanics Research Unit conducts research into applying micromechanical modeling techniques to industrial and engineering materials. It has specialized in finite element methods, large strain deformation in materials and also in analytical and computational modeling of materials, developing microstructural models to predict constitutive behavior. NUIG has been responsible for the development of wear models for thick coatings and their validation on an automotive synchroniser test bench.

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RÜBIG has specialized in the construction of surface-treatment-plants and heat treatment. Rübig plants are sold all over the world (mainly Europe and Asia) and the plants and processes are used in the company own job shop. Rübig employs 172 people, of these six are scientists engaged in R&D activities. Rübig has been involved in several local and multi-partner research projects, funded both nationally and at the EU level.
Rübig has been a task leader, involved in developing and producing multilayer coatings by the innovative technique of pulsed plasma assisted CVD (P-PACVD).

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Teer Coatings Ltd., is an SME formed in 1985. It employs 59 staff of whom 9 are graduates and

currently 2 more are studying for degrees. The main activity is concerned with advanced surface coatings for many applications, deposited by magnetron sputtering. About 1/3 of the effort is devoted to R&D, 1/3 with the supply of a job shop coating service and 1/3 with the design, development and supply of advanced coating equipment and related test equipment. The strategy of the company is to heavily invest in modern equipment and in R&D to maintain a world-wide lead in thin film PVD coating technology and to exploit this lead commercially. They have been involved in developing and producing multilayer coatings by PVD.

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IVECO is an end user manufacturer of industrial vehicles with a completely integrated production process including: component heat treatments, engine and vehicle assembly. IVECO was established in 1975, bringing together the commercial vehicle business of FIAT, OM and Lancia in Italy, Unic in France and Magirus Deutz in Germany. This combination created the first truly European truck manufacturer, with a significant presence across all major European markets. IVECO is an organization with 24 production plants and 20 research centers located in Germany, France, the UK, Italy, Spain and Switzerland, which together form a fully integrated industrial system. IVECO, with 32,800 employees is one of the biggest producers in its field. In the project IVECO was in charge of performing the industrial tests of the coating solutions provided by the concortium.

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The Institut de Ciència de Materials de Barcelona (ICMAB) is a research center belonging to the Spanish state organism Consejo Superior de Investigaciones Cientificas(CSIC). ICMAB was founded in 1987 and inaugurated in April 1991. The ICMAB is also part of the European associated

Laboratory LEA SIMAP", together with the IMP Odeillo-Perpignan and the LPCM Montpellier. The ICMAB research work is focused on the synthesis , preparation, crystallization and characterization of high performance functional materials with direct industrial relevance. Examples of research projects include high temperature super-conducting materials, new batteries, transport assisted membranes chemical sensor and hydroxyapatite coatings. Among other up to date characterization methods, the ICMAB possesses all the equipment necessary to this project for the physical characterization of the coatings. SEM-EDAX, XRD diffractometer with texture analysis, ellipsometry and optical techniques. In the project, they provided their expertise in material science to the consortium.

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INSTM (formerly INCM, Consorzio Interuniversitario Nazionale per la Scienza e Tecnologia dei Materiali). The INSTM is the largest institution in Italy involved in research in the field of materials. It collects and coordinates as subunits most of the Italian university groups involved in this area. In the present project the INSTM units will be coordinated by Prof. Ugo Bardi and their tasks will be: develpment of powders by SHS methods and development of reactive plasma spray techniques.

3 TECHNICAL ACHIEVEMENTS

The main achievements to date are:

i) **a new industrial technology and metodology for the spraying of synchroniser rings using unconventional 'semi-axial' torch.**

To ensure a long and constant in-service performance, synchronizer rings internal parts are actually coated with a unique and world-wide exclusive material: Molybdenum. Molybdenum is wire sprayed on all kind of rings configurations in terms of diameters, rotating speed, couple, and load. Due to environmental reasons,

the tendance is to substitute Molybdenum by other materials, more healthy-friendly and. As a consequence, research efforts are made to find an alternative solution. In order to extend the choice of materials, PRAXAIR conducted a research to make the well-known plasma-spray process envisageable i.e. cost realistics.

The selection of plasma spray coatings for synchronizer rings is thus a recent concept. Despite the unfavorable cost-effectiveness of plasma spraying comparing with the traditional Wire Metallisation, one of the main achievement is the succesfull introduction of plasma spray that allows, providing new process concepts, an industrial realistic tool for coating the internal part of the automotive synchronizer rings. On the other hand, its use makes wider the range of spraying materials.

In the project, a set of several new coatings have been applied on rings and tested thanks to an appropriate testing bench. A very large set of datas has been collected and analysed.

ii) The demonstration that the process (SHS) permits the production of sprayable carbide powders.

SHS process is a process, coming originally from the ex-Soviet Union, allowing the sintering of materials. The intrinsec character of 'Self Propagating Synthesis' leads to an easy, versatile and very low cost process for the sintering of ceramics, cermets and over kind of materials.

Several partners contributed to this research task. It turned out that SHS powders are usable for Thermal Spray. Moreover it presents a real cost effectiveness attraction for carbide phases powders and great potential for nitride and boride phases, where usual production processes are significantly more expensive. It must be mentioned that thanks to the project, a patent has been deposited by one partner.

iii**) The development of new and superhard multilayer PACVD coatings**

The thin film technology CVD is a well know technique for the production of corrosion and wear resistant coatings, among them TiN thin films remain one of the most popular. However, due to applications that require more and more higher performance surface properties, the state of the art technology in CVD start consisting of producing multilayer systems in order to cumulate the intrinsec and complementary mechanical and corrosion properties of the single layers. To produce such coatings , in addition to the design of the multilayer itself, new facilities must be designed and manufactured.

Within the project, after a complete study of a new CVD machine and its manufacturing, a set of performant multilayer coatings has been developed. The main phases are TiN, TiBN, and TiB_2 and TiAlN.

iv) Development of engineered equipment and production of a set of new high-performance-PVD-multilayer coatings

Thanks to the design and manufacturing of a new sputtering machine, nitride multilayer coatings and oxide multilayer coatings hav been succesfully developed; both exhibit outstanding physical and chemical properties. The nitride based multi systems (NbZrN, MoZrN, NbTiN) are targeted to high load stamping applications while multilayer oxide ceramics coatings (AlO_x-ZrO_x) are targeted to high temperature processes.

The nitride multisystems have been succesfully submitted to a long in-service testing on IVECO's production site, for the special application of truck chassis stamping dies.

v) Design, production and in-service testing of high performance steels in the framework of hot forming stamping

The developments have leaden to the production of high performance steels in the special frame of hot forming. Different grades of steels, heat treatments and nitriding processes have been optimized to provide the very best material performances. At the end of the project, the most performant steels have been employed to manufacture samples pins and dies and their relative in-servive life measured under real industrial operating conditions. The results obtained are outstanding: for the pins, the duration get a 60% bonus while concerning stamping dies, improvements by a factor of 2 and 3 have been registered.

vi) Modeling method, based on micro-mechanics and finite element analysis, to predict the in-service behavior of truck chassis stamping dies coated by PVD thin films

The modelling and prediction of wear is something that has proved extremely difficult to achieve, and usually experimentally fitted relationships and empirical methods are employed to predict wear rates and component lifetimes. This is especially true for surface coatings, now seen more commonly in industrial applications. This result, a modelling methodology based on a fracture mechanism approach for the prediction of the propagation of cracks i.e. of the life-time of the coating, is innovative and in accordance to the experimental datas. The result has significant relevance to the research community, in that new scientific methods have been developed, and to industry, in that a predictive methodology now exists to aid in coating selection and assessment. Currently the methodology is being disseminated through journal and conference.

vii) Modeling method, based on micro-mechanics and finite element analysis, to predict the in-service behavior of synchronizer rings coated by Plasma Spray thick films.

A formulation of a model that establishes a link between a local and global friction coefficient for thick coatings on synchronizer rings has been developed. Synchronizer engagement has been modeled using various friction coefficients to determine the effects that the wear of the coating has on the surface geometry and subsequently on the computed friction coefficients. This computer code will be of extremely relevance for the understanding of physics laws that govern the behavior of the synchronizer rings and serve to the design of more competitive surface treatments.

4 EXPLOITATION AND FOLLOW-UP ACTIONS

The main deliverables at the end of the project are a set of techniques and methods for innovative and cost effective coatings manufacturing. The development approach is novel and the results represent a considerable technological breakthrough. The partners recognize that the results have great potential for exploitation for the benefit of EU competitiveness.

The coating producers will exploit the methodology to attract customers by being able to offer more reliable and less expensive coatings. TCL, Rübig, by involving their marketing and sales department, will exploit the methodology to attract customers for their vacuum coating machines. It is hoped, as stated in the original project proposal, that the successful exploitation of the multilayer coatings will open up new application areas for thin film coatings and consequently lead to an increase in plant sales. The economic benefits to the companies will be derived from such equipment sales, from the licensing of these specific coating processes by other coating companies, and from the provision of a coating service. Within the DESCO project, the appropriate multilayer coatings will be applied to forming dies (hot forming, sintering and cold forming).

PRAXAIR SYRFACE TECHNOLOGIES will use the methodology to achieve rapid development of high quality optimized coatings by Plasma Spray techniques. As several coatings are been developed and tested, PRAXAIR will exploit the research and broaden his experience in Thermal Spraying. IVECO will exploit the methodology to produce higher quality, more reliable and longer life products, and to greatly accelerate the introduction of wear-resistant coatings over a wider range of their machines and products. ACOM will increase his customers market set. Moreover, concerning employment and as underlined by ACOM during the Midterm assessment meeting, the insertion in the automotive industry offers great perspective for employment because of the high volume production. The objective is to move from 30 people to 35 people within 1 or 2 years.

By participating in the project the industrial partners establish strong relationships with one another. They will exploit this to establish stronger contacts with industries in the participating member states and in other states which will enhance their respective international profiles and increase their export sales.

The university and research institute partners will exploit the high level of knowledge and expertise gained by teaching and training research students and introducing aspects of the results into undergraduate courses. They will use the developed techniques in further research projects with industry and other research institutions. NUIG and IC will generalize and enhance the computational and material modeling capabilities. IC has arrangements in place to exploit inventions through its wholly-owned company; ICMAB will generalize and enhance their microscale and macroscale experimental testing and material characterization techniques.

This is an industrial project and the deliverables are of considerable technological importance and of direct economic and exploitable benefit to the partners, even though further development will be necessary in order to fully exploit the commercial potential of the products developed in the present project.

5 COLLABORATION SOUGHT

Three partners are searching collaboration. All details are given in the TIP document.