

**Programme for Research-Development-Innovation for  
*Space Technology and Advanced Research - STAR***

Multilayer inorganic/organic tribological  
coatings for space applications  
SpaceCoat

Dr. Ing. Gheorghe Mateescu

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	<b>Partner organizations</b>	<b>Partners team leaders</b>	<b>Contact address</b>
CO	SC. AEG Progresiv SRL	Gheorghe Mateescu	Adress: Bucuresti, str.Nucsoara, nr. 6, B 42/ 70 Ccod poștal 060524 Tel/fax: 0212208084 Email: gheorghe_mateescu@yahoo.com
P1	Universitatea din Bucuresti	Adriana Bălan	Adress: Bucuresti, sector 5, Bd. Mihail Kogalniceanu nr. 34-36, Cod 050107 Tel/fax : 0214574838 Email: andronie@3nanosae.org
P2	Universitatea Transilvania Brasov	Cornel Samoilă	Adress: B-dul Eroilor nr.29, Brasov Tel: : 0268413000, fax: 0268410525 Email: csam@unitbv.ro

## ■ **Project goal**

To design novel tribological coatings for metallic aerospace or aircraft components, based on dry lubricant coatings from materials with complementary-cumulated properties and with the structure of the following types:

-**mono-layer** with constant or gradual composition

-**multi-layer** with micrometric, nanometric or superlattice structure with polymer interlayer

obtained using the well-proven technologies (Magnetron Sputtering Deposition Method and Plasma Treatment Method) on one hand and opening the perspective of implementation the Open Atmosphere Cold Plasma Powder Deposition Method (OACPP-DM) as an economical and very new deposition method on the other hand.

## ■ **Objectives**

- O1. To evaluate the specific requirements of tribological coatings on movable components for space applications
- O2. To optimize a deposition protocol of new tribological coatings: magnetron sputtering and plasma deposition techniques.
- O3. To develop a new approach in tribological coatings using an alternative open atmosphere cold plasma powder deposition method.
- O4. To undertake research studies on tribological coatings of movable components in laboratory conditions.
- O5. To evaluate the possibilities of using the achieved knowledge for aerospace or aircraft movable devices.
- O6. To disseminate the results by conference participations, articles in ISI quoted journals and patent applications.
- O7. To develop institutional capabilities and human resource skills dedicated to applicative research and development of innovative technologies in Romania in the field of tribological coatings for space applications.

## ■ Estimated results:

- Tribological Coatings (*Dry Lubricant Coatings*) with improved wear resistance, obtained by the use of a new structural and compositional design (*nanostructured coatings with mono-layer or multi-layer structure, obtained from materials with complementary-cumulated properties such as:*
  - *good adhesion to the metallic substrates;*
  - *high hardness and toughness;*
  - *high resistance at oxidation and corrosion at extreme temperatures;*
  - *low friction coefficient,*

in order to increase the working life of the aircraft movable devices;

- Innovative solutions for achievement of the tribological coatings, using the advanced deposition technologies in vacuum (*Magnetron Sputtering and Plasma Polymerization*) and in open atmosphere (*Cold Plasma Powder Deposition Method, combined with Plasma Surface Treatment*);
- Technological transfer of the research results to the SME partner.

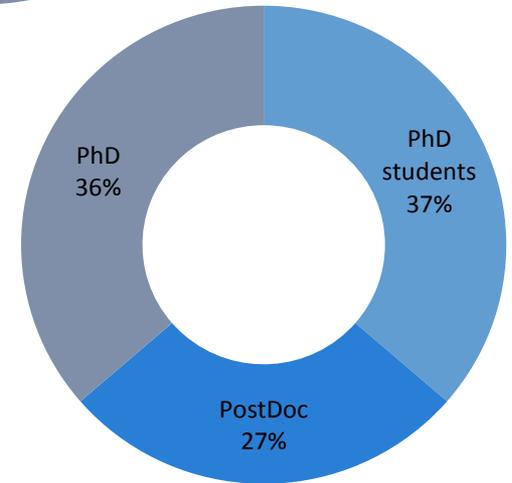
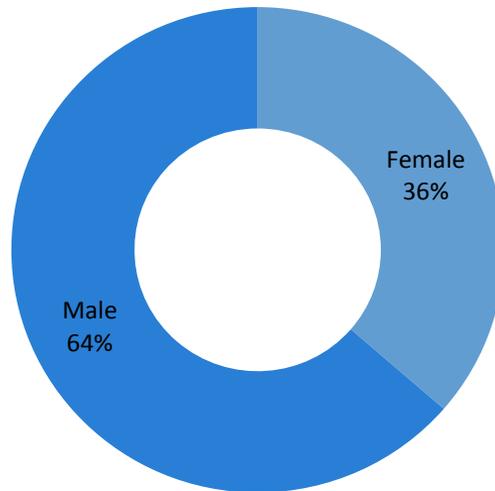
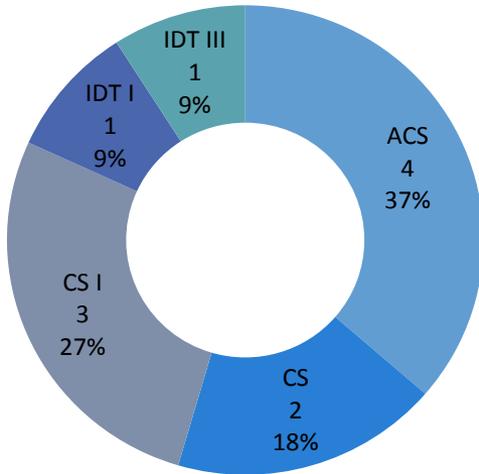
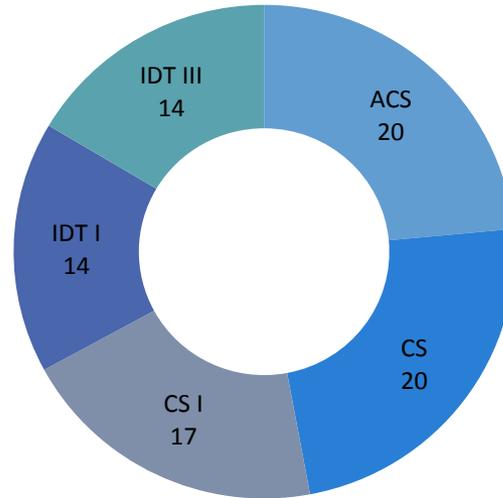
## ■ Start date of the project / End date of the project:

**Nov 2013 -> Nov 2016**

■ **Human resources involved**

- **Total man-month = 85**
- **11 persons involved**

**Man-month**



# Work plan of the project

WP/Task name	WP/Task leader	1st year				2nd year				3rd year			
		3	6	9	12	15	18	21	24	27	30	33	36
<b>1 Tribological coatings with nanostructured multilayers obtained by magnetron sputtering and plasma deposition technics</b>	P1	[Bar from 3 to 12]											
1.1 Defining the requirements of tribological coatings for space applications in terms of materials, methods and testing.	Co, P1	[Bar from 3 to 6]											
1.2 Design and synthesis of nanostructured multilayer coatings on glass/Si/stainless steel substrates by magnetron sputtering deposition	Co, P2	[Bar from 6 to 12]											
1.3 Design and synthesis of nanostructured multilayer coatings with polymer interlayers on glass/Si/stainless steel substrates by magnetron sputtering deposition and plasma deposition	Co, P1, P2					[Bar from 18 to 24]							
1.4 Characterization of the tribological coatings- structural and specific physical properties.	P1, P2	[Bar from 6 to 12]				[Bar from 15 to 24]							
1.5 Optimizing synthesis conditions	Co, P1, P2	[Bar from 6 to 12]				[Bar from 15 to 24]							
<b>2 Tribological coatings with nanostructured multilayers obtained by Atmospheric Pressure Cold Plasma Powder Deposition</b>	Co					[Bar from 15 to 24]							
2.1 Design and synthesis of nanostructured multilayer coatings on glass/Si/stainless steel substrates by Atmospheric Pressure Cold Plasma Powder Deposition	Co					[Bar from 15 to 18]							
2.2 Characterization of the tribological coatings- structural and specific physical properties.	P1, P2					[Bar from 15 to 24]							
2.3 Optimizing synthesis conditions	Co, P1, P2					[Bar from 21 to 24]							
<b>3 Tribological coatings on mechanical components/devices in laboratory conditions</b>	Co									[Bar from 27 to 36]			
3.1 "Trade-off" studies and selection of the appropriate technical solution	Co, P1, P2									[Bar from 27 to 30]			
3.2 Verifying the readiness of the coating procedure and the compatibility with the specific	Co, P1, P2									[Bar from 27 to 30]			
3.3 Coating the mechanical aerospace components/devices using the validated deposition method.	Co, P1, P2									[Bar from 27 to 33]			
3.4 Characterization of the validated tribological coatings on mechanical mechanical components in laboratory conditions.	P1, P2									[Bar from 30 to 33]			
3.5 Preliminary studies regarding specific space conditions (vacuum and cosmic radiation).	Co									[Bar from 33 to 36]			
<b>4 Management</b>	Co	[Bar from 3 to 12]				[Bar from 15 to 24]				[Bar from 27 to 36]			
4.1 Reporting, exploitation, and publication	Co, P1, P2	[Bar from 3 to 12]				[Bar from 15 to 24]				[Bar from 27 to 36]			
4.2 Scientific know-how- intellectual property	Co, P1, P2	[Bar from 3 to 12]				[Bar from 15 to 24]				[Bar from 27 to 36]			

- **Implementation status of the project**

## **Technology Readiness Level**

- **Start TRL=2:** concept- new nanostructure layered materials
- **Target TRL=3:** new materials and depositions techniques- process characterization (laboratory environment)

## **WC and WCN nano-structured dry-lubricant coatings obtained by Magnetron Sputtering deposition method**

- **Wolfram carbide (WC)**- hard and lubricant compound material for applications in the mechanical tools, chemical industry, electronic industry, national defense, etc.
- **Wolfram carbonitride (WCN)**- a suitable barrier material against copper diffusion.

Both materials could be used as tribological materials in the micro-structured tribological coatings with monolayer structure or as tribological surface and intermediary layer in the nano-structured tribological coatings with multi-layer structures.

➤ *WC or WCN nanostructured multi-layers* are obtained when the thickness of these surfaces or intermediary layers must be in the nanometric scale (1...100nm) or the grain size (*as nanostructured units*) of the coatings with monolayer structure must be with individual volume between molecular dimensions (*with values in Angstroms*) and microscopic dimensions (*with values in micrometers*), on the entire volume of the mono-layer.

The scientific work presents the influence of the magnetron sputtering deposition process parameters (*substrate deposition temperature, power injected in plasma that establishes the deposition rate, working pressure and working flow gases, substrate polarization voltage, etc.*) on the nanostructured parameters of the WC and WCxNy layers (*used as surface layers in the dry lubricant coatings*), obtained by Standard Magnetron Sputtering in DC of a WC target, respectively by Reactive Magnetron Sputtering in DC of a WC target, using N<sub>2</sub> as reactive gas.

**Characterization methods:** AFM, Corrosion Resistance and Electrical Conductivity.

**Influence of the Deposition Process Parameters on the Nanostructured Tribological Coatings Parameters (AR and GS),  
achieved from a binary compound material (WC) or a ternary compound material (WCN)**

Sample No	Deposition Process Parameters								Results						
	Sputtering target material	Flow of the working gases		Working pressure [hPa]	Substrate parameters for the coating process		Magnetron sputtering parameters in DC, at 20% of $P_{max}$		Deposition time [minutes]	Deposition rate [ $\text{\AA}/s$ ]	Estimated thickness [nm]	Deposited material	Average Roughness** (AR) for a investigated surface of:		Grains size** (GS) [nm]
		Ar [sccm]	N2 [sccm]		Temperature [°C]	Bias voltage [kV]	Voltage [V]	Current [mA]					2,5 $\mu\text{m}^2$ [nm]	5 $\mu\text{m}^2$ [nm]	
1	WC	100	0	2,9-2,95x10 <sup>1</sup>	50	0	568-548	119-118	10	4,40-4,00	252	W.C.			1,21
2	WC	100	0	2,9-2,95x10 <sup>-3</sup>	50	0,6	567-552	118-117	10	4,40-4,00	252	W.C.			1,09
3	WC	100	20	2,95-3x10 <sup>3</sup>	50	0,6	569-547	118-117	18	4,40-3,90	448	W.C.N.			3,13
4	WC	100	0	2,9-2,95x10 <sup>-3</sup>	450	0	581-577	118-116	15	4,40-4,00	378	W.C.			1,30
5	WC	100	40	2,95-3x10 <sup>3</sup>	550	0,4	585-571	117-116	20	4,20-3,80	480	W.C.N.			11,00
6	WC	300	0	5,4-5,6x10 <sup>-3</sup>	50	0	459-430	119-118	30	4,10-3,70	702	W.C.	1,56	6,23	8,62
7	WC	300	60	1,4-1,5 x10 <sup>-2</sup>	50	0,5	469-466	119-118	30	4,00-3,60	684	W.C.N.	1,59	0,77	18,10
8	WC	300	0	5,4-5,6x10 <sup>-3</sup>	550	0	469-443	118-117	30	4,10-3,70	702	W.C.	0,54	0,92	2,64
9	WC	300	60	1,4-1,5 x10 <sup>-2</sup>	550	0,5	464-463	118-117	30	4,00-3,60	684	W.C.N.	0,40	0,82	1,80
10	WC	300	0	5,4-5,7x10 <sup>-3</sup>	250	0	452-441	118-116	30	4,10-3,70	702	W.C.	0,59	1,53	4,00
11	WC	300	60	1,4-1,5 x10 <sup>-2</sup>	250	0,5	473-468	118-117	30	4,00-3,60	684	W.C.N.	0,33	0,61	2,22

\* measured in-situ with crystal quartz microbalance

\*\* measured by Atomic Force Microscopy (AFM)

# Nanostructured dry-lubricant coatings with ternary composition from compound materials, by Magnetron Sputtering deposition method

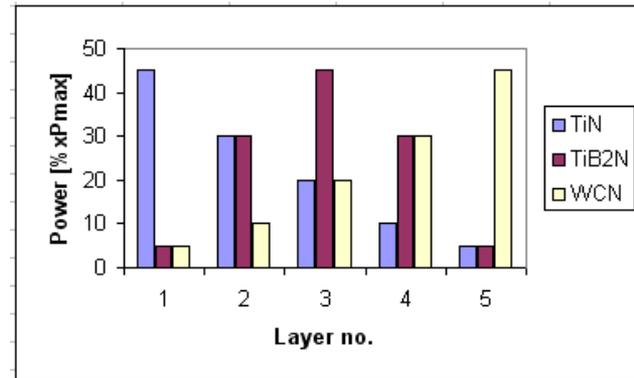
- Preliminary tests of new and complex dry lubricant coatings with ternary composition from compound materials (*Ti<sub>x</sub>N<sub>y</sub>; TiB<sub>2</sub>; WC* or *Ti<sub>x</sub>N<sub>y</sub>; Ti<sub>x</sub>ByN<sub>z</sub>; W<sub>x</sub>CyN<sub>z</sub>*) and multiple structure of: mono-layer type with constant or variable composition and multi-layer type, using AFM and tribological characterizations.

**- Deposition methods:**

- Standard or Reactive Magnetron Sputtering Deposition Method in DC from commercial TiB<sub>2</sub> and WC targets

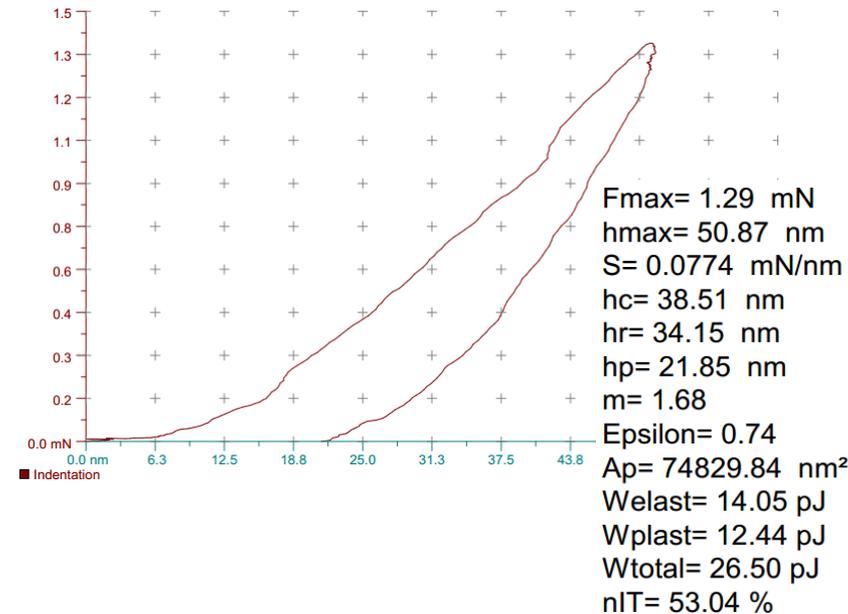
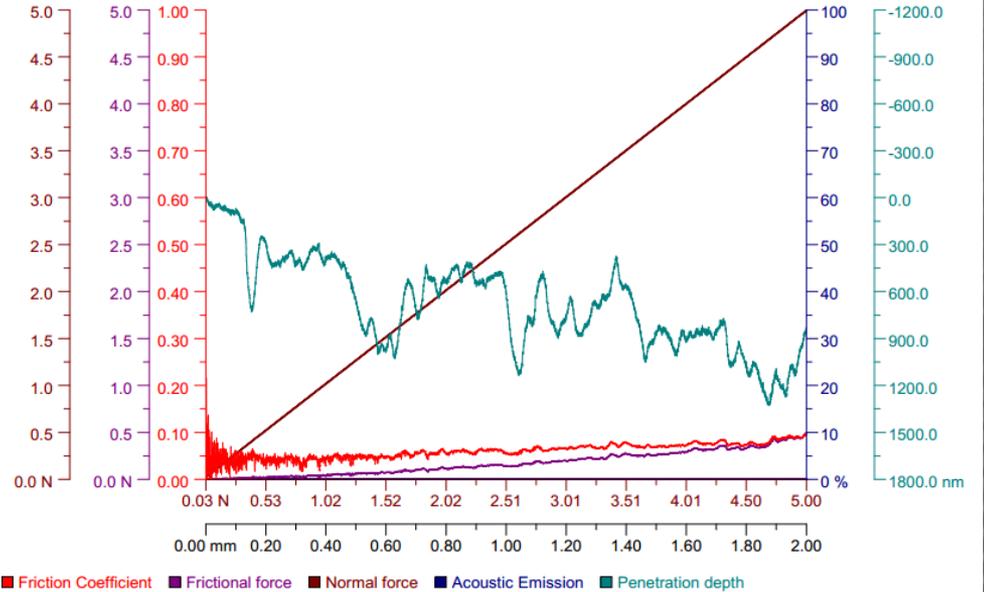
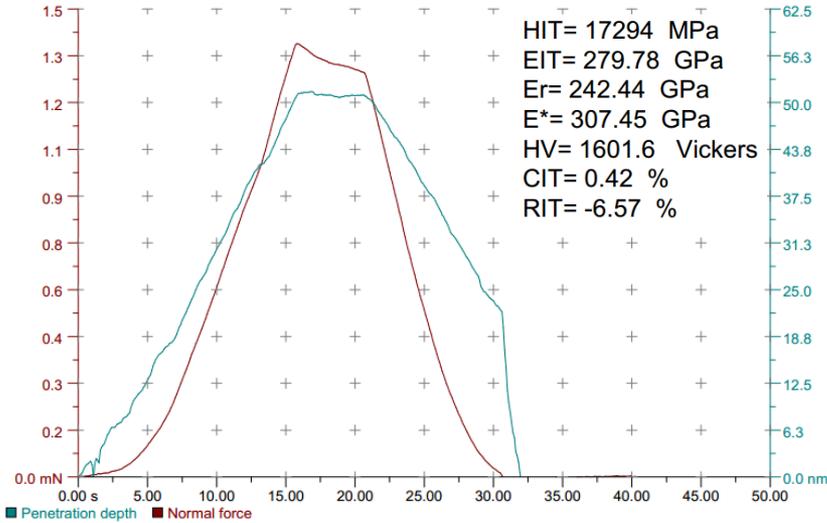
- DC Reactive Magnetron Sputtering Deposition Method with N<sub>2</sub> as reactive gas using Ti/ TiB<sub>2</sub>/ WC commercial targets.

Samples with ternary multiple layers					
Sample No.	Material of the target and Deposition Material		Pressure [mbar]	Por./ layer and package [% x Pmax]	Deposition time
	Target/ Gun	Package no. x (Types of deposited materials on a layer)			
1	Ti/ Gun-4 TiB <sub>2</sub> /Gun-2 WC/ Gun-1	6x(TiN+TiB <sub>2</sub> N+WC N <sub>2</sub> )	3,5x10 <sup>-3</sup>	45% for Ti 5% for TiB <sub>2</sub> 5% for WC  30% for Ti 30% for TiB <sub>2</sub> 10% for WC  20% for Ti 45% for TiB <sub>2</sub> 20% for WC  10% for Ti 30% for TiB <sub>2</sub> 30% for WC  5% for Ti 5% for TiB <sub>2</sub> 45% t WC	(5min/layer; 25min/package) 150min/6 package



Working gases: Argon 100 sccm; Nitrogen 40 sccm  
 Substrate temperature: 550 °C  
 Substrate bias voltage: 0,5 kV

# Tribological tests



## Tribometer module / Version 4.4.K

### Acquisition

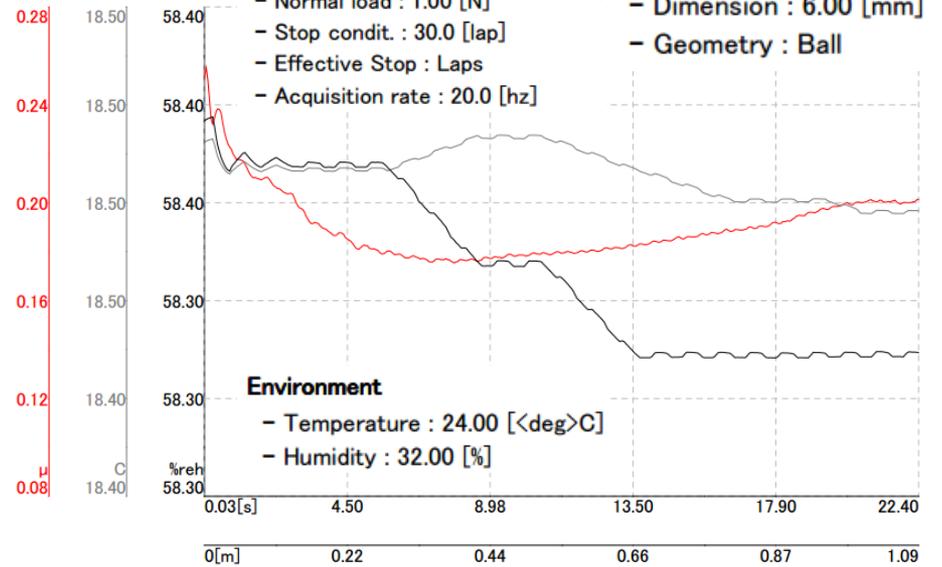
- Radius : 6.00 [mm]
- Lin. Speed : 5.00 [cm/s]
- Normal load : 1.00 [N]
- Stop condit. : 30.0 [lap]
- Effective Stop : Laps
- Acquisition rate : 20.0 [hz]

### Static partner

- Coating : 100Cr6
- Dimension : 6.00 [mm]
- Geometry : Ball

### Environment

- Temperature : 24.00 [deg>C]
- Humidity : 32.00 [%]



- **Risk analysis and contingency plan (lessons learned)**

- **Identified risks :**

1. *Difficulties in achieving of the required tribological characteristics by magnetron sputtering or APCPPD for space applications:* Low risk- previous work has lead to promising results, patent applications support a technology readiness level 3, therefore an “iterative” synthesis procedure should lead to the expected results and to a specific synthesis protocol.

2. *Failure of the APCPPD method as coating procedure for space applications:* Medium risk- APCPPD is a new but versatile technology. It allows coating processes with metal, polymers or semi-conductor layers on a wide variety of basic substrates such as paper, cardboard, textiles, ceramics, glass, metal and polymers. As contingency plan, other deposition method- magnetron sputtering and/or plasma deposition- should be considered.

3. *Partner withdrawal:* Low risk- highly unlikely that a partner withdraws. In that case, the other partners shall take over and the specific tasks shall be accomplished by externalization (after fulfilling legal formalities).

## Project's contribution to the goal of the STAR Programme

(how the project contributes to the increasing of the capacity for organizations involved to participate in ESA Programmes)

By the results obtained in this research project the organizations involved will improve their expertise in designing, achievement and characterization of the Dry Lubricant Coatings for friction couples with application in the aerospace field and finally will improve their competitiveness to participate at ESA Programmes.

## Context and contribution to ESA Programmes

(please specify how the project activities can contribute to present and future ESA programmes)

The very new technology (Atmospheric Cold Plasma Powder Deposition Method) that was not used yet in this field could be a “disruptive innovative technology with special priority” asked by the Basic Research Programme (TRP Programme) of the European Space Agency.

The claims of the Patent A00603 of AEG PROGRESIV, granted by OSIM in December 2013, as also the Patent Applications of AEG PROGRESIV, registered at OSIM with numbers A01074 and A01075 from 2012 will be validated in this project and will find applications as Dry Lubricant Coatings in ESA Programmes.

- **Dissemination activities**

- **Planned:**

- 2 scientific papers for peer reviewed journals
    - 3 participations (oral or poster) at International Conferences or Symposia
    - 1 patent application

- **In preparation:**

- **1 conference:** The International Conference “Materials and Modeling Technologies-2014”
    - **2 articles**

## ■ **Conclusions**

In the phase from this year we have started:

1. The research activities for designing & achievement of the tribological coatings, using Magnetron Sputtering Deposition Method in DC and RF as also their structural, compositional and tribological characterization as was presented above.
2. Acquisition of the Equipment for Open Atmosphere Cold Plasma Powder Deposition and Plasma Surface Treatment.

SpaceCoat is complex and ambitious project, since there is a multitude of the tribological materials with low friction coefficient (“chalcogenide” type-MX<sub>2</sub> where M=Metal; X=S; Se; Te; PTFE; Graphite; hBN; nitride, carbide and carbo-nitride type, etc) as also a multitude of deposition methods with development both in vacuum and in open atmosphere (starting from nanopowders or micropowders and with lower manufacturing cost) that have a high potential for use in industrial applications.