Nanocomposite Coatings on Tribological Application in Automotive Engine

V. Sathiyaraj, P. Naveenchandran and C. Balakrishnan

Abstract— *The structure, hardness, grinding and wear of tungsten nitrides arranged by DC receptive magnetron sputtering were researched. The coatings were stored with various nitrogen to argon proportions; the aggregate weight was kept consistent. The tribological tests were performed on the covered example in earthbound air with SS304 stainless steel. The coatings showed diverse stages as a component of the nitrogen content: movies with low N content displayed the α-W stage; β-W stage was prevailing for nitrogen substance from 7 to 10 at.% and β-W2N was watched for nitrogen content at 15at.%. We report the readiness of tungsten nitride movies developed by receptive sputtering in the encompassing N2/Ar blend at different weights on stainless steel substrates at different constant temperatures. The structural property is analysed using XRD, mechanical properties are evaluated by nano indentation and metallurgical properties by AFM, EDAX in view of the affidavit conditions. The relationship between the microstructure and mechanical properties is talked about. The mechanical and tribological properties of the tungsten nitride coatings were unequivocally affected by the structure. The hardness and the Young's modulus qualities were in the reaches (27–39GPa) and (239-280GPa), individually; the most minimal qualities compare to the coatings with the most astounding nitrogen content.*

For the most part, the grating and wear rate of tungsten nitride coatings sliding against fumes gas expanded with nitrogen content achieving a greatest at 12 at.%; additionally increment of the nitrogen content prompted to a reduction of the erosion and wear.

Keywords--- *Hard Coatings, Nanoindentation, Tungsten Nitride, Reactive Sputtering, PVD.*

I. INTRODUCTION

Nano coatings have become indispensable as tribological layers of Engine compartments. In modern automobiles, for high-temperature applications and high friction in the engine parts, anti-friction coatings, multilayer nano coatings, tribological nano coatings are introduced for their best wear and corrosion resistance properties. For high operating temperature and a high-compression loading capability applications, Tungsten Nitride coatings is introduced to reduce the corrosion, 3X wear and friction. So I have selected Austenitic stainless steels (SS304) which is used for exhaust valve. Due to less thermal conductivity and an adequate coating surface quality, this can lead to a low friction coefficient between the parts. Hence it is treated by tungsten nitride of 4 samples with different conditions coatings kept by receptive magnetron sputtering.

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This formation of thin layer forms a shielding outer-layer of base material. This metal oxide will create a thin layer coating or shell against wear resistance and corrosion resistance. It will gives

a. corrosion resistance (Salt spray life)
b. good tribological application
c. Elevated temperature strength applications of automotive parts
d. Low coefficient of friction
e. Less stress corrosion cracking
f. Mechanical properties such as strength, ductility, and toughness will be modified.
g. Due to the development of a defensive nitride layer at the surface, expanded oxidation resistance at hoisted temperature.
h. Due to phase transformation changes and solid solution hardening, hardness is creased in the deposited films (surface)

The hardness and the Young's modulus qualities were in the reaches (23–33 GPa) and (300–390 GPa), separately; the most minimal qualities compare to the coatings with the most elevated nitrogen content.further increment of the nitrogen content prompted to a lessening of the grinding and wear. This fumes valve with high temperature did not wear/erosion the coatings under the chose testing conditions

II. EXPERIMENTAL CONDITION

SPUTTERING is a non thermal vaporization handle in which surface iotas are physically launched out from a surface by force exchange from a vigorous besieging types of nuclear/sub-atomic size. Normally, sputtering utilizes a sparkle release or a particle pillar to create a flux of particles episode on the objective surface. These particles cause iotas, and once in a while bunches of molecules, to be thumped free from the objective surface by effect exchange, or sputteringns. Sputtering is used in two principal applications: sputter etching, in which the primary objective is removal of material from the target surface and sputter deposition, in which redeposition of these sputtered molecules onto another surface, or substrate, is the primary goal. The latter application is discussed in this article. The fundamentals of plasma formation and the interactions on the target surface are discussed first, followed by the differences between reactive and nonreactive sputtering, and several methods of process control. In the third section, the basic principles and relative advantages and disadvantages of the most common sputtering techniques are examined, specifically direct-current (dc) diode, radiofrequency(rf) diode, triode, magnetron sputtering, and a relatively new technique known as "unbalanced" magnetron sputtering.

Contrasted with other thin-film affidavit strategies, sputter testimony procedures have a few unmistakable preferences:

· Use of an unlimited range of source and film materials (i.e., metals, semiconductors, insulators, alloys, and compounds)

- Small sputtering-yield variations from one material to another as compared to the relative variation in the evaporation rates at a given temperature
- Ease of low-temperature deposition of refractory materials
- Elimination of droplet emission from the source that can occur in thermal evaporation
- Absence of droplets, which are common in arc-deposited films
- Ease of forming multicomponent films

- Uniformity of film thickness over expansive zones
- High level of film attachment
- Environmentally friendly processing
- Sputter deposition processes have several limitations as well:
- Target (source) materials must ordinarily be in sheet or tube form.
- Deposition rates are regularly under 300 nm/min (3000 A/min).

Setup costs are high because of the required vacuum environment.

- The brakes must be strong to stop the vehicle during emergency within a shortest distance.
- The driver must have proper control over the vehicle during braking and vehicle must not skid.
- The brakes must have well anti fade characteristics i.e. their effectiveness should not decrease with constant prolonged application.
- The brakes should have good anti wear properties.

Fig. 1 Vacuum Sputtering machine

Four samples are taken for the coatings with different condition. Technical specification is given below

Table: 1 Sputtering Condition

Condition	Sample 1	Sample 2	Sample 3	Sample 4
Base Vacuum	$5.9x10^{-6}$ mbar	$4.8x10^{-6}$ mbar	$5.9x10^{-6}$ mbar	$4.8x10^{-6}$ mbar
Argon pressure	20 SCCM	20 SCCM	20 SCCM	20 SCCM
N_2 pressure	3 SCCM	5 SCCM	10 SCCM	15 SCCM
RF Power	100 W	100 W	100 W	100 W
Cr inter layer	10 min	10 min	10 min	10 min
DC	360 V	380 V	360 V	380 V
Current	0.72 amp	0.30 amp	0.42 amp	0.58 amp
Time	1hour	1hour	1hour	1hour
Temperature	500º C	500º C	500º C	500º C
Chamber Pressure	$5x10^{-3}$ mbar	$8x10^{-3}$ mbar	$4x10^{-3}$ mbar	$9x10^{-3}$ mbar

III. CHARACTERIZATION OF NANOCOMPOSITE FILMS

Film portrayal is the unavoidable and vital stride for guaranteeing of excellent film for the expected application. Distinctive portrayal procedures can be utilized to recognize nanocomposite coatings.

3.1 Scanning electron microscope (SEM)

Examining electron microscopy is an exceptionally valuable method in the portrayal of thin movies and without a doubt the most generally utilized of all electron pillar instruments. SEM is a technique for high-determination imaging of surfaces. Examining electron microscopy gives amplified pictures by utilizing electrons rather than light waves (in the event of ordinary light magnifying lens). The benefits of SEM over light microscopy incorporate much higher amplification (> 1,000,000×) and more prominent profundity of field up to 100 circumstances. The determination of advanced checking magnifying instruments is normally <10 nm (picture determination of around 0.5 nm can be presently accomplished in the latest era field-outflow weapon SEM), so on a fundamental level SEM could be utilized to gauge nanocrystalline grain measure. The issue is getting the fitting surface complexity for grain sizes in the nanoscale administration. To make the SEM picture, the occurrence electron shaft is filtered in a raster design over the specimen's surface. At the point when an electron shaft communicates with a mass example, an assortment of signs can be created. Figure 7 demonstrates the different sorts of electrons and electromagnetic radiation delivered when a high vitality electron shaft cooperates with a mass example (Liu, 2005).

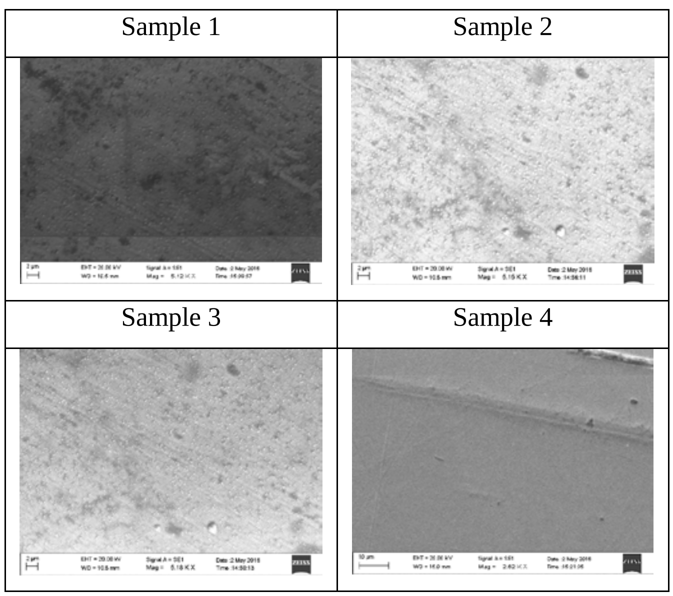

Fig.4.SEM micrographs of all coating cross-sections showed typical columnar morphology

3.2 Chemical composition (EDAX)

The compound structure of the stored coatings (measured by EDAX) is appeared in Fig. 1 It makes utilization of the X-beam range radiated by a strong specimen shelled with centered light emission to get a restricted concoction examination. All components from nuclear number 4 (Be) to 92 (U) can be identified on a fundamental level, however not all rinstruments are prepared for "light" components (Z < 10). Subjective examination includes the recognizable proof of the lines in the range and is genuinely direct inferable from the effortlessness of X-beam spectra. Quantitative examination (assurance of the groupings of the components display) involves measuring line powers for every component in the specimen and for similar components in alignment Standards of known piece.

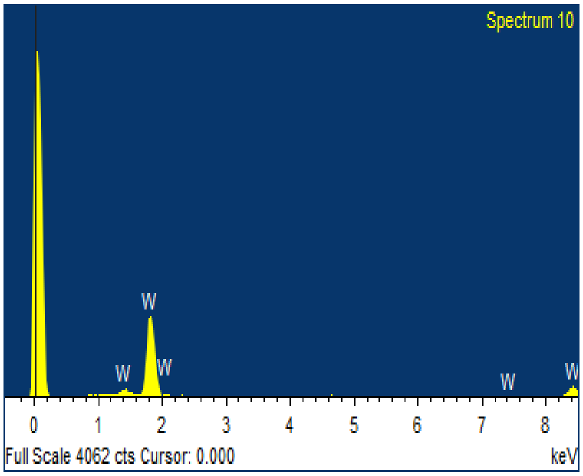

Fig. 5. EDAX of tungsten nitride coatings

Table: 2 - EDAX Results

Element	Weight%	Atomic%
W	100.00	100.00
Total	100.00	

3.3 X-ray Diffraction

The XRD diffractograms of tungsten nitride coatings are delineated in Fig. 2. The structure of the coatings can be connected with their concoction sythesis. In Fig. 1 three primary zones are considered:

(i) for low nitrogen content (N< 9 at.%),

the coatings showed a structure run of the mill of W with (1 0) particular introduction, (ii) for coatings with middle of the road nitrogen content, W88 N12 and W85 N15, the structure is a blend of stages with the normal W stage blended with W and W2N, individually,

(iii) for coatings with the most astounding nitogen substance (N > 33 at.%) the structure is just shaped by the NaCl-sort nitride compound, _-W2N, with (2 0)preferential introduction.

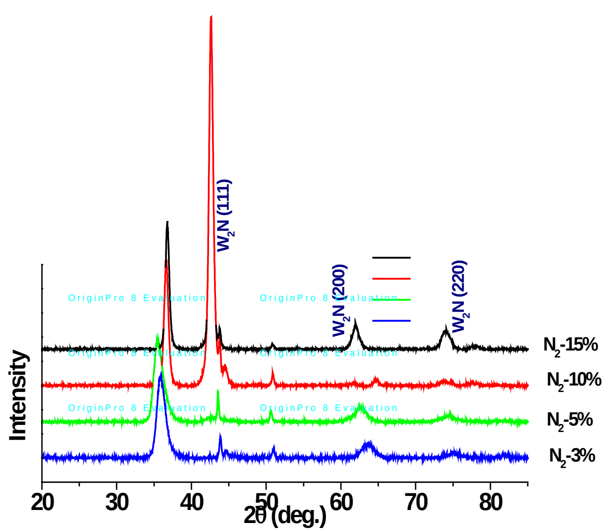

Fig.6. XRD diffractograms of tungsten nitride

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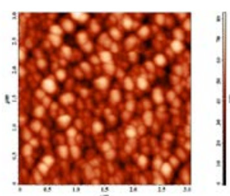
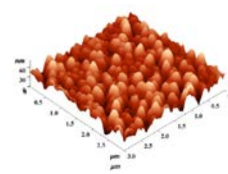
3.4Atomic force microscopy (AFM)

Nuclear compel microscopy has turned into a capable device to distinguish auxiliary changes at the nanoscale. The significance of AFM strategy, is further expanding with late improvements in nanoscience and nanotechnology. The extent of AFM applications incorporates high-determination examination of surface geography compositional mapping of heterogeneous examples and investigations of neighborhood mechanical, electric, attractive and warm properties. These estimations can be performed on scales from several microns down to nanometers.

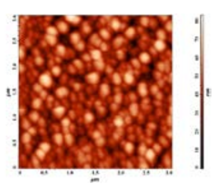
AFM gives a 3D profile of the surface on a nanoscale, by measuring powers between a sharp test (<10 nm) and surface at short separation (0.2-10 nm test division). The test is bolstered on an adaptable cantilever. The AFM tip "tenderly" touches the surface and records the little constrain between the test and the surface. Here harshness and geography have studied for 4 tests' at various covered condition.

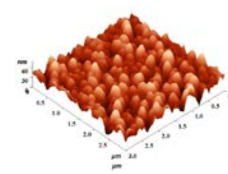
Sample 1



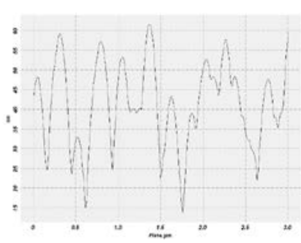


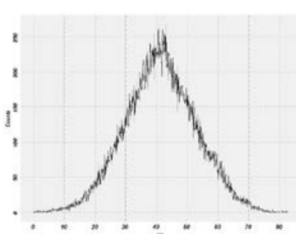
2D3x3





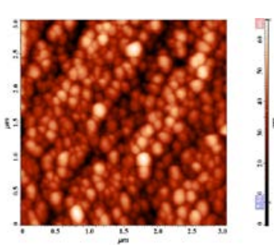
3D– 3x3

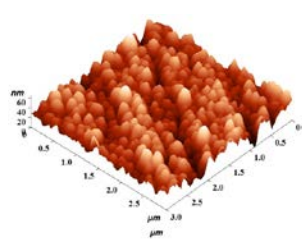




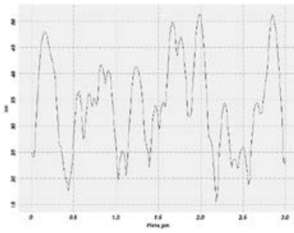
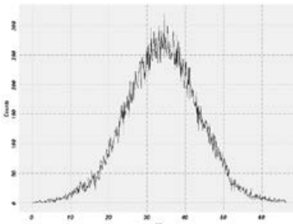
Histogram analysis **X section Line profile**

Sample 2



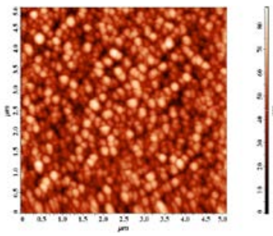
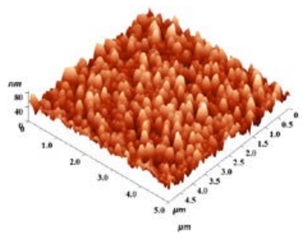


2D – 3x3 **3D– 3x3**

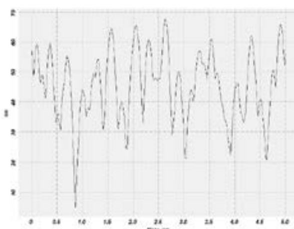
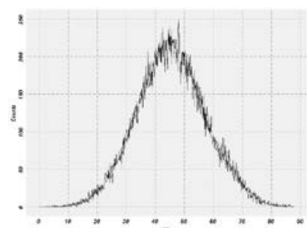



Histogram analysis **X section Line profile**

Sample 3

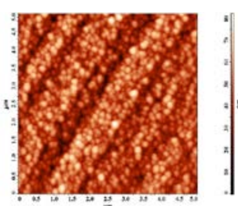
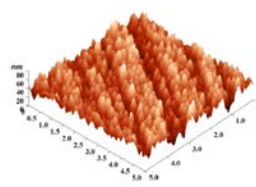



2D – 5x5 **3D–5x5**

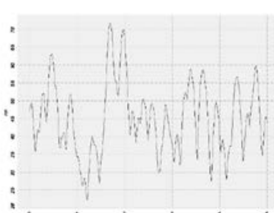
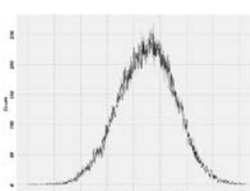



Histogram analysis **X section Line profile**

Sample 4

2D – 5x5 **3D– 5x5**

Histogram analysis **X section Line profile**

Fig. 7. AFM topography of the WN coating

This atomic micro photography reveals fine grain size which means that very less friction and withstand the tribological properties.

Table: 3 - AFM / Roughness analysis

Properties	Sample 1	Sample 2	Sample 3	Sample 4
Amount of sampling	65536	65536	65536	65536
Max	66.3686 nm	82.0749 nm	82.8411 nm	87.7253 nm
Min	0 nm	0 nm	0 nm	0 nm
Peak-to-peak, Sy	66.3686 nm	82.0749 nm	82.8411 nm	87.7253 nm
Ten point height, Sz	33.3567 nm	41.6887 nm	41.4397 nm	44.6863 nm
Average	33.9025 nm	45.1078 nm	41.3728 nm	45.4906 nm
Average Roughness, Sa	7.56173 nm	8.56787 nm	9.179 nm	9.49295 nm
Root Mean Square, Sq	9.53021 nm	10.6748 nm	11.5725 nm	11.9149 nm
Second moment	1240.21	2148.67	1845.63	2211.36
Surface skewness, Ssk	-0.0140797	-0.0616565	0.0033619	0.0652525
Coefficient of kurtosis, Ska	0.087297	-0.0999154	-0.0333027	-0.0740959
Entropy	8.67212	8.83643	8.95148	8.99365
Redundance	-0.438037	-0.393498	-0.408627	-0.39688

3.5 Grain size

In many investigations of the grain size of nanocrystalline materials X-beam line-widening examination or direct estimations by transmission electron microscopy are performed. To assess the grain estimate, the outstanding Scherrer equation is utilized by measuring the full width at half most extreme (FWHM) of the XRD diffraction crest:

$$d = \frac{K\lambda}{\beta \cos\theta}$$

Where d is crystallite size, K is the shape factor, is the wavelength of the X-rays ,is the full width at half-maximum XRD peak in radians

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Table: 4 – Grain Size

Properties	Grain Size
Sample 1	33.9025
Sample 2	45.1078
Sample 3	41.3728
Sample4	45.4906
Substrate	5 ASTM

3.6 Nano Indentation

A nano hardness survey is conducted on the four samples with different condition. The first line shows that load vs depth and the return lines reveals unloading vs depth.

Results:

Table: 5 - Nano Indentation

Properties	Hardness in GPa	Young's Modulus in GPa
Sample 1	33.306829	280.226577
Sample 2	27.306002	239.898735
Sample 3	39.994859	277.977281
Sample4	33.240213	254.973059
Substrate	1.96 GPA	140.93

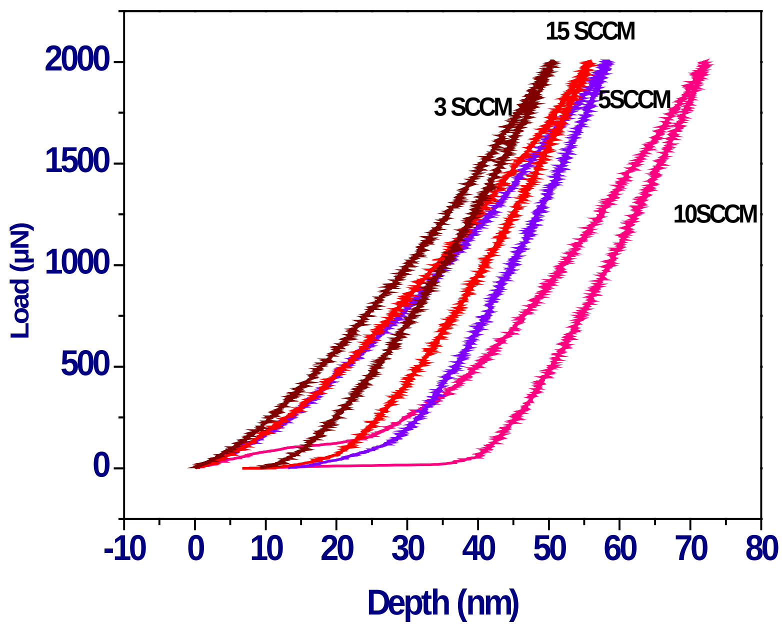
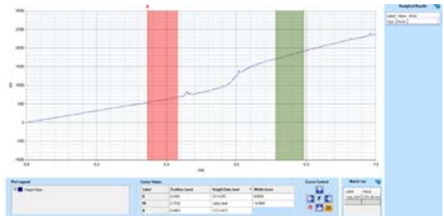


Fig. 8. Nano indentation graph of the WN coating

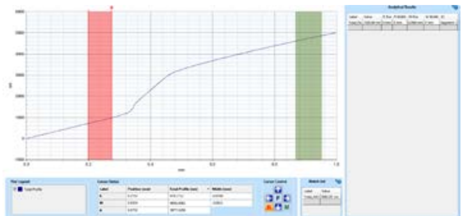
3.7Thickness Survey

A thickness survey is conducted on the four samples to find the surface thickness on the sample. The results are given below.

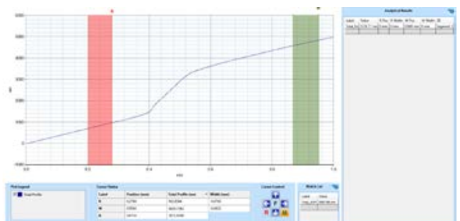
Sample 1



Sample 2



Sample 3



Sample 4

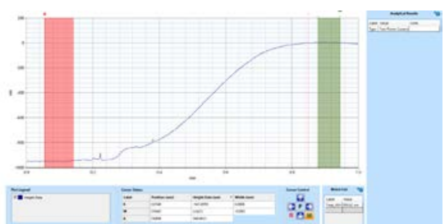


Fig. 9. Thickness measurement

Results

Table: 6 - Surface Thickness

Properties	Surface Thickness in nm
Sample 1	1208
Sample 2	3882
Sample 3	3867
Sample4	950

IV. RESULTS & CONCLUSION:

Structural and surface morphological properties of epitaxial Tungsten Nitride thin flims deposited on SS304 stainless steel substrate were investigated.

Mechanical properties such as strength, ductility, Hardness, young’s modulus and toughness will be modified due to the formation of a protective nitride layer at the surface.

Surface roughness and thickness is very smooth and hence the friction is retained.

Table: 7 - Results & Conclusion

Properties	Grain Size	Hardness in GPa	Young's Modulus in GPa	Surface Roughness in nm	Surface Thickness in nm	EDAX	Chemistry
Sample 1	33.9025	33.306829	280.226577	7.56173	1208	Tungsten peak	NA
Sample 2	45.1078	27.306002	239.898735	8.56787	3882	Tungsten peak	NA
Sample 3	41.3728	39.994859	277.977281	9.179	3867	Tungsten peak	NA
Sample4	45.4906	33.240213	254.973059	9.49295	950	Tungsten peak	NA
Substrate	5 ASTM	1.96 GPA	140.93	-	-	Cr & Ni Peak	C- 0.068% Cr-18.93% Ni -9.81% Si -0.48% Mn - 1.55%

V. FUTURE TRENDS

The nanocomposite coatings are still at the beginning of their development. Further research activity in the field of nanocomposite films and coatings should be concentrated on the following problems:

- development of films with controlled grains size,
- development of new technology system for production of nanocomposite coatings in new physical and/or chemical conditions,
- increased utilization of coatings in many types of applications including those in the aerospace and automotive industries,
- development of protective coatings with oxidation resistance exceeding 2000ºC,
- development of hybrid coatings with nanophase biomaterials for biomedical applications,
- nanocrystallisation of amorphous materials,
- development and scale-up of deposition processes,
- development of high fracture toughness coatings,
- development of new advanced coatings with unique physical and functional properties.

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