HABILITATION THESIS

MULTIFUNCTIONAL THIN SOLID FILMS

Lect. Dr. Eng. Daniel CRISTEA

Domain: Materials Engineering

Universitatea Transilvania din Brașov
FACULTATEA DE ȘTIINȚA ȘI INGINERIA MATERIALELOR

Content

Professional achievements

- Timeline:
  - 2009 - Bachelor's degree in Materials Science
  - 2011 - Master's programme titled "**Engineering and Management of Advanced Metallic, Ceramic and Composite Materials**"
  - 2009 - 2011: mechanical engineer at Brasov Fasteners Inc.
  - 2013 - PhD thesis: **"Research on the synthesis and characterization of MeO_xN_y system thin films deposited by reactive magnetron sputtering"**
  - 2014 -2015: postdoctoral fellowship **POSDRU/159/1.5/S/134378**
  - 2015 - present day: **Lecturer** at Materials Science Department, Transilvania University of Brasov.

Professional achievements

- Traineeship or research exchange programs:
  - 2017 - Instrumented indentation at **Queen Mary University of London**, **United Kingdom**;
  - 2019 - Simulated concentrated solar energy experiments at **Middle Eastern Technical University, Ankara, Turkey**, 2019;
  - 2019 - Sputtering deposition chamber maintenance and repair at **Minho University, Physics Department, Portugal**;
  - 2019 - Additive manufacturing summer school at **University of Modena and Reggio Emilia, Italy;**
  - 2020 - Dual magnetron sputtering and corrosion analysis at the **Research Institute for Precious Metals and Metal Chemistry**, **Germany**;
  - 2020 - Concentrated solar energy experiments at **PROMES-CNRS, Odeillo, France**;

Professional achievements

- Research subjects and publications:
  - DC and RF Sputtering, pulsed laser deposition, High-power impulse magnetron sputtering (HiPIMS), surface characterization (nanoindentation, wear and tribology, adherence of thin films to substrates, corrosion resistance, biocompatibility, photocatalysis, electrical, and optical analysis);
  - **47 ISI-WOS papers** (42 indexed, 42 in journals and 5 proceedings papers) and **14 BDI papers**

Professional achievements

- Projects (leader):
  - 2014 - **Postdoctoral fellowship** POSDRU/159/1.5/S/134378;
  - 2017 - **Young Researchers Grant** (Transilvania University of Brasov) 8022/2017 – "Wear resistant thin solid films";
  - 2019 - **SFERA III Access Grant**: "Concentrated solar radiation fast sintering of novel metastable Al-Si-Ni alloys, as potential raw materials for additive manufacturing", Middle Eastern Technical University, Ankara, Turkey, 2019**;**
  - 2020 - **SFERA III Access Grant**: "Novel Ti-based biocompatible alloy coatings from powders sintered onto Ti6Al4V substrates using concentrated solar radiation", PROMES-CNRS, Odeillo, France, 2020;
  - 2020 - RM-Mg-N multiple component nitride-type thin films, obtained by simultaneous sputtering of two metallic targets. **DAAD German Academic Exchange Service**;

Professional achievements

- Projects (leader):
  - 2020 - PN-III-P1-1.1-TE-2019-1209: "**Magnetron sputtered Me-Me binary oxynitride multifunctional thin solid films**", financed by UEFISCDI;
  - 2020 - **nr. ctr: 13440 -16/11/2020 (value 72000 RON)**. "Performing mechanical and tribological tests for samples with tribological coatings (Hardness Test, Scratch Test, Pin/Ball on Disk Tribometer Test)" financed by: IFIN-HH Horia Hulubei National Institute for Physics and Nuclear Engineering, Magurele, Romania.

Professional achievements

- Projects (member):
  - **RIA - Horizon 2020, (2016 - 2019) - FOF-13-2016**: Photonics Laser-based production: DREAM (Driving up reliability and efficiency of additive manufacturing);
  - **PN-III-P2-2.1-BG-2016-0241** - Optimizing the inductive quenching eco-technology for large bearing rings. Bridge Grant 2016-2018.
  - Multilayer inorganic/organic tribological coatings for space applications - financed in the Research-Development-Innovation for Aerospace Technologies and Advanced Research Program **(STAR) - ctr. no. 68/2013. 2013 – 2016**.
  - **Grant FP7-INFRA-312643- SFERA II (2016)**: "Researches regarding the influence of the heat treatments with solar energy over the wear resistant steels properties".

Professional achievements

- Projects (member):
  - Survey Services: Surface Features of Dental Implants: Mechanical Characteristics. Period: 2017-2018 Funding: DENTIX contract no: 144 / 09.01.2017;
  - **PCCDI 58** - New Diagnosis and Treatment Methodologies: Active Challenges and Technological Solutions Based on Nanomaterials and Biomaterials - Acronym: SANOMAT, period 2018-2020, financed by UEFISCDI.
  - "Solar-assisted treatment of some new stainless steels for biomedical applications", Acronym: SOLARBIOMAT CIEMAT-PSA contract no: FP7-INFRA-312643
  - **Premiere H2020** – DREAM, 2017-2019 financed by UEFISCDI nr.ctr: 15/2017 PN-III-P3-3.6-H2020-2016-0077

Professional achievements

Books:

1. I. Ghiuta, **D. Cristea**. *Silver nanoparticles for delivery purposes* (Chapter) - Nanoengineered Biomaterials for Advanced Drug Delivery 1st Edition, Elsevier, isbn: 9780081029855, 2020;
2. **Daniel Cristea**, Luis Cunha, Aurel Crișan, Daniel Munteanu. *Oxynitride thin solid films*, Transilvania University Publishing, isbn: 978-606-19-0450-1, 2014, 201 Pages;
3. Ioana Ghiuță, **Daniel Cristea**, Daniel Munteanu. *Metallic nanoparticles synthesis,* Transilvania University Publishing, isbn: 978-606-19-1011-3, 2018, 183 Pages;
4. Camelia Gabor, **Daniel Cristea**, Mariana Axente. *Isostatic compaction of thin layers obtained by thermal spraying*, Printech, isbn: 978-606-23-0988-6, 2019, 319 Pages;
5. **Daniel Cristea**. *Advanced materials for renewable energies*, Printech, isbn: 978-606-23-1156-8, 2020, 204 Pages;
6. **Daniel Cristea**. *Nanomaterials*, Printech, isbn: 978-606-23-1144-5, 2020, 254 Pages.

Professional achievements

- **Reviews:**

  - Applied Surface Science (Elsevier),
  - Surface & Coatings Technology (Elsevier),
  - ACS Applied Materials & Interfaces (American Chemical Society),
  - Colloids and Surfaces A (Elsevier),
  - Materials Research Express (IOP Science),
  - Materials (MDPI),
  - Coatings (MDPI),
  - Physics Open (Elsevier),
  - PloS One,
  - Arabian Journal for Science and Engineering (Springer),
  - Metals (MDPI),
  - ACS Combinatorial Science (American Chemical Society),
  - Applied Sciences (MDPI),
  - Materials Research Express (IOP),
  - Surface and Interface Analysis (Wiley),
  - Materials Letters (Elsevier),
  - Results in Physics (Elsevier),
  - Journal of Environmental Chemical Engineering (Elsevier),
  - Journal of Materials Science (Springer),
  - Polymers (MDPI),
  - Materials Today Communications (Elsevier).

Professional achievements

- Minimum criteria (A1):

Nr. crt	Field of activities	Type of activities	Categories and restrictions	Subcategories	Minimum criteria	Achieved	Achieved score
1	Didactic and professional activity	1.1 Books and chapters in specialty books in recognized publishers	1.1.1 Books / chapters as an author	1.1.1.1 International	-	1	**6.25**
				1.1.1.2 National; of which: minimum 2 for Professor, of which 1 first author	2 Books, of which 1 first author	3, 1 as first author	**43.51**
			1.1.2 Books / chapters as an editor	1.1.2.1 International	-	-	-
				1.1.2.2 National	-	-	-
		1.2 Teaching support	1.2.1 Teaching manuals, monographs, including electronic: For Professor at least 2, of which 1 as first author		Minimum 2, of which 1 as first author	2, single author	**45.8**
			1.2.2 Laboratory guides / applications		-	-	-
				Score (A1)	**60**		**95.56**

Professional achievements

- Minimum criteria (A2):

Nr. crt	Field of activities	Type of activities	Categories and restrictions	Subcategories	Minimum criteria	Achieved	Achieved score
2	Research activity	2.1 Articles in ISI Thomson Reuters-Web of science Core Collection and ISI Proceedings indexed volumes	2.1.1 Minimum 15 articles for Professor, of which minimum 10 in ISI Th.R. (of which min. 5 with impact factor of min. 1, and minimum 5 as main author with F.I. min. 0.5		15 articles for Professor, of which at least 10 in ISI-listed journals, at least 5 as main author with F.I. min 0.5	40 ISI (8 as main author, F.I. >1) 5 ISI Proc.	**782.31**
		2.2 Journal articles and volumes of BDI indexed scientific events			-	14 BDI	**13.37**
		2.3 Patents			-	-	-
		2.4 Grants / projects won through competition	2.4.1 Director / Partner Manager (Minimum 2 for Professor)	2.4.1.1 International	Minimum 2	3	**60**
				2.4.1.2 National		2	**15**
			2.4.2 Team member	2.4.2.1 International	-	3	**20**
				2.4.2.2 National	-	5	**22**
				Score (A2)	**320**		**912.68**

Professional achievements

- <u>Minimum criteria (A3):</u>

Nr. crt	Field of activities	Type of activities	Categories and restrictions	Subcategories	Minimum criteria	Achieved	Achieved score
3	Recognition and impact of the activity	3.1 Citations in ISI-listed journals - Web of Science Core Collection and other BDIs	Minimum 30 citations for Professor, in ISI Thomson - Web of Science and SCOPUS	3.1.1 ISI	30 citations	197	**522.56**
				3.1.2 BDI		3	**0.82**
		3.2 Invited presentations in the plenary of national and international scientific events			-	-	-
					-	-	-
					-	-	-
		3.3 Member of the editorial boards or scientific committees of scientific journals and events / Reviewer of scientific journals and events		3.3.1 ISI	-	Reviewer for 20 journals	**103**
				3.3.2 BDI	-	Reviewer for 3 journals	**9**
				3.3.3 National and international non-indexed	-	-	-
		3.4 Expert evaluation of research projects		3.4.1 International	-	-	-
				3.4.2 National	-	-	-
				Score (A3)	**120**		**635.38**

Professional achievements

- Minimum criteria (summary):

Score (A1)	60	95.56
Score (A2)	320	912.68
Score (A3)	120	635.38
Optional criteria score	-	14
TOTAL SCORE	Minimum required 500	Achieved 1657.62

Scientific achievements

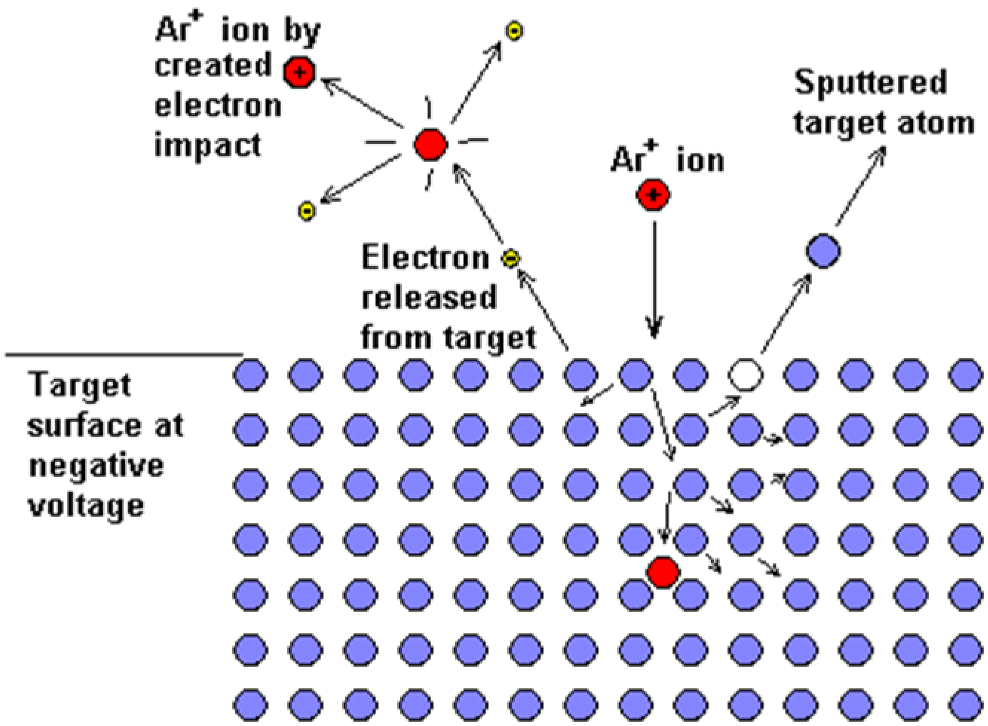
Multifunctional thin solid films: means of development

- One of the most frequently used methods to alter the surface properties of a material is to deposit a thin film or coating on top of the base material, which will significantly improve the functionality of the final part

- Sputter deposition stands out as a reliable, cost-effective, and flexible means of surface properties enhancement

Scientific achievements

- <u>Multifunctional thin solid films: means of development</u>



Scientific achievements

Multifunctional thin solid films: means of development

- When a reactive gas is added to the discharge or when the sputtering process is applied to at least two targets from the same chamber simultaneously, it becomes possible to deposit compound materials.

- One of the major problems of the reactive sputter process is its complexity

Scientific achievements

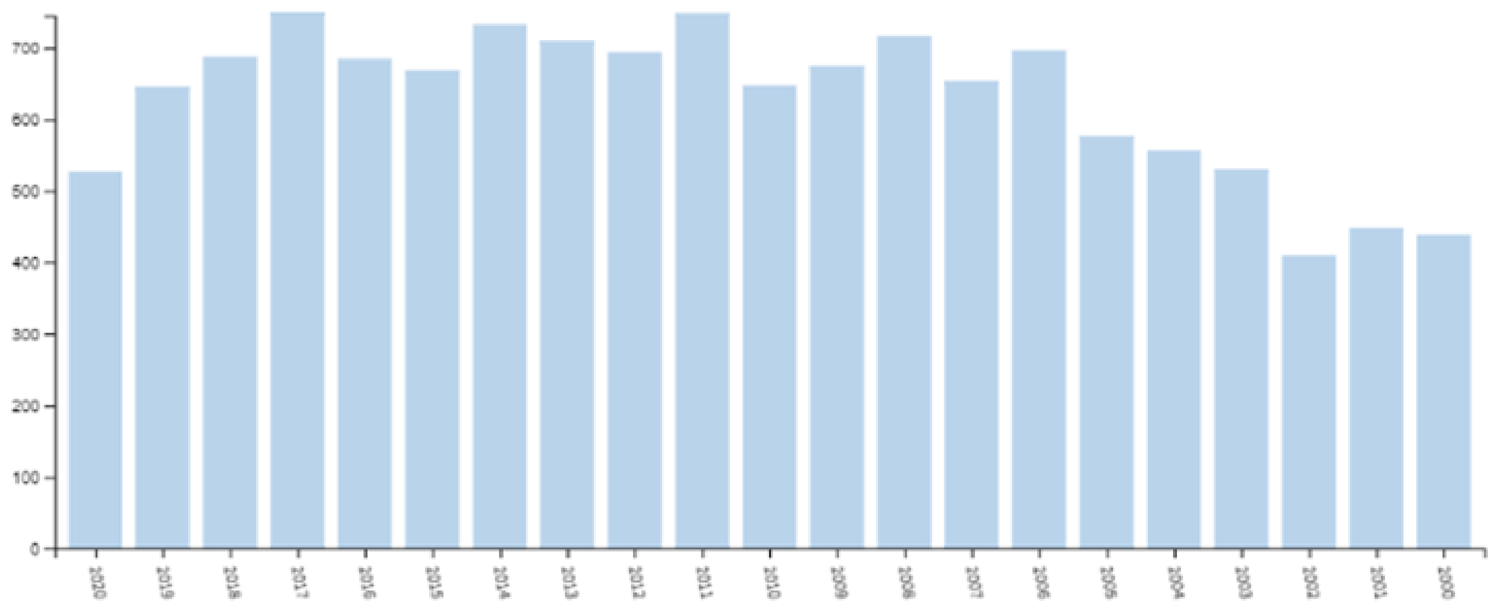
- Multifunctional thin solid films: means of development

  - **Target reactivity.** The reactivity between the gas and the target metal is clearly important.
  - **Sputtering yield.** The sputtering yield value for the metallic target is typically significantly higher than the sputtering yield value for the corresponding compound.
  - **Two (or more) reactive gases.** Oxygen and nitrogen may be added to the deposition chamber to form an oxynitride of some target material. It is likely that one of the gases may be more reactive than the other.
  - **Reactive co-sputtering.** Carrying out reactive sputtering from two metal targets increases significantly the complexity of the process

Scientific achievements

- **<u>Multifunctional thin solid films: means of development</u>**

  - **Motivation of the thesis subject.** The last two decades have seen the gradual rise at a steady but consistent pace of scientific publications related to reactive sputtering. Several key aspects remain to be solved.



Scientific achievements

- **Multifunctional thin solid films: means of development**

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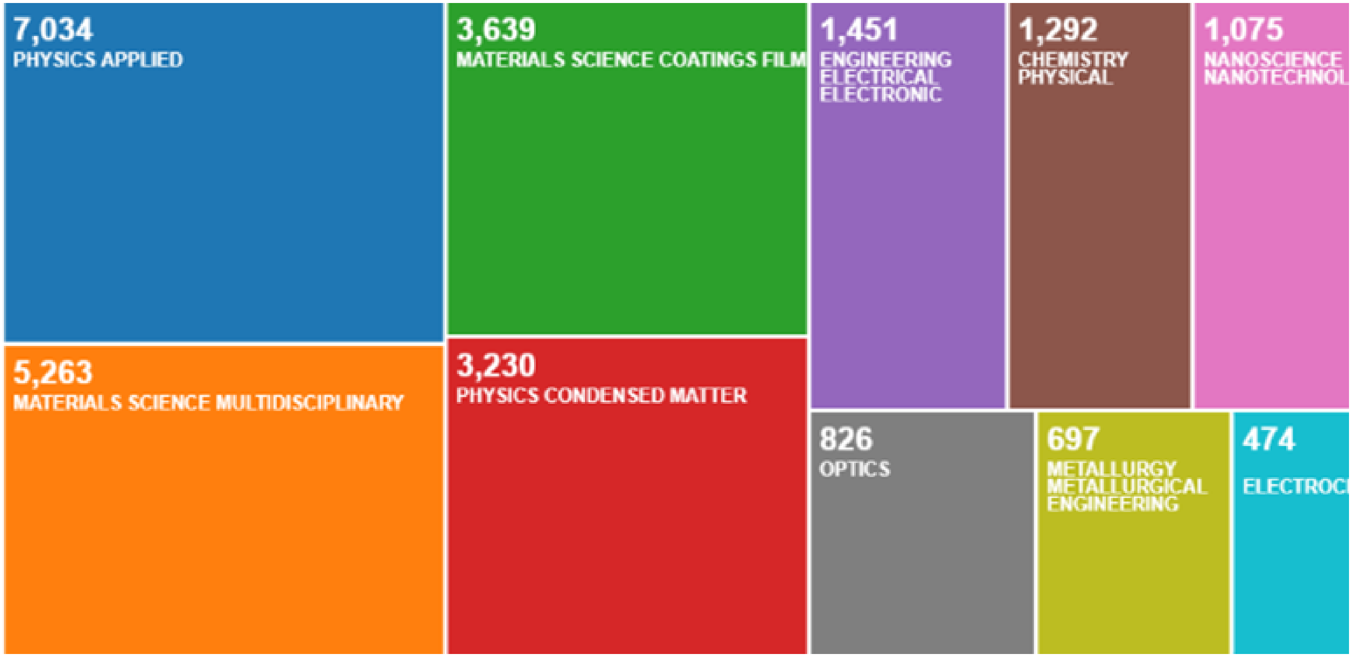


Scientific achievements

Transition metal oxynitride thin films

- Group of ceramic materials which are characterized by the possibility of changing the ratio between nitrogen and oxygen, which leads to a variety of properties.

- Oxynitride coatings based on transition metals (MeO_xN_y) can potentially benefit from the properties of the respective oxides or nitrides of the particular metal or can be characterized by entirely new properties, distinct from those of the oxides or nitrides.

Scientific achievements

Transition metal oxynitride thin films

- Thin layers based on **tantalum**, either oxide, nitride or oxynitride-type, are characterized by remarkable properties, useful in various fields, from microelectronics, to protective coatings, to optical coatings, to biocompatible coatings, etc.

- The main objective of the experimental work conducted after the completion of the Ph.D. program was to isolate and improve certain key configurations of samples from the **TaO_xN_y** class.

Scientific achievements

Transition metal oxynitride thin films

- The coatings were deposited by reactive magnetron sputtering, using a mixture of reactive gases whose composition remained constant (**O_2 + N_2 = 15% + 85%**), while varying the total flow of reactive gas mixture introduced into the deposition chamber.

- Several configurations of samples were obtained: grounded, biased with different voltages, and with different reactive mixture gas flows.

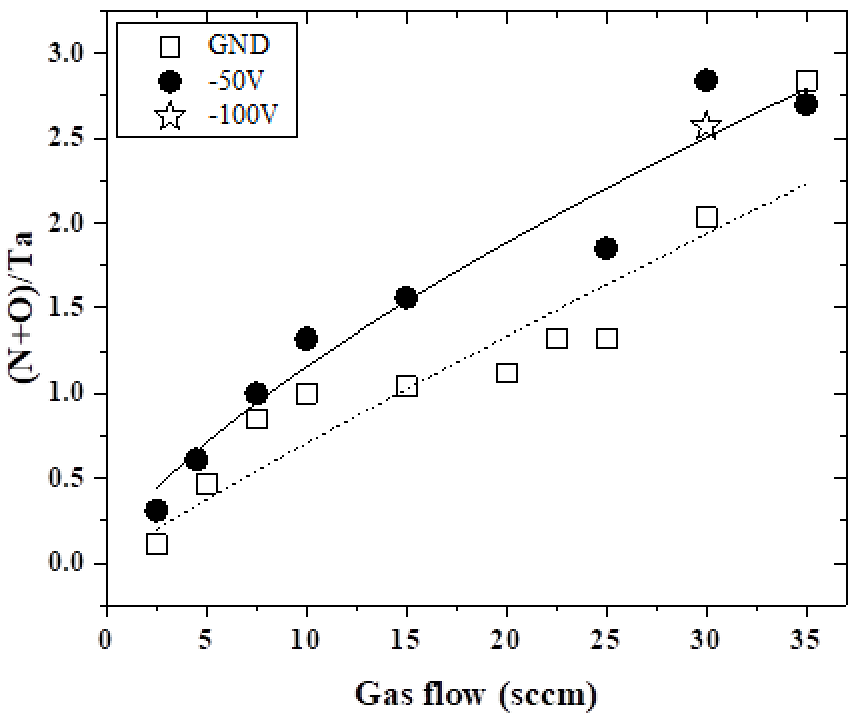
Scientific achievements

- Transition metal oxynitride thin films

Scientific achievements

Transition metal oxynitride thin films

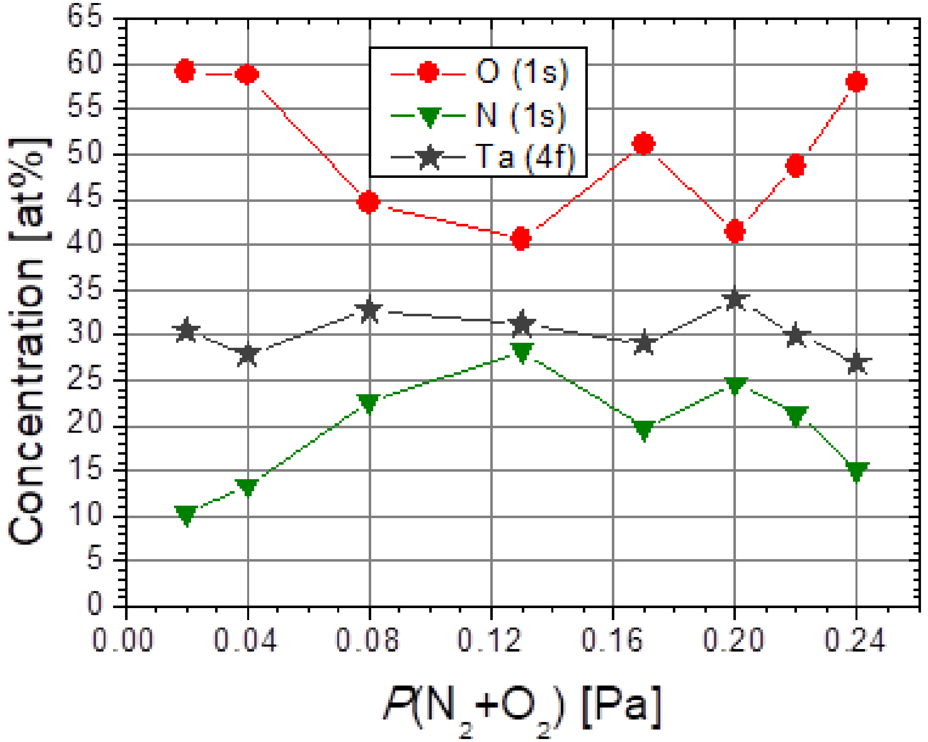
- The **chemical composition**, in atomic percentages, for the tantalum oxynitride samples was obtained by Rutherford Backscattering Spectrometry (RBS) .



Scientific achievements

Transition metal oxynitride thin films

- **X-ray Photoelectron Spectroscopy** (XPS) measurements were performed on selected silicon substrate samples (GND).



Scientific achievements

- <u>Transition metal oxynitride thin films</u>
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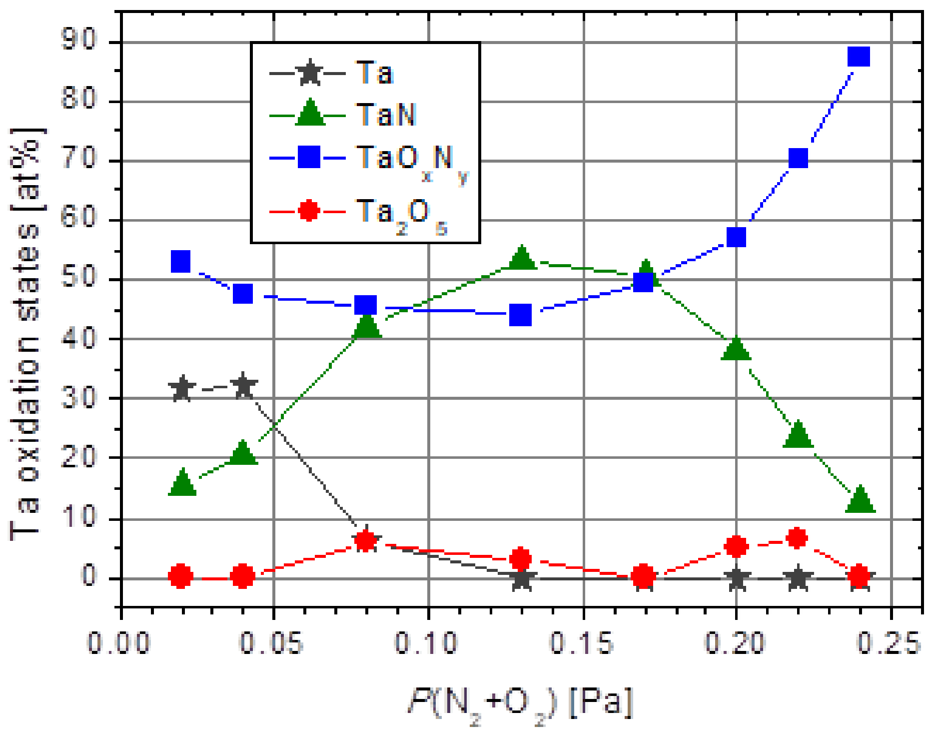
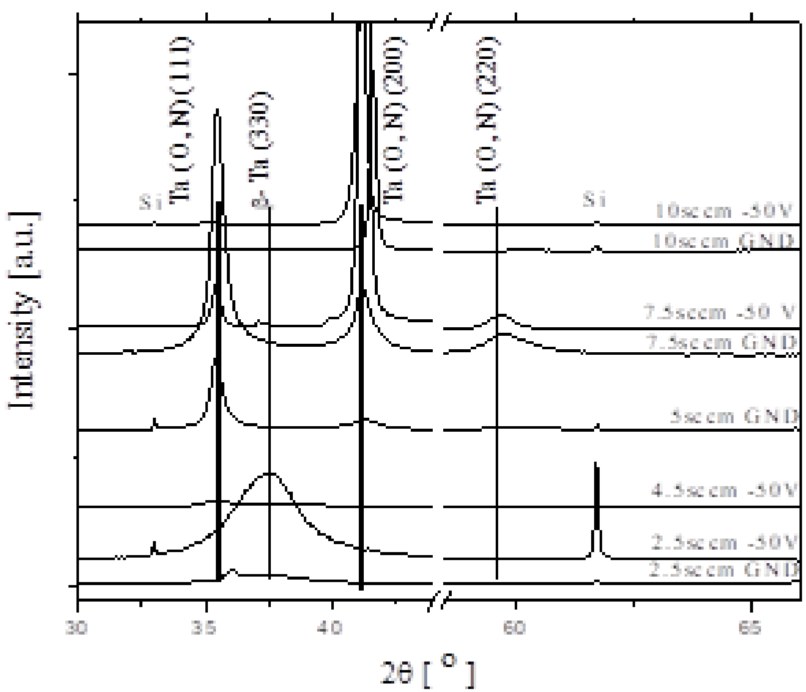
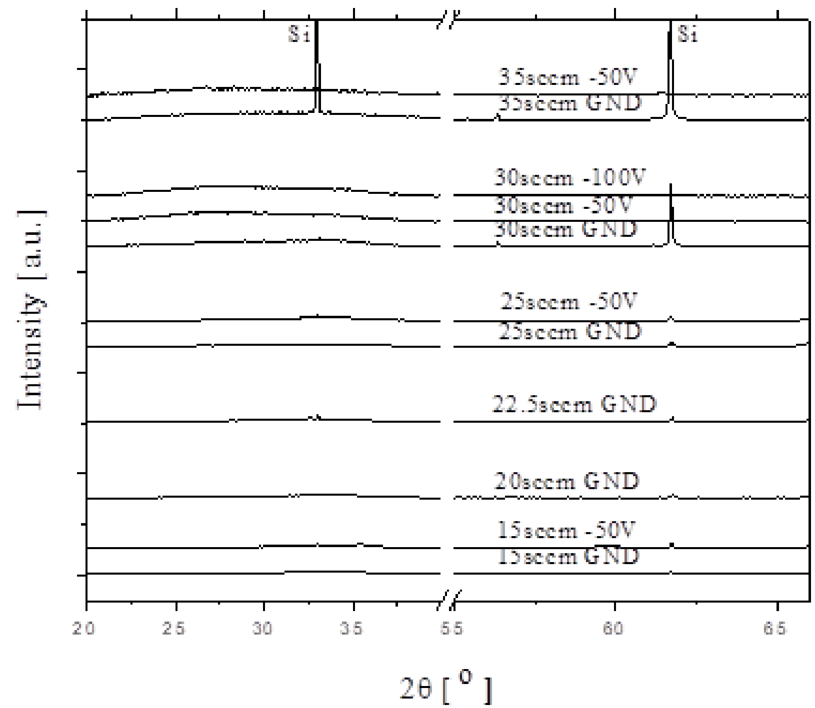


Scientific achievements

Transition metal oxynitride thin films

- **X-ray diffraction** (XRD) patterns were obtained for the samples deposited onto Si substrates.

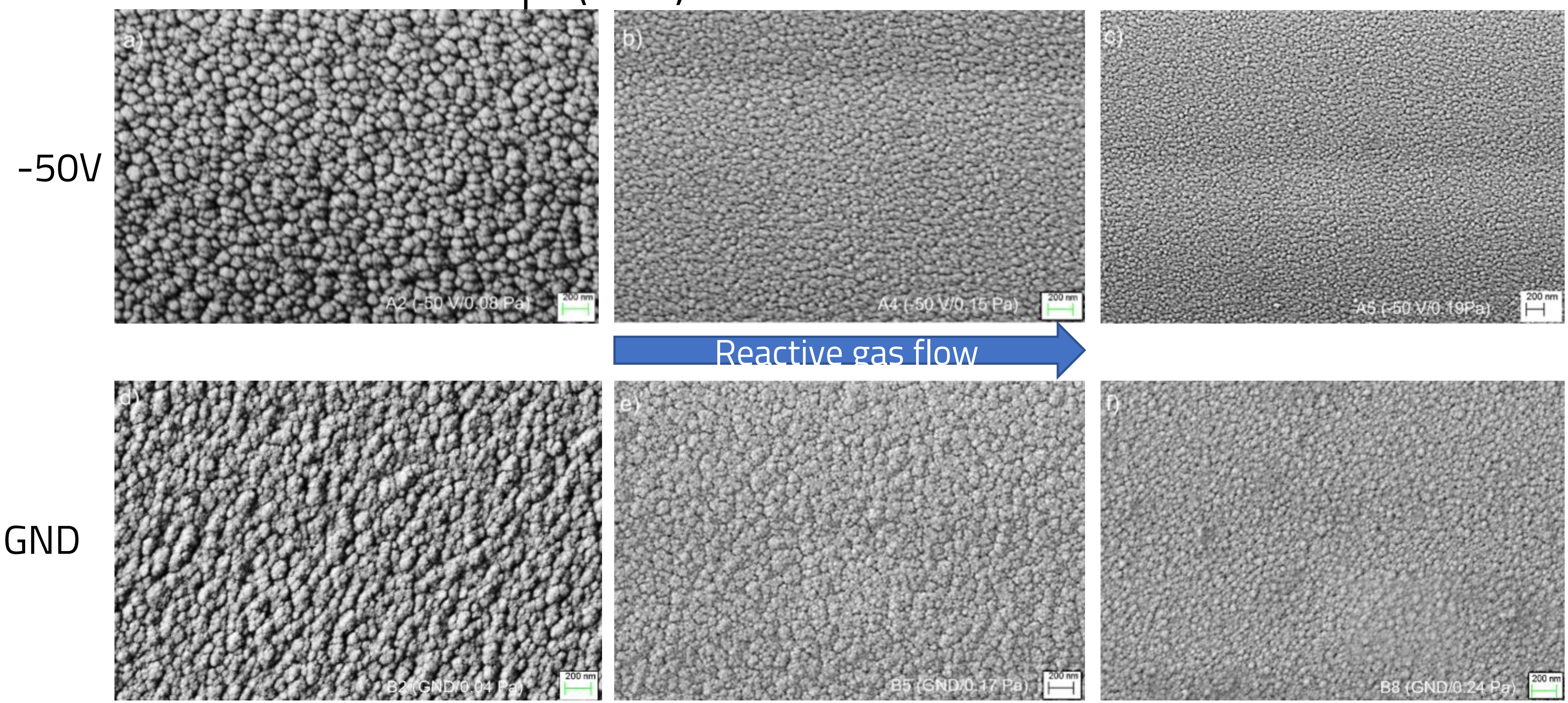




Scientific achievements

- <u>Transition metal oxynitride thin films</u>

  - The **morphology** of the tantalum oxynitride thin layers, both of the surface and in cross-section was studied using a scanning electron microscope (SEM).



Scientific achievements

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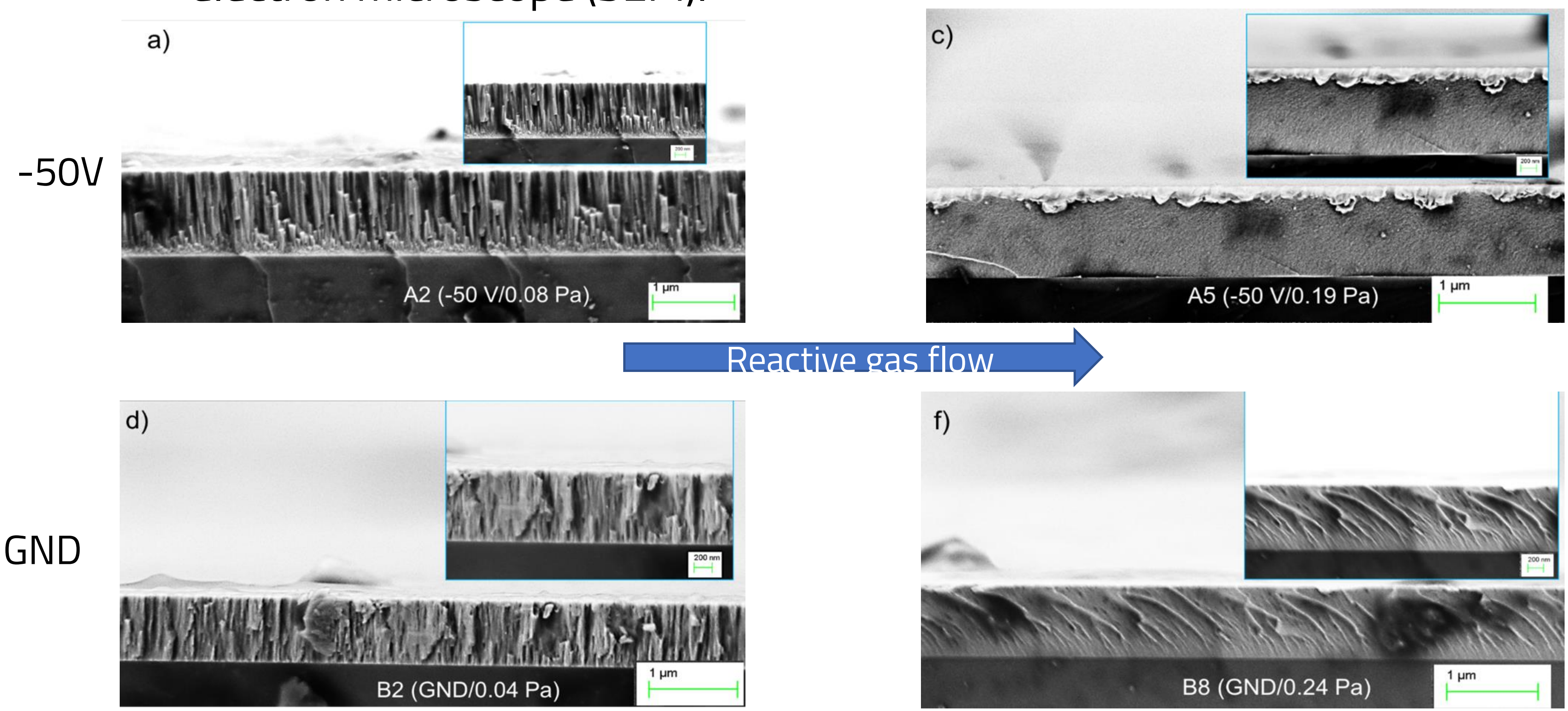
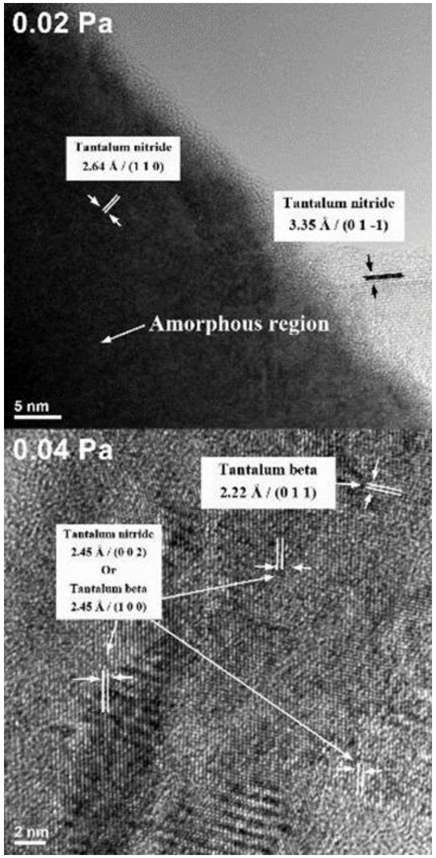


Scientific achievements

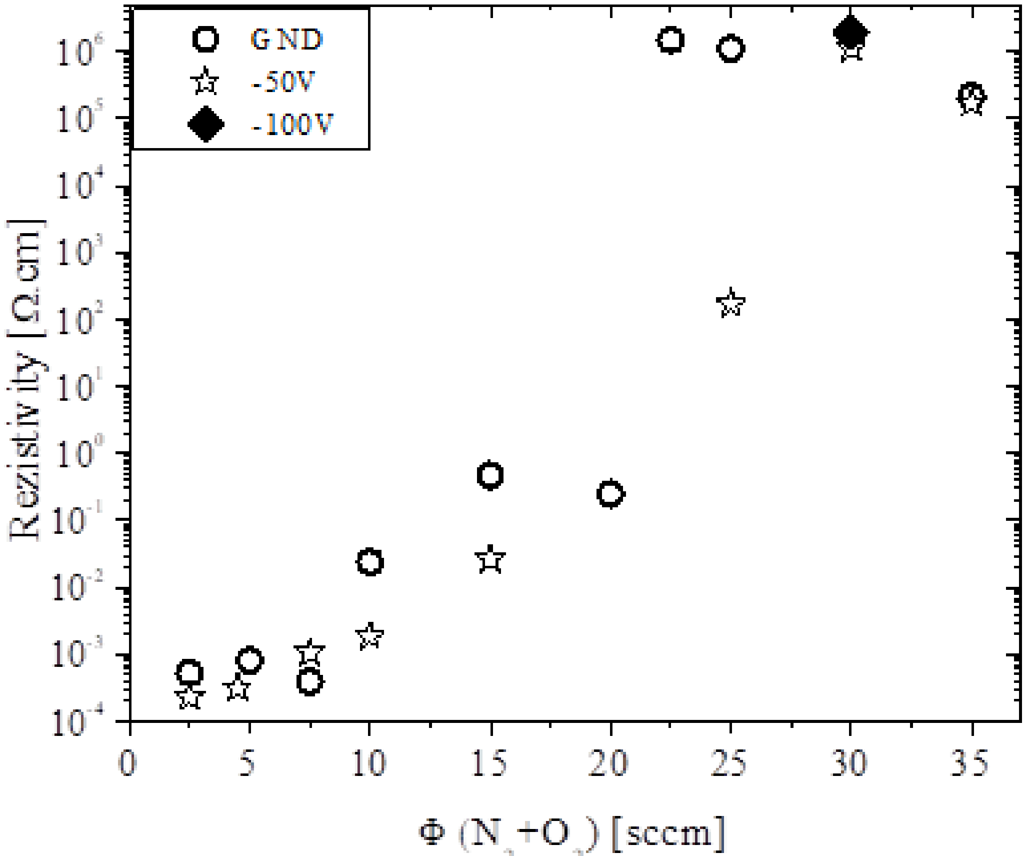
- <u>Transition metal oxynitride thin films</u>
  - **Transmission electron microscopy** (TEM) analyses were performed on selected samples (coatings deposited on silicon wafers)



Scientific achievements

Transition metal oxynitride thin films

- The **electrical resistance** was measured using the four-point probe and the two-point probe methods.

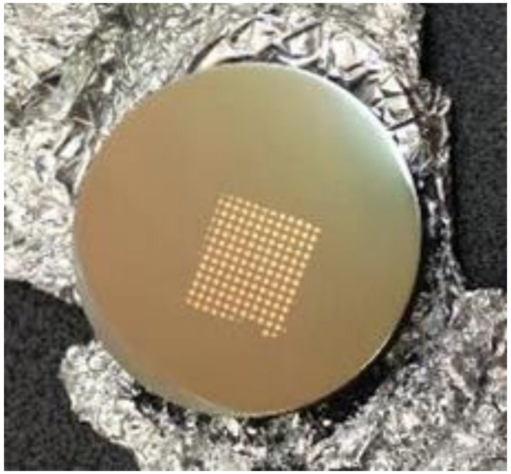


Scientific achievements

Transition metal oxynitride thin films

- **Dielectric measurements** were performed using an Agilent 4294A impedance analyzer, in the frequency range 100Hz - 5MHz

- The starting point for the measurement of the dielectric characteristics was to fabricate some MIM (metal-insulator-metal) type structures



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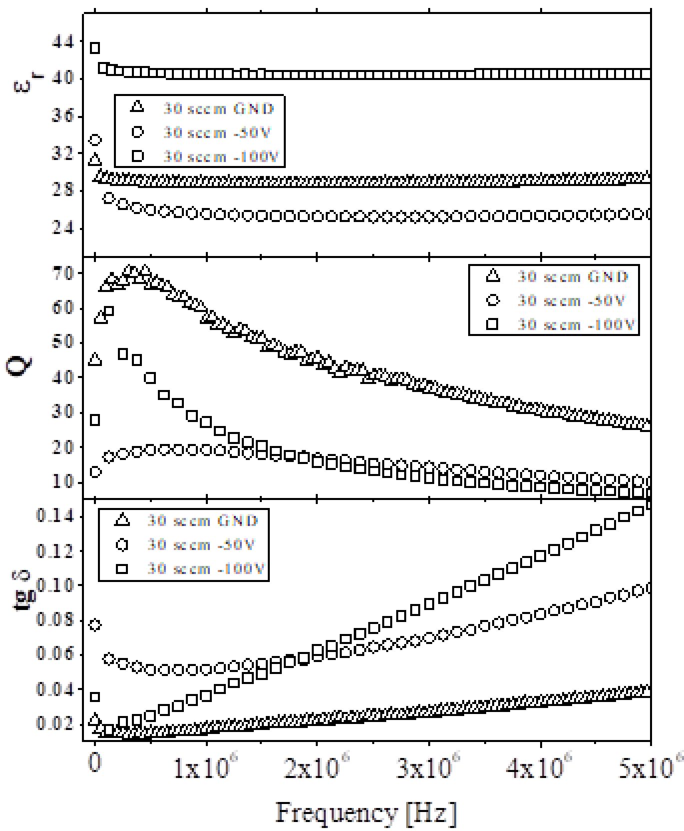
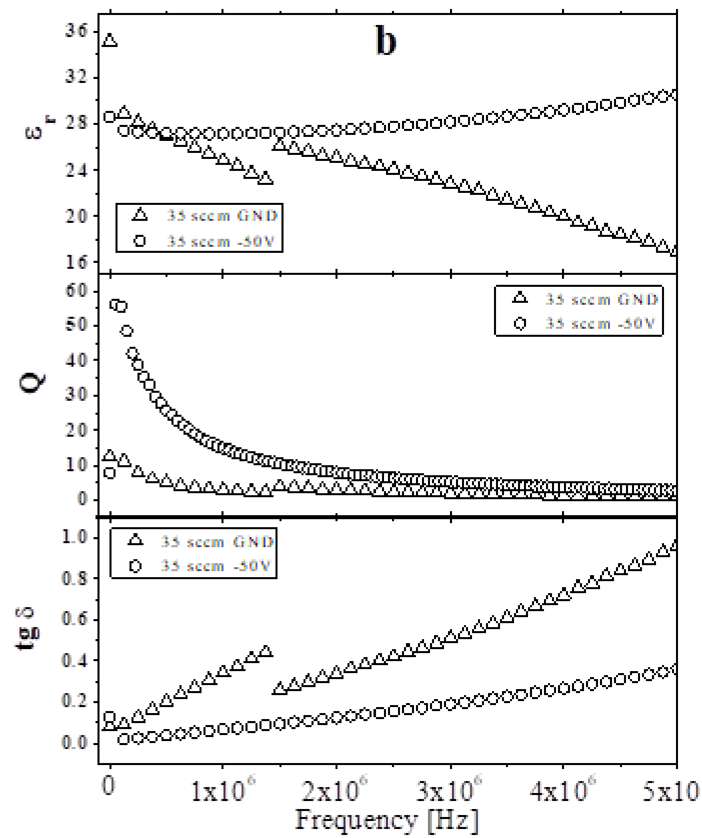
Scientific achievements

- Transition metal oxynitride thin films

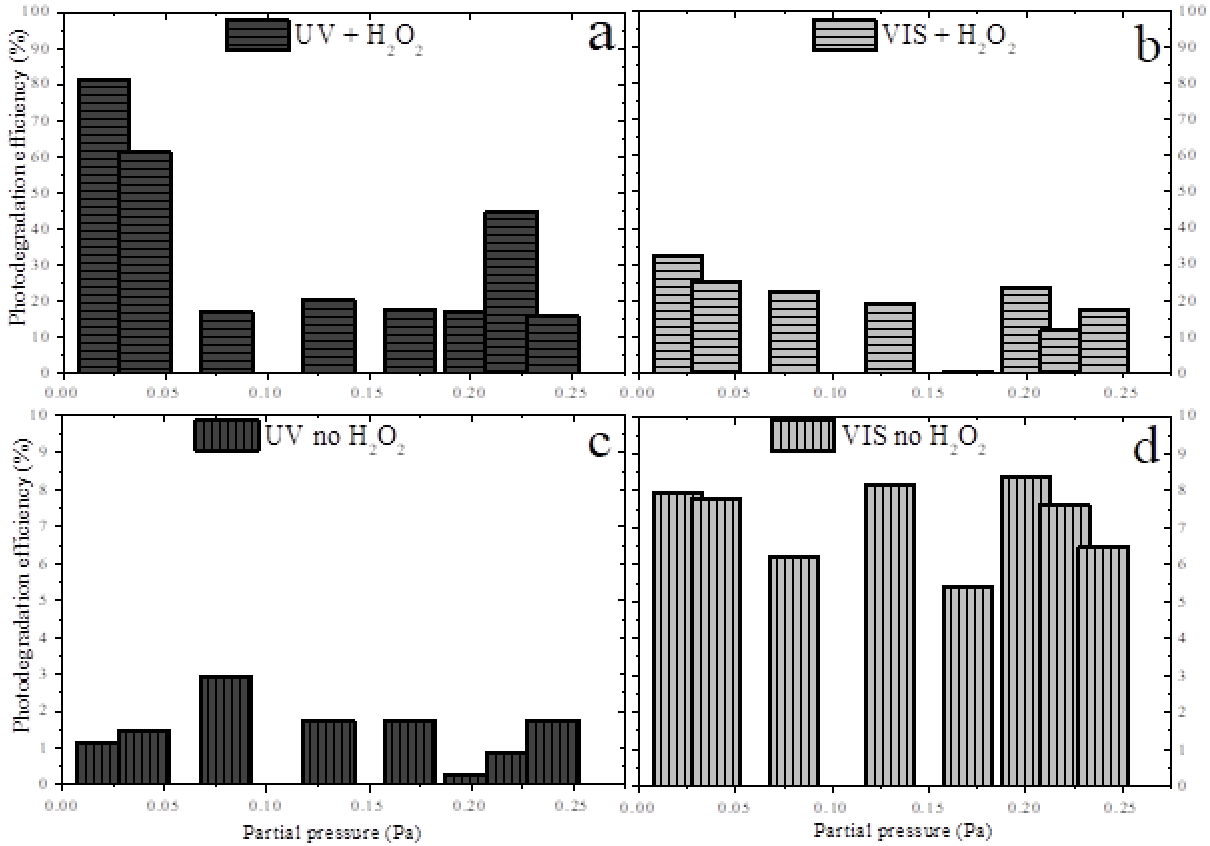




Scientific achievements

Transition metal oxynitride thin films

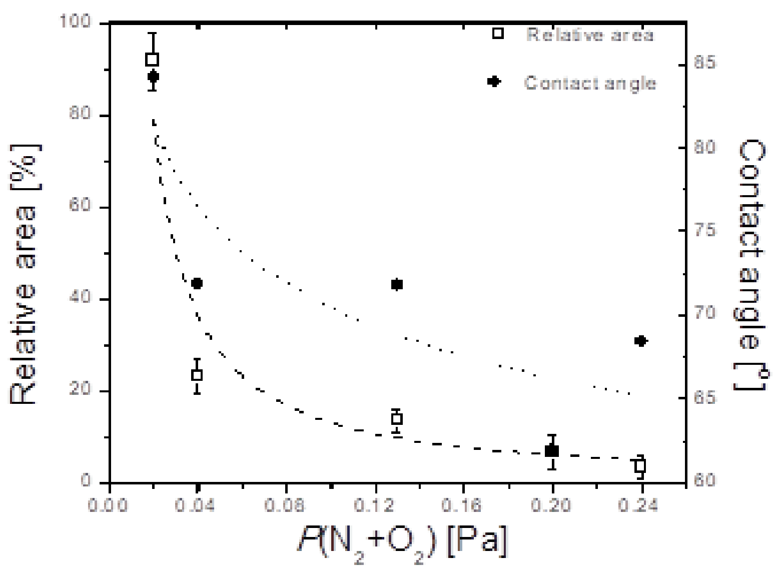
- The **photodegradation behavior** of the TaO_xN_y thin films was tested using methyl orange (MO) and methylene blue (MB)

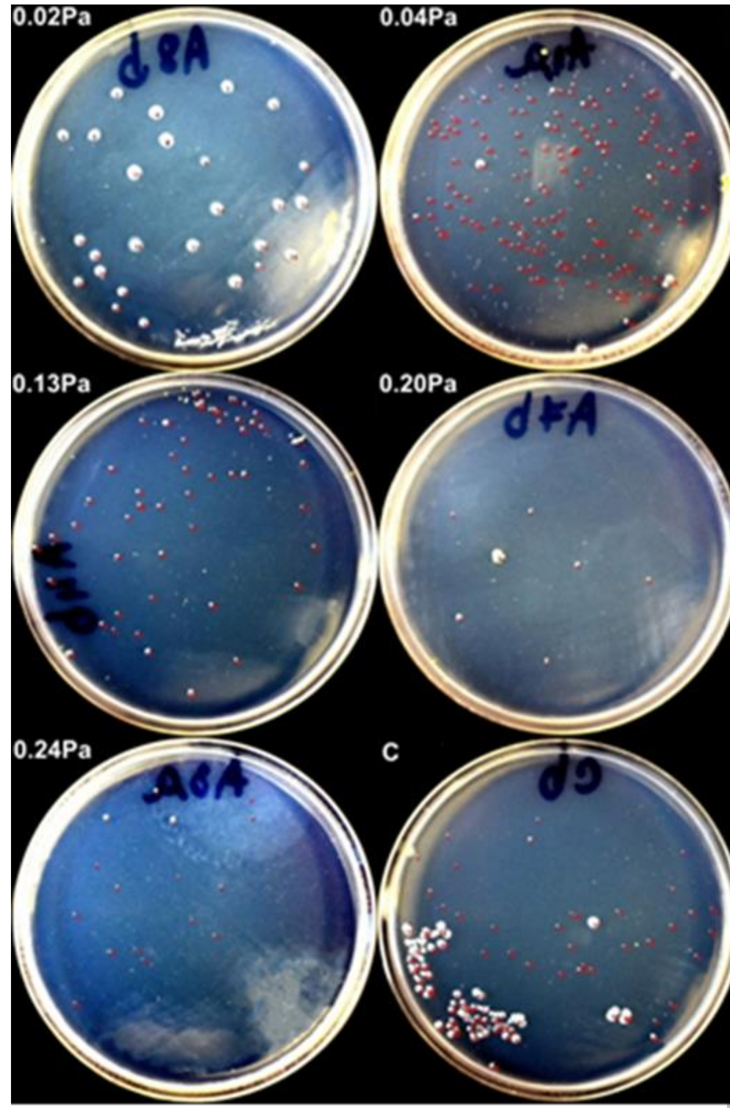


Scientific achievements

Transition metal oxynitride thin films

- The antibacterial/anti-biofilm capacity of the tantalum oxynitride coatings (deposited with a grounded substrate - GND) was assessed against *Salmonella*





Scientific achievements

Ceramic composite thin films

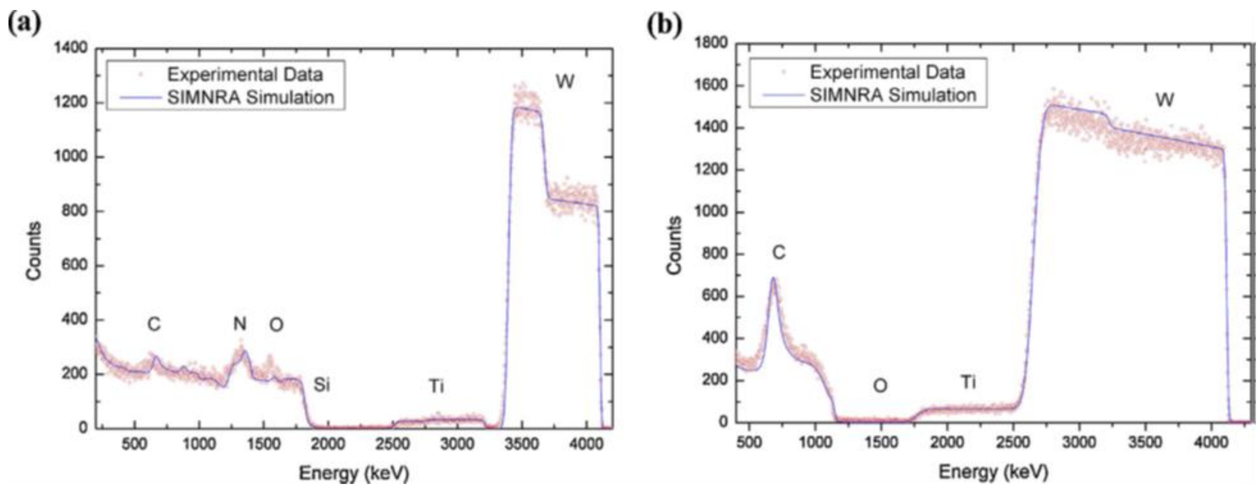
- Results concerning the composition, morphological and mechanical characteristics (wear, adhesion to the substrate, hardness) for ceramic composite magnetron sputtered (**Ti + TiB_2 + WC**) coatings. The films were obtained by simultaneous standard/reactive magnetron sputtering from three targets (Ti, TiB_2 and WC).

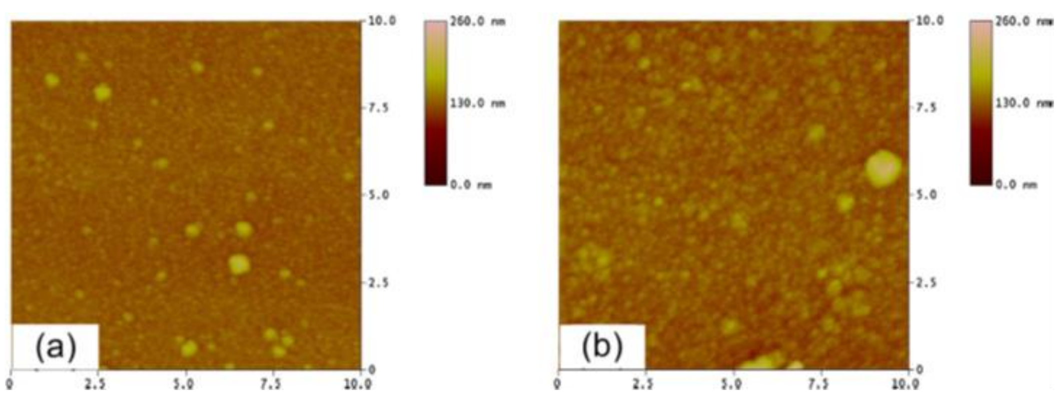
- **Nanocomposite coating** consists of at least two immiscible phases, of which at least one has at least one dimension in the nanoscale (a nanocrystalline phase which can act as a reinforcing agent, and an amorphous phase (which takes the role of the matrix phase) or two nanocrystalline phases (one as the matrix phase)).

Scientific achievements

- Ceramic composite thin films

$Ti + TiB_2 + WC + N_2$ $\qquad$ $Ti + TiB_2 + WC$





Scientific achievements

Ceramic composite thin films

$Ti + TiB_2 + WC +$ N_2

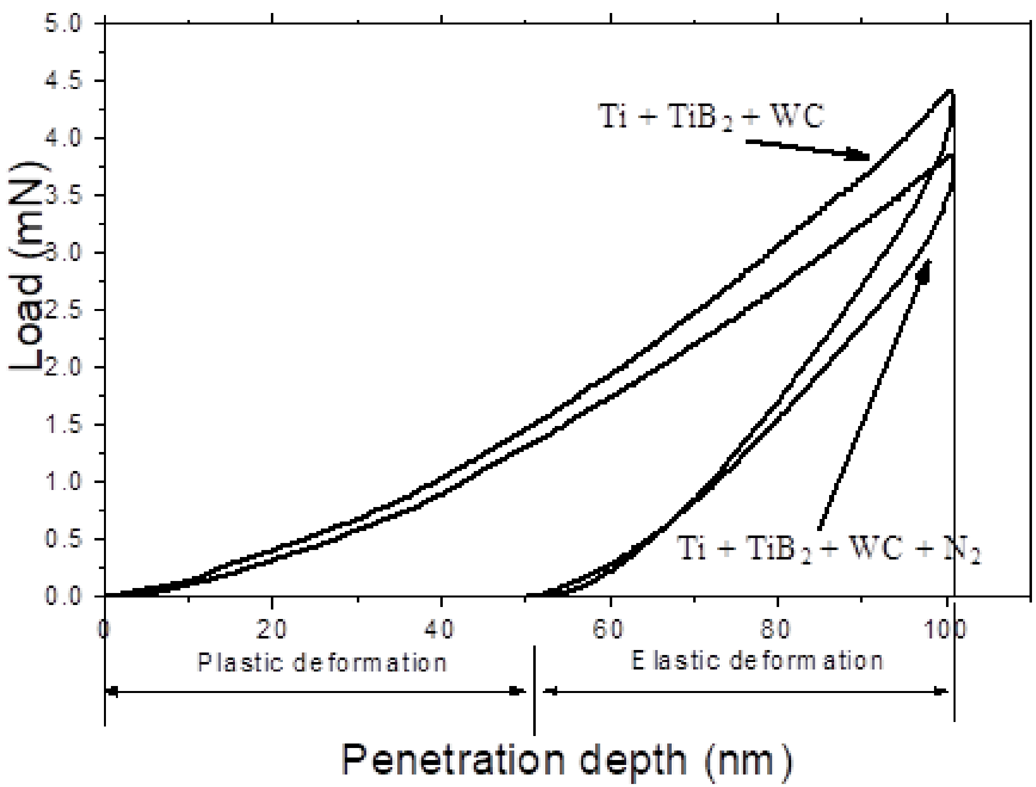
$Ti + TiB_2 + WC$

Sample	Nanoindentation				Adhesion			Wear
	H_{it} [GPa]	E_{it} [GPa]	H/E	H^3/E^2	Lc1 [N]	Lc2 [N]	Lc3 [N]	Friction coefficient
$Ti + TiB_2 + WC + N_2$	20.53 ± 9.18%	247,31 ± 5.60%	0.08	0.14	-	0.4 ± 12.5%	6.61 ± 5.95%	0.18 ± 27%
$Ti + TiB_2 + WC$	22.9 ± 9.67%	300,38 ± 7.44%	0.07	0.13	0.63 ± 3.72%	1.24 ± 3.23%	7.61 ± 4.6%	0.23 ± 6.78%
Substrate (steel)	3.43 ± 5.31%	232.24 ± 5.18%	-	-	-	-	-	-
Pin	4.62 ± 12.07%	209.36 ± 6.50%	-	-	-	-	-	-

Scientific achievements

- Ceramic composite thin films

$Ti + TiB_2 + WC + N_2$ $Ti + TiB_2 + WC$

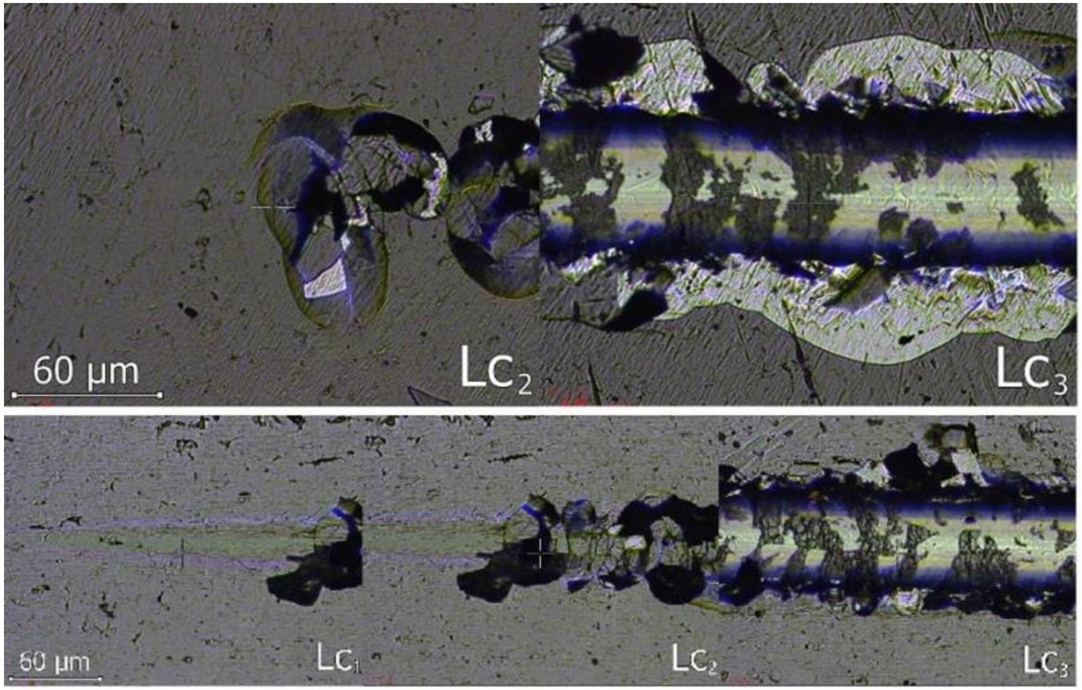


Scientific achievements

- Ceramic composite thin films

$Ti + TiB_2 + WC + N_2$

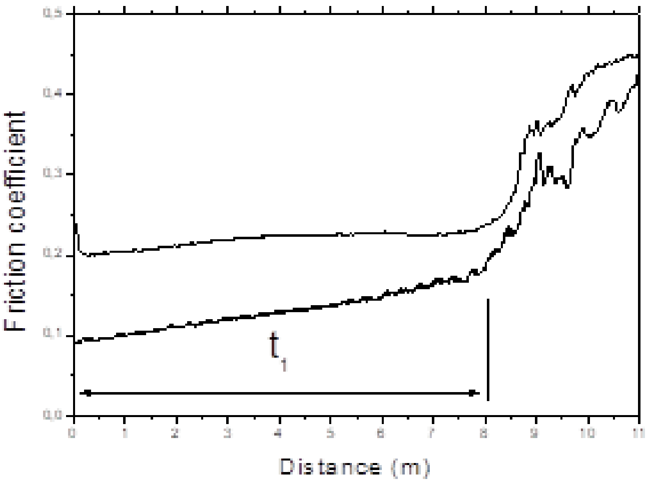
$Ti + TiB_2 + WC$



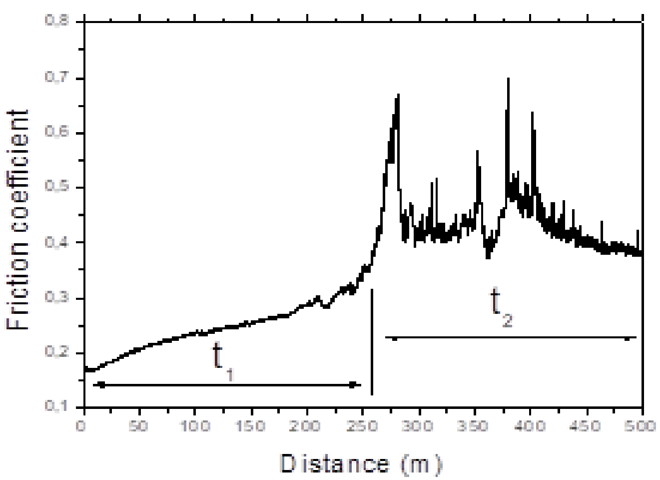
Scientific achievements

- Ceramic composite thin films

$Ti + TiB_2 + WC + N_2$



$Ti + TiB_2 + WC$



Scientific achievements

Magnesium-based ternary nitride thin films

- Improving the **corrosion resistance** of corrodible parts by applying proper surface coatings and surface modifications can lead to significant economical benefits.
- Coated materials like steel, brass, Al or Mg alloys, when exposed to a corrosive medium, suffer significant corrosive damage due to inherent **defects or inhomogeneities** in the thin film (cracks, pores, transient grain boundaries), even if the coatings have intrinsic corrosion resistance.
- To suppress or minimize the occurrence of **surface defects** which can lead to poor corrosion performance, several coating or surface treatment technologies can be employed sequentially = **duplex treatments**

Scientific achievements

Magnesium-based ternary nitride thin films

- Another promising approach in terms of corrosion protection of a less noble substrate is to consider the coating as a **sacrificial agent**, while trying to maintain as much as possible the mechanical characteristics (high hardness, high wear resistance, etc).

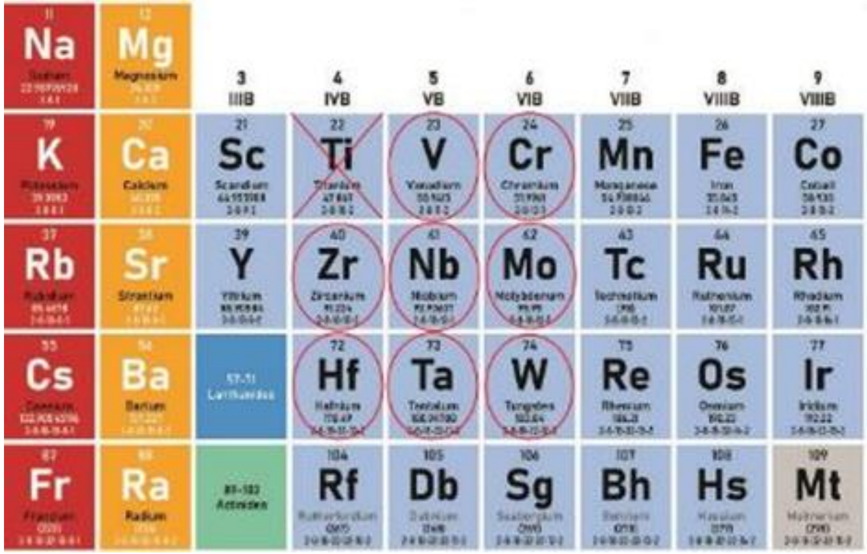
- **RM-Mg-N** (RM – refractory metal) multiple component nitride-type thin films, obtained by simultaneous sputtering of two metallic targets (high purity RM and Mg) with the addition in various proportions of reactive gas (N_2).

Scientific achievements

- <u>Magnesium-based ternary nitride thin films</u>

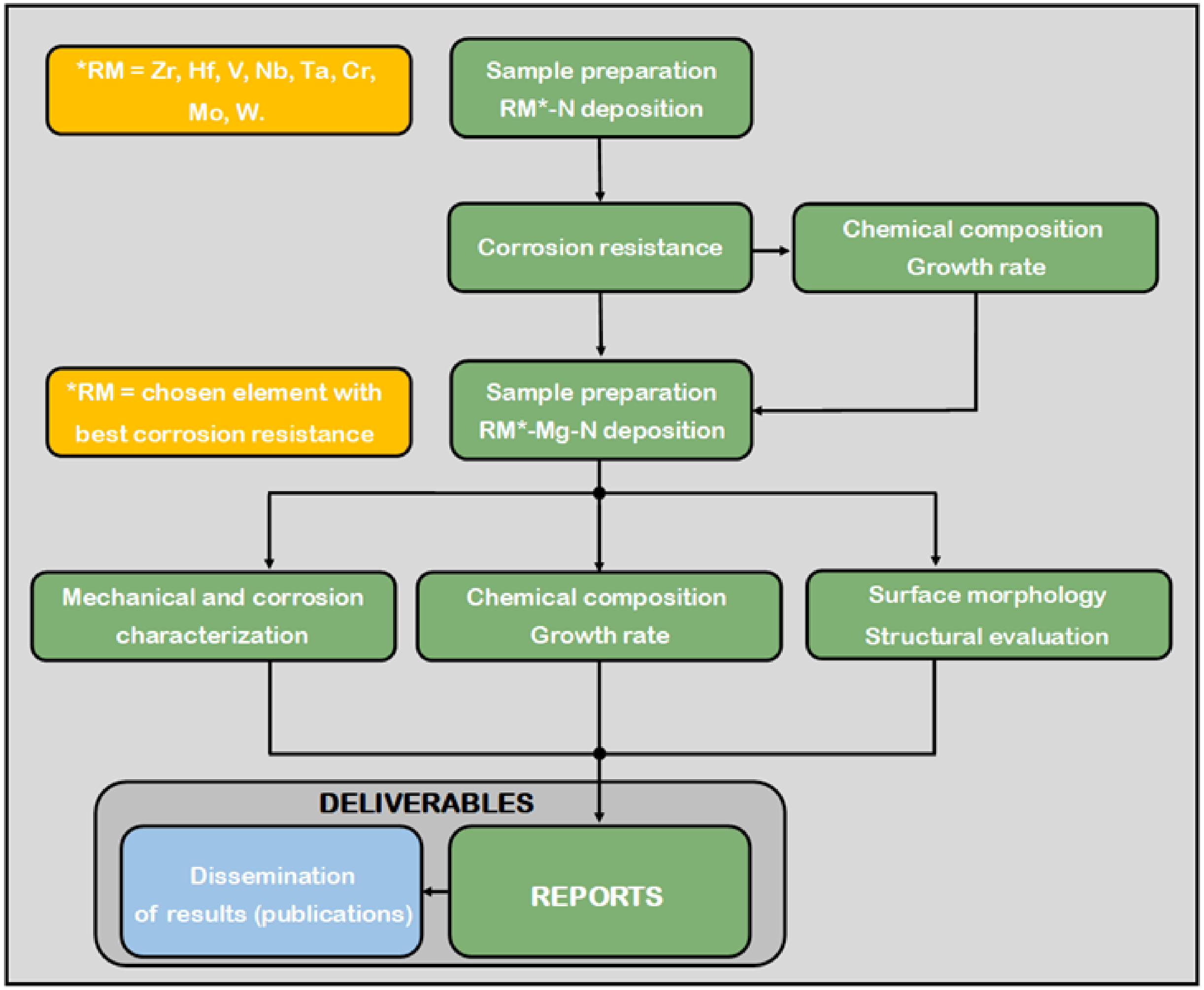
Phase I

Target	N2 flux	
material	medium	high
Zr	1	1
Hf	1	1
V	1	1
Nb	1	1
Ta	1	1
Cr	1	1
Mo	1	1
W	1	1
Total number of depositions:		**16**



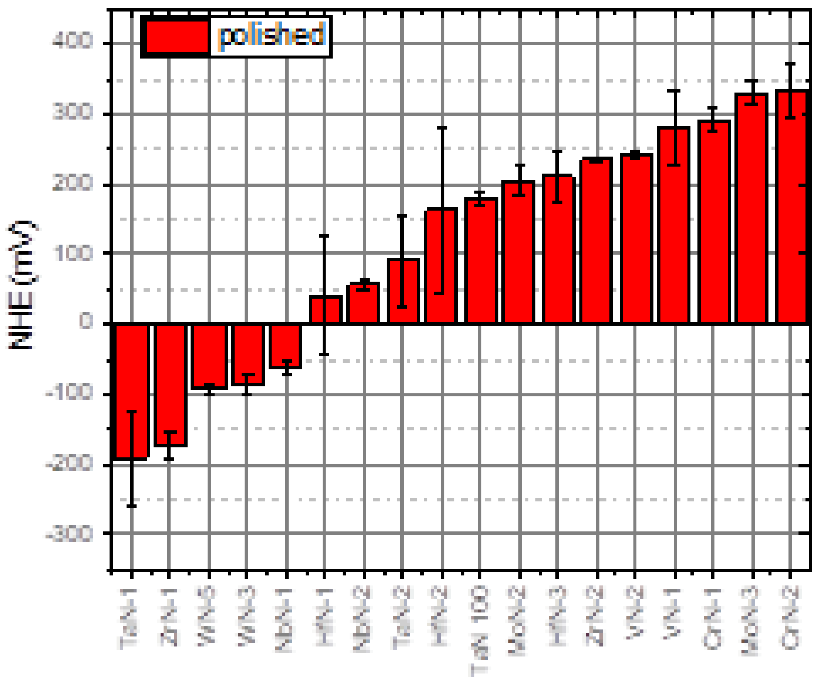
Scientific achievements

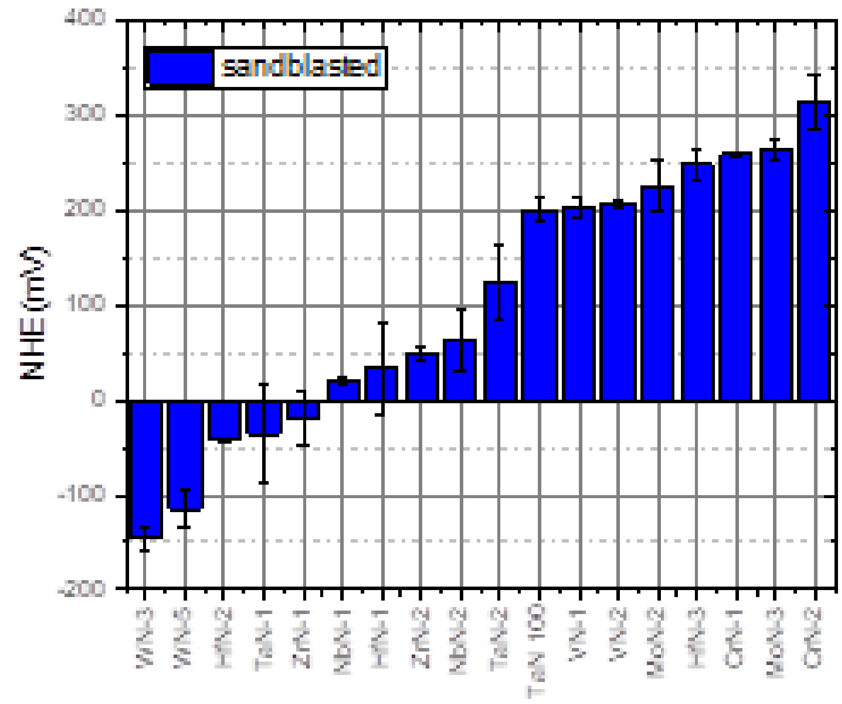
- Magnesium-based ternary nitride thin films



Scientific achievements

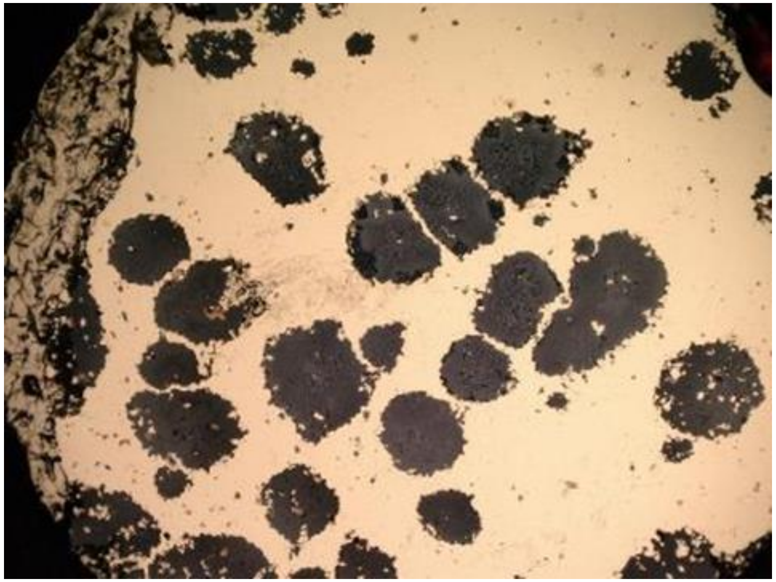
- Magnesium-based ternary nitride thin films



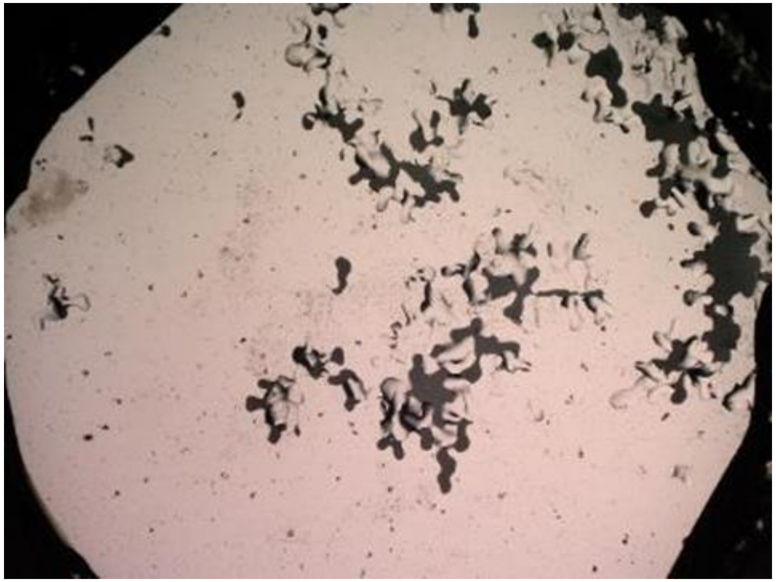


Scientific achievements

- Magnesium-based ternary nitride thin films



ZrN



TaN

Scientific achievements

Magnesium-based ternary nitride thin films

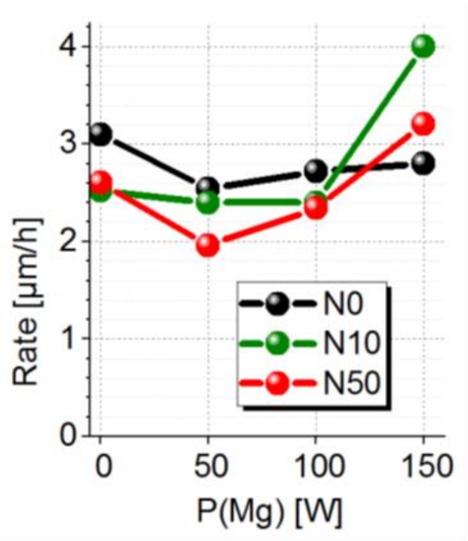
Phase II

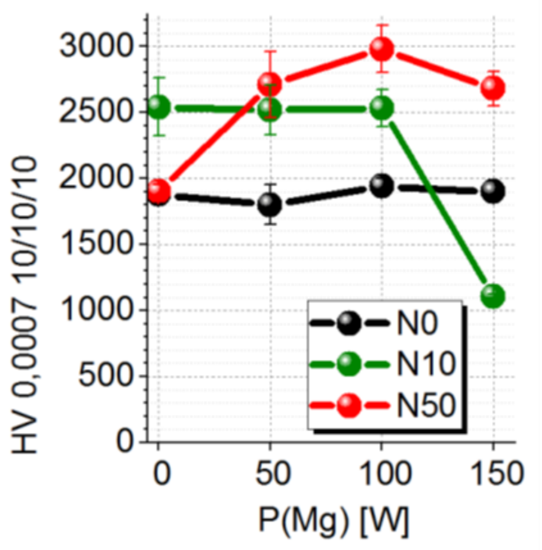
Sample	N_2 flow [sccm]	Mg power [w]	Thickness [µm]	Hardness HV
W	0	0	1.6	1880±70
WMg50	0	50	1.3	1800±15
WMg100	0	100	1.3	1940±60
WMg150	0	150	1.4	1840±60
WN10	10	0	1.3	2540±220
WN10Mg50	10	50	1.2	2520±200
WN10Mg100	10	100	1.2	2530±140
WN10Mg150	10	150	2.0	1110±12
WN50	50	0	1.2	1900±50
WN50Mg50	50	50	1.0	2700±250
WN50Mg100	50	100	1.2	3000±200
WN50Mg150	50	150	1.6	2700±130

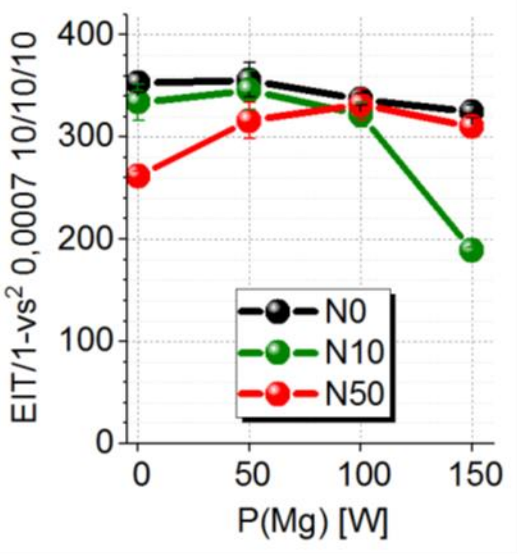
Scientific achievements

- Magnesium-based ternary nitride thin films

Phase II



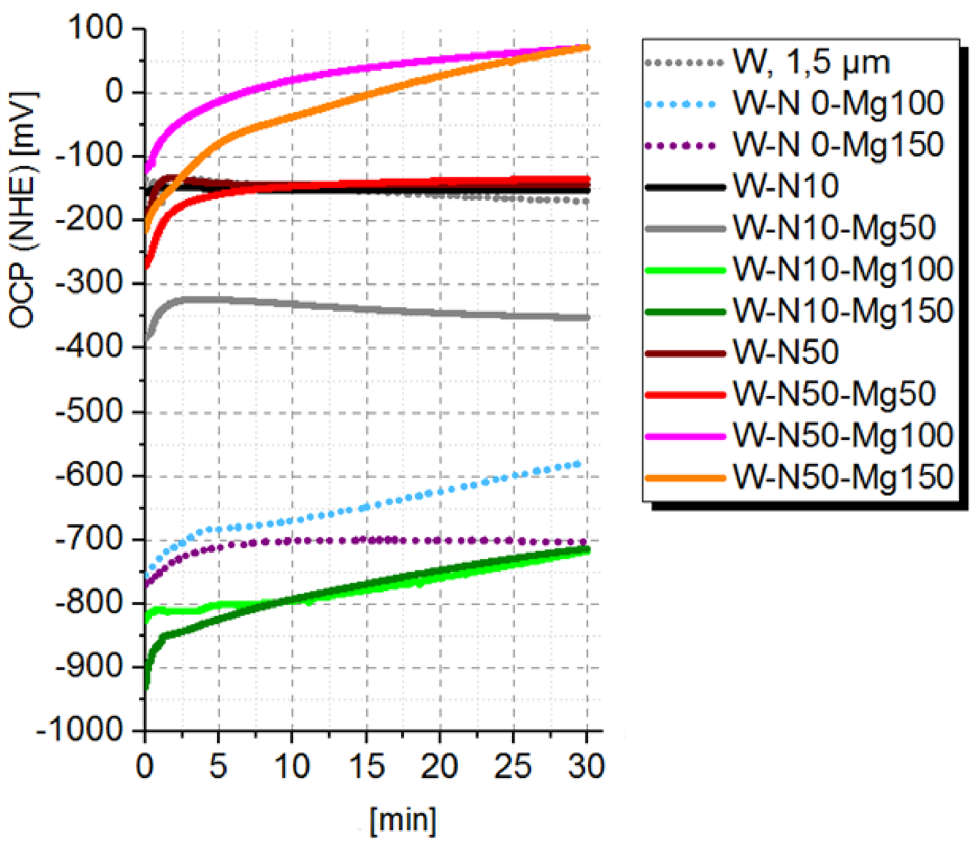




Scientific achievements

- Magnesium-based ternary nitride thin films

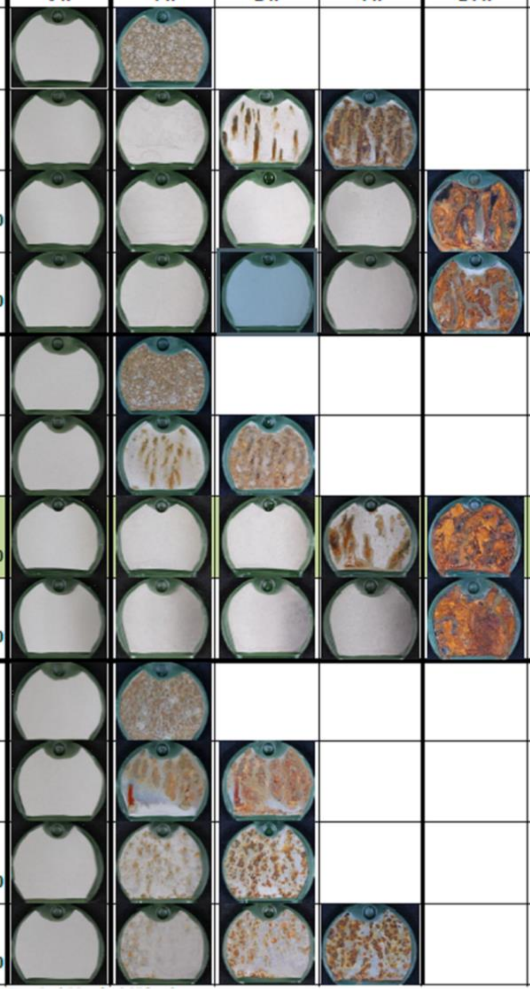
Phase II



Scientific achievements

- Magnesium-based ternary nitride thin films

Phase II

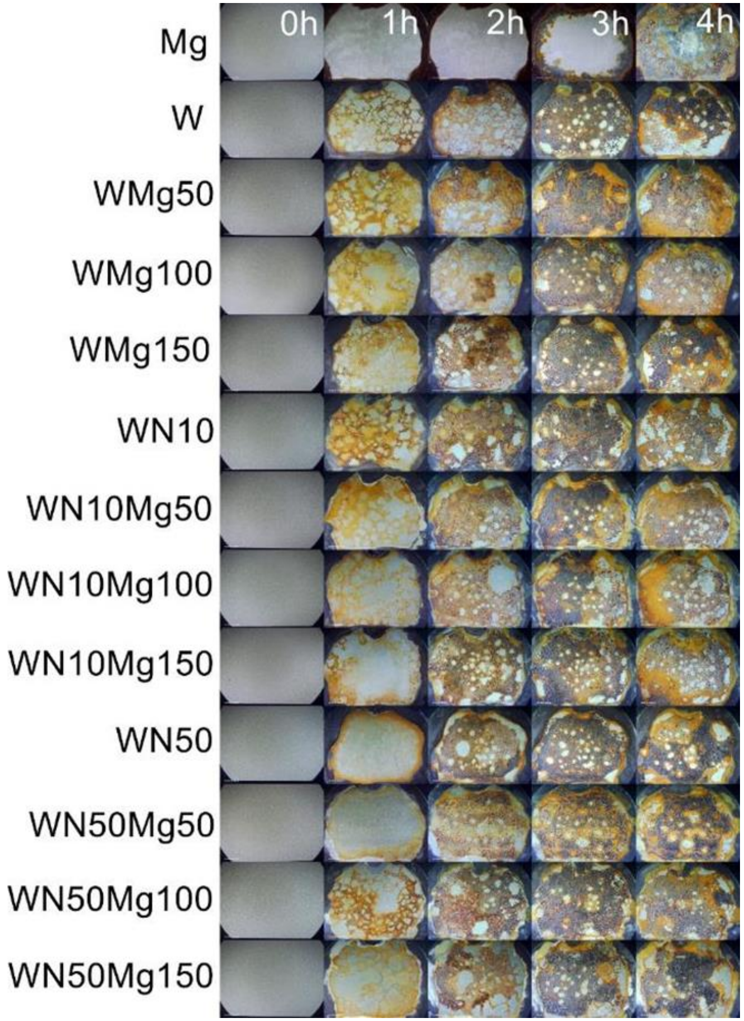


		0 h	1 h	2 h	4 h	24 h	48 h	
L200 225a	W							HV1880 ±70 1,6 µm
L200 224a	W Mg50							1800 ± 150 1,3 µm
L200 224b	W Mg100							1940 ± 60 1,3 µm
L200 224c	W Mg150							1840 ± 60 1,4 µm
L200 214a	W N10							2540 ± 220 1,3 µm
L200 214b	W N10 Mg50							2520 ± 200 1,2 µm
L200 217a	W N10 Mg100							2530 ± 140 1,2 µm
L200 218a	W N10 Mg150							1110 ± 12 2 µm
L200 213b	W N50							1900 ± 50 1,2 µm
L200 214c	W N50 Mg50							2700 ± 250 1 µm
L200 217b	W N50 Mg100							3000 ± 200 1,2 µm
L200 218b	W N50 Mg150							2700 ± 130 1,6 µm

Scientific achievements

- Magnesium-based ternary nitride thin films

Phase II

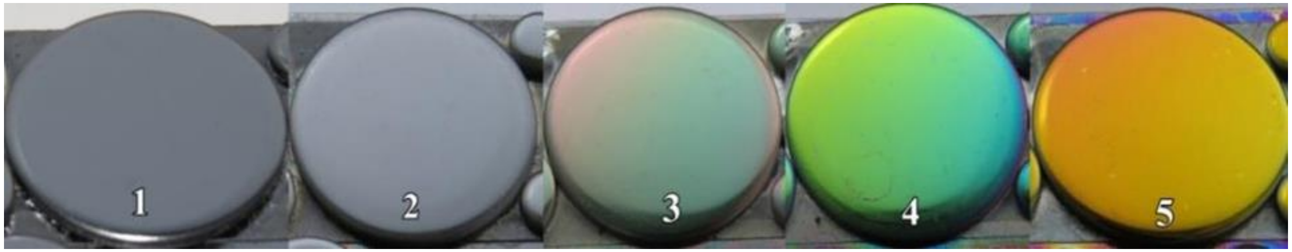


The evolution and development plans for career development

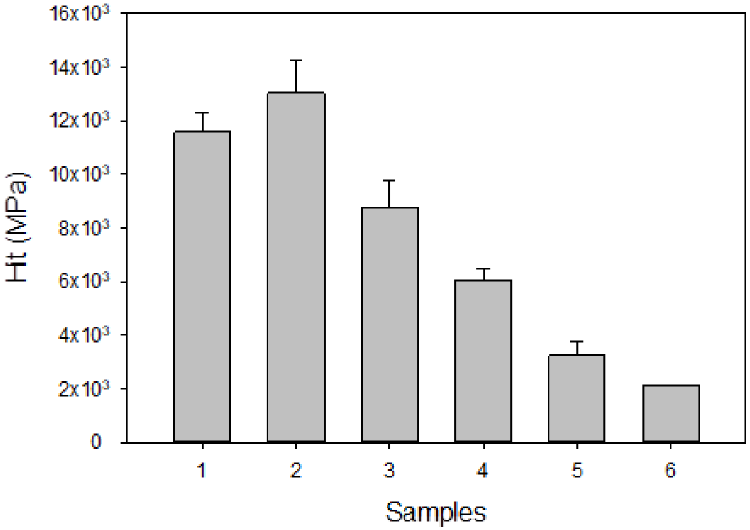
- Teaching activities
  - I intend to publish a specialized book on Surface Engineering and a guide of applications on materials analysis techniques
  - I will maintain the novelty of the courses by adapting them every year to the new discoveries that appear in the field, by continuously studying the specialized literature
  - the development of new didactic laboratory works for fundamental and applied research, and to involve the bachelor's and master's students in practical activities
  - attract possible candidates for the PhD program, and build a research team which will be able to attract funding through project proposals

The evolution and development plans for career development

- Research topics
  - **Complex oxynitride coatings** ($Me_1Me_2O_xN_y$, where Me_1, Me_2 = Ti, Al, Sr, Nb, Mg, Mo, etc.) have received attention in recent years due to their original properties compared to their oxide or nitride parents.



1) Ta target = 1A, Ti target = 0A (TaO_xN_y film);
2) Ta target = 0.75A, Ti target = 0.25A ($TaTiO_xN_y$ film);
3) Ta target = 0.5A, Ti target = 0.5A ($TaTiO_xN_y$ film);
4) Ta target = 0.25A, Ti target = 0.75A ($TiTaO_xN_y$ film);
5) Ta target = 0A, Ti target = 1A (TiO_xN_y film)



The evolution and development plans for career development

- Research topics

  - **Refractory metal and magnesium-based coatings:** Chemical composition, surface morphology, structural evaluation, mechanical characterization on the WMgN samples, plus other variations based on transitional metals: NbMgN, CrMgN, etc.

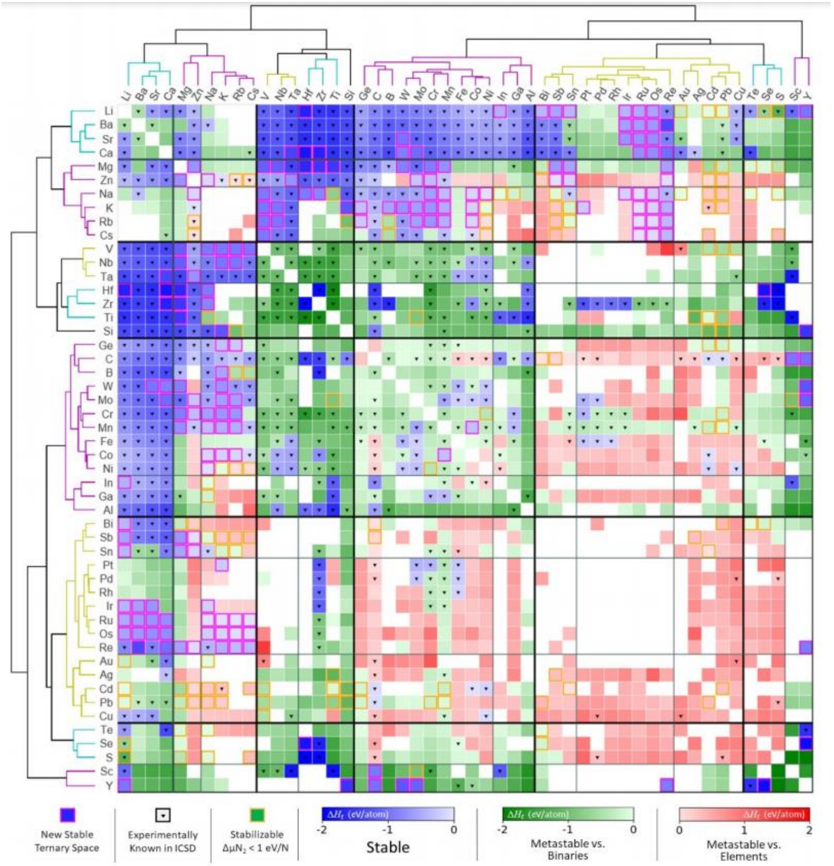
The evolution and development plans for career development

- <u>Research topics</u>
  - **Refractory metal ternary nitrides (Me_1Me_2N).** The multitude of possible applications for A-B-N-type materials stems from the numerous types of structures and chemical compositions that can arise, controllable through the processing parameters (deposition parameters, thermal annealing, etc.)

  - After a thorough examination of the available literature concerning these types of compounds (Me_1Me_2N), results concerning certain configurations were observed to be missing from the literature (e.g., coatings based on **hafnium**, most likely due to the material's cost).

The evolution and development plans for career development

- <u>Research topics</u>
  - Refractory metal ternary nitrides (Me_1Me_2N)



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  - **National Institute of Materials Physics**, Romania
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  - **Research Institute for Precious Metals and Metal Chemistry**, Germany
  - **Horia Hulubei National Institute for Physics and Nuclear Engineering**, Romania
  - **The Institute for Nuclear Research Pitesti**, Romania
  - **The National Institute for Research and Development in Electrical Engineering** ICPE-CA Romania
  - **The National Institute of Research and Development for Technical Physics**, Romania

Thank you for your attention!