RSD 2024

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About

The RSD conference

The International Conference on Reactive Sputter Deposition was established in Ghent in the year 2000. It provides a platform among leading international scientists, engineers and students to discuss recent achievements in reactive sputter deposition and thin films. The Conference has developed to an annual tradition, steadily growing without loosing its focus on reactive sputtering and its fundamental aspects. The symposium covers the production and use of coatings, from basic research to devices and new applications.

DRAFT

RSD2024 is organized by the research group DRAFT. Our mission statement of the research group DRAFT (Dedicated Research on Advanced Films and Targets) reads "At DRAFT we want to become the recognized leader in the understanding of thin film growth by reactive magnetron sputtering and to enjoy research by experiments and simulations." More information on this research group can be found on www.draft.ugent.be

Code of Conduct

By registering for and attending RSD2024 we all agree to conduct ourselves in a professional manner.

We will respect everyone regardless of their age, race, religion, ethnicity, sexual orientation, gender identity, gender expression, marital status, nationality, political affiliation, health, caring responsibilities, physical appearance, disability or educational background. Furthermore, we will also avoid language and behavior which reinforces discrimination or repression based on any of these attributes.

We will refrain from any form of discrimination, harassment or retaliation. This can include intimidating behavior or language, inappropriate jokes or comments, unwanted attention, stalking or the display of offensive images.

If you feel unhappy or uncomfortable with the conduct of others at RSD2024, please contact any of the organizers. Your comments will be taken seriously and acted upon in confidence. If you do not treat others with the respect and tolerance that we all deserve, you may be asked to leave.

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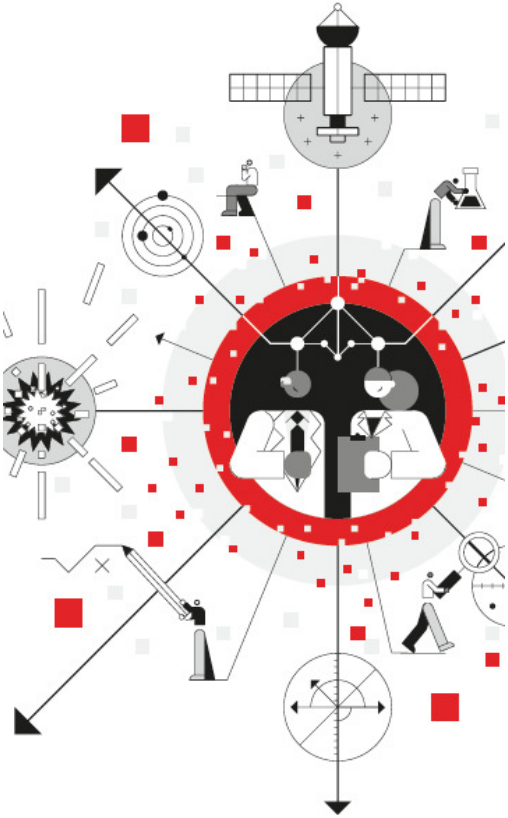


Our venue

Saint Peter's Abbey (Dutch: *Sint-Pietersabdij*) is a former Benedictine abbey in Ghent, Belgium, now a museum and exhibition center. History

Saint Peter's was founded in the late 7th century by Amandus, a missionary sent by the Frankish kings to Christianize the pagan inhabitants of the region, who founded two monasteries in the area, St. Bavo's, and Saint Peter's on the Blandijnberg. During the winter of 879-80, the abbey was raided and plundered by the Normans, and it remained relatively poor until the 10th century, when donations of property and relics by Count Arnulf I considerably enriched it, as did further donations by Elthruda, the niece of King Alfred, who donated in 918, St. Mary's Church in Lewisham, Greenwich and by Arnulf's cousin King Edgar of England. By the second half of the century it was the wealthiest abbey in Flanders, and the reputation of the abbey school extended far beyond the town.

In 984, Gerbert of Aurillac, director of the cathedral school of Reims, (later Pope Sylvester II) inquired whether students from Reims could be admitted to Saint Peter's, and its renown as a center of artes liberales continued into the 11th century. Saint Peter's, through its ownership of large tracts of land, also played a pioneering role in cultivation during the twelfth and thirteenth centuries, transforming forests, moors and marshes into farmland. In the fifteenth century a large scale program of construction created the abbey library and scriptorium, enlarged the refectory, and the abbey church and other buildings were considerably beautified.

Saint Peter's first decline began following the Revolt of Ghent in 1539, and by the 1560s the Low Countries were plunged in a religious crisis that resulted in an attack by iconoclasts in 1566 in which the abbey church was wrecked, the library looted, and

other buildings badly damaged. The infirmary was pressed into service as a temporary home for the monks and the refectory used as a place of worship. However opposition continued and in 1578 the abbot and monks were forced to flee to Douai. The abbey buildings were sold at public auction and were partly demolished, the materials being used to construct the city walls. The abbey finally came back into the hands of the church in 1584, and it was eventually rebuilt, with a new abbey church, begun in 1629, in the Baroque style, as well as several other new builds and refurbishments. During the 18th century, the abbey was once again flourishing, as new buildings were constructed and older ones enlarged, including the conversion of the old dormitory into a library with more than ten thousand books.

However, the end was not far off, first with the Brabant Revolution of 1789–90, then the French invasion of 1793. Finally, on 1 September 1796, the Directory abolished all religious institutions. In 1798 the library was emptied and eventually taken to Ghent University. From 1798 the abbey church was used as a museum, but was returned to the ownership of the church in 1801 and renamed Onze-Lieve-Vrouw-Sint-Pieterskerk ("Our Lady of Saint Peter's Church"). In 1810, the rest of the abbey became the property of the city of Ghent, and was partially demolished for the construction of a military barracks, which remained on the site until 1948. Around 1950 the city launched a program of restoration, which is still ongoing, which began with the cloister and chapter house, then the west wing, including the old refectory and kitchens. Work on the wine cellars and attics was completed in the 1970s, and in 1982 work on the abbey gardens was completed, and in 1986 the terrace. In the 1990s restoration of the refectory wing began.

The abbey is now used as a museum and exhibition centre, which in 2000 housed a major exhibition as part of the Year of Emperor Charles, and in October 2001 hosted the 88th meeting of the European Council[1].

[1]Text taken from Wikipedia. Pictures kindly received from *Marketing en zalenverhuur – Historische Huizen Gent*

Timetable

CT: Contributed Talk, IT: Invited Talk.

Thursday, December 12th

8:30–8:55	**Registration**		
8:55–9:00	**Welcome remarks**		
9:00–9:50	IT	**T. Minea** Université Paris-Saclay	Plasma behavior in pulsed magnetrons and new trends for plasma excitation
9:50–10:20	**Coffee break**		
first session	**Microstructural and textural control**		
10:20–11:10	IT	**N. Martin** FEMTO-ST	Structuring of thin films combining reactive gas pulsing and GLAD
11:10–11:30	CT	**J. Müller** University of Namur	In silico optimization of reactively sputtered meso-porous titanate-based thin films by genetic algorithm

11:30–11:50	CT	**K. Solanki** Université de Poitiers	Real-time growth monitoring of ultrathin Ag layers: impact of N_2 additives and seed layers on morphological evolution
11:50–12:10	CT	**T. Suszko** Koszalin University of Technology	MeMC/a-C:H type coatings with nanocolumnar, composite structure-synthesis and some properties
12:10–12:40	**Presentation of the posters–first group**		
12:40–13:50	**Lunch**		
13:50–14:25	**Presentation of the posters:second group**		
14:25–14:45	CT	**S. Frick** EMPA	Accelerating oxynitride coating development: Combinatorial investigation on the Al-Si-O-N system
14:45–15:05	CT	**F. Farahani** Ghent University	Do impurities have an influence on the phase composition of deposited tungsten films?

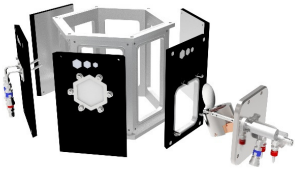
Second session	Emerging techniques		
15:05–15:55	IT	**A. Shukurov** Charles University	Reactive sputter-driven synthesis of transition metal nanoparticles and nanofluids
15:55–16:25	**Coffee break**		
16:25–16:45	CT	**P. Baroch** University of West-Bohemia	High-rate reactively sputtered Cu_2O thin films post-treated with high-power infrared laser
16:45–17:05	CT	**C. Schiffers** CemeCon AG	HiPIMS coatings for sub-micro tools for AI applications
17:05–17:25	CT	**P. Vašina** Masaryk University	Exploring different models of operation of multipulse HiPIMS
19:30–22:30	**Conference dinner**		

Session:	Poster presentations I		
12:10–12:15	P	**D. Loch** Trumpf Hüttinger GmbH	Silicon Dioxide Coatings by Reverse Pulse HIPIMS
12:15–12:20	P	**M. Akagawa** Ghent University	Real-time in-situ sheet resistance measurements to study silver thin film nucleation during magnetron sputtering
12:20–12:25	P	**F. Lourens** Ruhr University Bochum	Exploring Engineered Artificial Minerals for Lithium Recovery from Recycling Slags: Insights from Thin Film Experiments
12:25–12:30	P	**J. Müller** Fraunhofer Institute for Mechanics of Materials	Reactive Superimposed HiPIMS / RF Deposition
12:30–12:35	P	**N. Rodkey** Empa	Accurate Reporting of Time-of-Flight Measurements with Gated Mass Spectrometry
12:35–12:40	P	**M. Aelbrecht** Soleras Advanced Coatings	New thermal spray ceramic NiOx(CrOx) targets for sputtering functional layers in low-E glass coatings

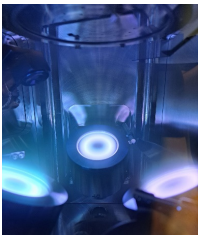
Session:	Poster presentations II		
13:50–13:55	P	**P. Marx** Ruhr University Bochum	Combinatorial Sputter Synthesis and Characterization of (La-)Co-based Thin-Film Spinel and Perovskite Materials Libraries
13:55–14:00	P	**E. Dobruchowska** Koszalin University of Technology	NiMo-C coatings synthesised by reactive magnetron sputtering as a catalyst for the hydrogen evolution reaction in an acidic environment
14:05–14:10	P	**E. Strods** University of Latvia	Optical and photochromic properties of yttrium oxyhydride thin films deposited by reactive magnetron sputtering
, 14:10–14:15	P	**A. le Febvrier** Linköping University	Embedding Fe Nanoparticles into CrN Films For enhancing thermoelectric properties
14:15–14:20	P	**O. Brune** Technical University of Munich	Enhancing Charge Transport in Metal (Oxy-)Nitrides for Efficient Solar Fuel Generation

14:20–14:25	P	**J. Ellingford** Plasma Quest Ltd	Conformal Coverage of Complex Topographies in a Reactive Process using Remote Plasma Sputtering

Korvus Technology Ltd



Korvus Technology manufacture completely modular PVD systems. Their product range is called “the HEX Series”, with each chamber having six detachable side panels, as well as top and bottom panels.



This style of chamber design offers users immense flexibility. Users can begin with a very simple chamber and add further deposition sources later. They can also add customisations from third-party suppliers.

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CT: Contributed Talk, IT: Invited Talk.

Friday, December 13th

Third session	Energy applications and process control		
9:00–9:50	IT	**P. Eklund** Uppsala University	Thin film ceramics for Energy Applications
9:50–10:10	CT	**L.I. Wagner** Technical University of Munich	Engineering Ti-doped Ta_3N_5 Photoanodes via Reactive Magnetron Co-Sputtering for Enhanced Solar Fuel Applications
10:10–10:30	CT	**T. Kubart** Uppsala University	Superconducting NbN thin films deposited by reactive magnetron sputtering
10:30–11:00	**Coffee break**		
11:00–11:20	CT	**L. Maroto-Diaz** Gencoa Ltd.	The study of defect reduction in optical coatings produced using reactive process feedback control
11:20–11:40	CT	**J. Van Bever** Ghent University	How to converge feedback control and measure double hysteresis?

11:40–12:00	CT	**K. Choglay** Manchester Metropolitan University	Exploring the Link between Plasma Gas Speciation and the properties of TiO_xN_y Thin Films
12:00–13:30	**Lunch**		
Fourth session	**Smart sensing and complex coatings**		
13:30–14:20	IT	**F. Vaz** University Minho	Specially architectured thin films for sensing applications
14:20–14:40	CT	**A. Crovetto** Technical University of Denmark	Phosphide and phosphosulfide thin films by reactive sputtering
14:40–15:00	CT	**P. Kelly** Manchester Metropolitan University	Deposition of $BiVO_4$ Thin Films by Reactive Magnetron Co-Sputtering for Visible Light Photoelectrochemical Water Splitting
15:00–15:20	CT	**D. Depla** Ghent University	The future of RSD and reactive sputtering
15:20–15:30	**Closing ceremony**		

Abstracts – Talks

December 12th

Plasma behavior in pulsed magnetron and new trends for plasma excitation

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High-Power Impulse Magnetron Sputtering (HiPIMS) is a mature technology that has been effectively transferred to the industry. Since its early stage at the end of the previous century, the understanding of HiPIMS plasma behavior has significantly advanced [1].

The first part of the talk will present a review of HiPIMS plasma physics as it emerges from the modeling backed by the experimental results. Both reactive and non-reactive cases will be treated.

In the second part, several novel ways to excite the plasma (multi-pulse, e-HiPIMS (Electron enhanced – HiPIMS) [2], and Hyper-Power Impulse Magnetron – HyPIM [3,4]), together with their specific parameters, will be introduced, essentially for the plasma species facing the substrate. This key information is of major importance for controlling thin film properties.

Throughout several examples (5-7), we will show how to tailor ultra-thin films, taking the benefit of mastering the HiPIMS technology.

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Structuring of thin films combining reactive gas pulsing and GLAD

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The GLancing Angle Deposition (GLAD) is a recent technique to play with the structure of thin films [1]. It was successfully developed to sputter deposit thin films exhibiting original architectures. This approach employs oblique angle deposition and controlled substrate motion to form a film composed of nanometer scaled columns of designed shape. It allows the growth of compounds with a carefully engineered structure at the sub-micron scale. Thus, various forms (zigzags, spirals, oriented columns and so on) through the film thickness can be produced, which give rise to new geometries of the film structure [2,3]. On the other hand, the Reactive Gas Pulsing Process (RGPP) is also a recent method to control the amount of reactive gas injected during the film deposition, and finally to play with the nature of the deposited material [4,5].
This presentation aims at illustrating how physical properties and anisotropic behaviors thin films based-on metals, oxides, nitrides and oxynitrides prepared by sputtering can be modified involving RGPP, or GLAD, or even by combining simultaneously these two techniques RGPP + GLAD. The basic principle of these "young" techniques will be presented in terms of key parameters and reachable architectures. Some characteristics of as-deposited thin films will be discussed especially showing the correlations between the dimensions, shapes and geometries of produced architectures and the resulting properties. The achievement of Janus-like structures of metals (W-Mo, W-Cu, Ti-Ag,. . .), periodic multilayers or other original designs will be discussed. Finally, anisotropic behaviors in terms of optic, electronic and acoustic properties of these structured thin films will also be pointed out.

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In silico optimization of reactively sputtered meso-porous titanate-based thin films by genetic algorithm

J. Müller[1,3], ***P. Moskovkin***[1,3], ***A.G. El Hachimi***[1], ***S. Lucas***[1,3], ***R. Adalati***[2], ***S. Konstantinidis***[2]

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Glancing Angle Deposition (GLAD) combined with reactive magnetron sputtering can be a method of choice where film structure must be controlled at the nanoscale for improved material performance. One promising application is the fabrication of nanostructured (Lithium) Titanate-based thin film anodes in the domain of Lithium-ion batteries.

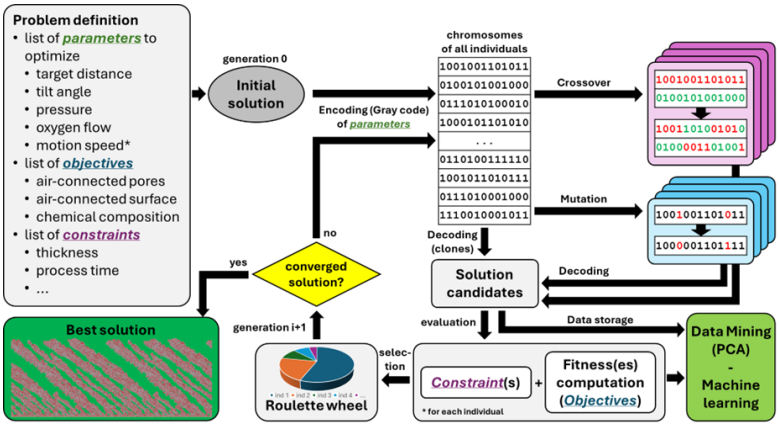

Figure 1: Flowchart of PVD process optimization by genetic algorithm.

In this work, we are investigating meso-porous thin films deposited by reactive magnetron sputtering process in GLAD mode. The aim is to show how the structuration of such coatings can be optimized to maximize their air-connected surfaces while keeping the pores size in a range of 2-50 nm. The deposition process is modelled by using the kinetic Monte-Carlo software Virtual Coater, allowing to simulate the complete sputtering process, from the design of the coater to the atomic-scale film growth simulation and film characterization.

For this purpose, this digital twin has been directly integrated to an optimization scheme which uses multi-objective genetic algorithm (Figure 1).

A special attention is paid to the influence of the coater configuration and typical deposition parameters (power, pressure,$\cdots$) on the structural properties of the coating. This approach allows to reduce substantially the R&D time which is necessary to find optimal process parameters.

This research was supported by a project of the Walloon Region's (Belgium) Recovery Plan entitled "BatFactory".

Real-time growth monitoring of ultrathin Ag layers: impact of N_2 additives and seed layers on morphological evolution

K. Solanki[1], ***G. Abadias***[1], ***F. Pailloux***[1], ***A. Michel***[1], ***K. Dussaillant***[1], ***D. Babonneau***[1]

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Ultrathin continuous silver (Ag) films with thickness below a few nanometers are interesting candidates for use as transparent conductive layers in flexible optoelectronic devices [1], because of several benefits such as low-cost production, high electrical and thermal conductivity, high optical transmittance in the visible range, and high ductility. However, Ag layers deposited by 'conventional' physical vapor deposition on weakly interacting (oxide) substrates have natural tendency to form three-dimensional nanoscale islands, which yield layers with rough surface morphology, high resistivity and broad absorption bands in the visible range due to the excitation of localized surface plasmons [2]. Deployment of gaseous additives during magnetron sputtering (MS) deposition or use of seed layers have been shown to be effective paths for promoting wetting of Ag on the substrate surface, resulting in the formation of a continuous layer at a lower nominal Ag thickness, without compromising the Ag-layer optoelectronic properties [3].

In the present work, we discuss the overall experimental strategy and demonstrate the way by which N_2 gas additives and seed layers such as Silicon (Si) and Silicon Nitride (Si_3N_4) affect the growth morphology of the film. In particular, information on the early stages, such as percolation threshold and onset of film continuity, are gained from a combination of in situ and real-time diagnostics during MS deposition, using Magnification Inferred Curvature and Surface Differential Reflectance Spectroscopy. Furthermore, the stability of these ultrathin Ag films is examined by studying relaxation processes after short-time growth interruptions as well as long-term aging. These investigations are augmented by ex situ characterizations by transmission electron microscopy, X-ray diffraction, spectrophotometry, spectroscopic ellipsometry, and electrical resistivity measurements.

We show that in pure Ar discharge, continuous layers are formed at nominal thicknesses of 6 nm and 11.5 nm for Ag films grown on Si and Si_3N_4 layers, respectively, which occurs considerably earlier than for Ag deposition on SiO_2. Moreover, for Ag deposition on both SiO_2 and Si_3N_4, our results show that the continuity thickness can be further decreased when N_2 is introduced in the plasma. The presence of N_2 also reduces post-growth relaxation effects and enables to produce Ag films with high optical transparency and low electrical resistivity.

Acknowledgments: This work is part of the IRMA project funded by the ANR and DFG (reference ANR-21-CE09-0041-01). and LABEX INTERACT-IFS (reference ANR-11-LABX-0017-01)

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MeMC/a-C:H type coatings with nanocolumnar, composite structure – synthesis and some properties

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As a result of reactive magnetron sputtering metallic targets in argon/acetylene atmosphere, carbon-oversaturated multicomponent MeMC/a-C:H coatings were synthesised. They have a specific nanocolumnar, composite structure consisting of quasiamorphous metallic columns with diameter of several nanometres surrounded by a layer of amorphous carbon (cf. Figure 1).

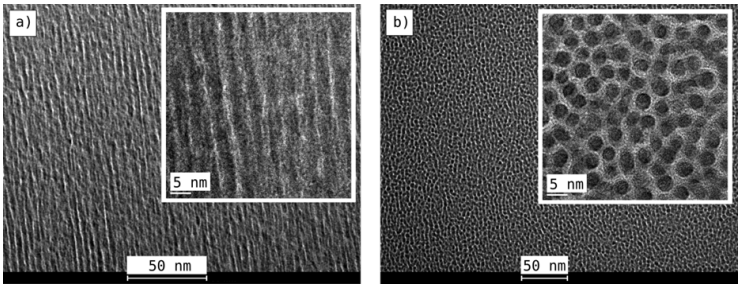

Figure 1: HRTEM images of the FeCrNiC/a-C:H coating containing 44 at.% carbon, in (a) cross-section and (b) plan-view with respect to the substrate surface.

This structure was observed for four systems: FeCrNiC/a-C:H, CoCrMoC/a-C:H, NiCrC/a-C:H and NiMoC/a-C:H [1–4]. The details of the synthesis will be discussed, including reports from other authors [5,6]. The role of weak and strong carbide-forming elements in the synthesis [7,8] as well as selected properties of the deposited coatings will also be discussed.

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Accelerating oxynitride coating development: Combinatorial investigation on the Al-Si-O-N system

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The material class of oxynitrides features promising candidates for various application fields, including photocatalysts, pigments, phosphors, high k-dielectrics or wear resistant coatings [1-2]. In order to accelerate the optimization of the composition for the respective application, combinatorial materials science is a commonly applied method. In case of the magnetron sputtering technique, the composition screening is in literature mainly limited to cation gradients. Since oxynitrides' properties are especially dependent on the oxygen-to-nitrogen ratio, the controlled deposition of anion gradients – perpendicular to a cation gradient – should accelerate the development of complex quaternary oxynitride systems significantly. In this work, three different approaches to obtain gradients in the oxygen-to-nitrogen ratio were investigated, including varying local gas inlets and target configurations, and their applicability was evaluated.

As an application case, the Al-Si-O-N system was chosen as a promising candidate for protective optical coatings due to the presence of a nanocomposite phase [3] and the tuneability of the refractive index via the oxygen-to-nitrogen ratio [4]. Therefore, thin film combinatorial libraries were deposited via reactive Hybrid Al-HiPIMS/Si-DCMS. Applying the most successful technique for controllable anion gradients, five depositions were sufficient to screen simultaneously an anion content of 1-45% O/(N+O) and a cation content of 5-40% Si/(Al+Si). Subsequently, those combinatorial libraries were comprehensively characterized using high-throughput procedures of XPS, XRD, UV-vis photospectrometry and nanoindentation. The refractive index was obtained from UV-vis transmission spectra applying the envelop method according to Swanepoel [5] revealing a span of 1.80-2.03 in the investigated

composition range. Corresponding hardness values varied between 11 and 25 GPa. The comprehensive data sets allow for deeper insight into this complex material system by correlating the target properties to compositional, structural and charge transfer characteristics.

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Do impurities have an influence on the phase composition of deposited tungsten films?

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This study investigates the phase composition and texture evolution of magnetron sputter deposited tungsten (W) thin films [1]. Using X-ray diffraction (XRD) analysis, two distinct phases were observed: the metastable β-W phase, associated with (200) and (210) Bragg reflections, and the thermodynamically stable α-W phase, characterized by the (110) reflection. To understand how deposition conditions affect W-phases, thin films with varying thicknesses (50–200 nm) on Si (100) substrates were deposited in a high-vacuum magnetron sputtering system changing the argon pressure (0.3-0.7 Pa) and discharge power (50-250 W). To explore the role of impurities, series of 200 nm thick films were deposited in two separate vacuum chamber at three different base pressures: 3×10^{-3}, 4×10^{-4}, and 3×10^{-5} Pa. Residual gas analysis (Extorr XT Series RGA) shows that the most abundant gas is water. Based on the deposition rate and the residual gas pressure, the flux ratio τ between the flux of impurities and deposited W atoms was determined. The results indicate that impurities do not define the phase composition of W films (Figure 1a). The same data shows that higher discharge power shifts the phase composition significantly towards α-W. Additional experiments demonstrate a clear transition from β-W to α-W with increasing film thickness while hardly any influence of the argon pressure was observed.

As mainly the discharge power seems to influence the phase composition, energy flux measurements were conducted using a calorimetric probe. This rules out a temperature driven mechanism. Using simulations [2,3], the phase composition could be connected to the momentum of reflected neutrals which is mainly defined by the discharge voltage, and hence the discharge power. The observed correlation is presented in Figure 1b which shows the phase composition as a function of the number of displacements per deposited atom (NDPA)

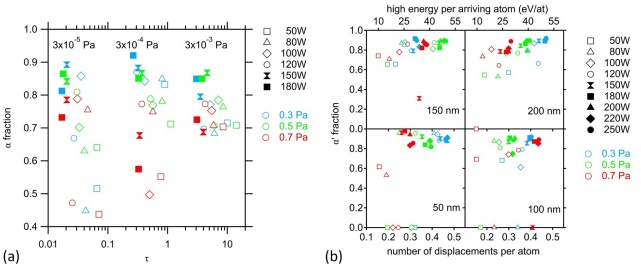

Figure 1: (a) The α fraction as a function of the ratio of the flux residual gas species (water) $F_{impurities}$ and deposited tungsten F_{metal}, defined as τ, (b) The α' fraction as a function of the NDPA. The fraction was calculated based on the α (110) and β (210) Bragg reflection.

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Reactive sputter-driven synthesis of transition metal nanoparticles and nano-fluids

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Human dependence on non-renewable energy sources is causing a global energy crisis. Possible solutions include the transition to renewable energy sources such as the wind and the sun. Here, we present the gas-phase synthesis of low-dimensional materials based on transition metal nitride nanoparticles (MeN NPs, Me = Ti, Zr, Hf, Ta, and Cu) that might be potentially suitable for solar harvesting or plasmonic sensing [1-3]. In contrast to conventional thin-film deposition, we show that reactive magnetron sputtering of metal targets in Ar/N_2 can be adapted to synthesize MeN NPs in the gas phase, avoiding high temperatures and toxic chemicals. Under proper conditions, highly crystalline and stoichiometric MeN NPs can be produced, with optoelectronic properties suitable for use in solar-driven electrochemical water splitting and as direct solar absorbers. Both planar and cylindrical magnetrons can be used for this purpose. Furthermore, we show that this method allows for direct deposition of MeN NPs into vacuum-compatible liquids to produce linker- and residual-free plasmonic nanofluids.

The plasmonic sensitivity of MeN NPs was found to exceed that of Au; however, they suffer from partial oxidation after contact with air. To overcome this issue, we developed a method to passivate Me NPs using rf magnetron sputtering of Si_3N_4 for the in-flight deposition of SiN protective layers over MeN NPs without contact with air. The SiN shell tunes the position of localized plasmon resonance (LSPR) of the MeN NPs from 580 to 850 nm by tuning the porosity and, consequently, the effective refractive index of SiN. Although the plasmonic sensitivity becomes attenuated, the SiN layer protects MeN NPs from post-deposition oxidation in air and preserves LSPR at temperatures above 400°C. Thus, our method offers an environmentally benign, annealing-free route to MeN NPs and nanofluids with controllable optical properties, enhanced thermal stability, and promising features for novel applications.

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High-rate reactively sputtered Cu_2O thin films post-treated with high-power infrared laser

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The cuprous oxide-based (Cu_2O) materials are one of the most promising and studied p-type transparent conductive oxide (TCO) materials. This is because of copper earth abundance, Cu_2O non-toxicity, and relatively suitable opto-electrical properties. However, the performance of all state-of-the-art p-type TCOs (including Cu_2O-based) is insufficient compared with well-established n-type TCOs (ITO, AZO, ...), especially in terms of electrical conductivity [1,2]. In this work, we have utilized different approaches to improve the optical and electrical properties of Cu_2O-based films. We combined the growth of Cu_2O films by high-power impulse magnetron sputtering (HiPIMS) at different pulse-averaged target power densities together with the utilization of a well-established high-power infrared laser.

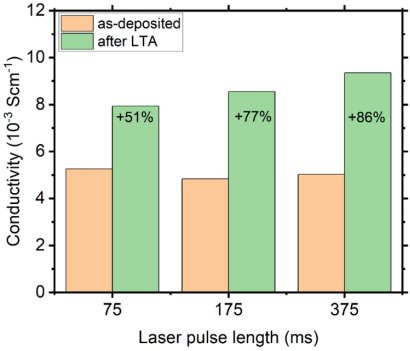

Figure 1: . Evolution of electrical conductivity of as-deposited Cu_2O films post-treated by laser pulses of various lengths.

We have systematically studied the effect of laser parameters on the optoelectrical properties of Cu_2O thin films, namely electrical conductivity, the concentration of holes and their mobility, optical band gap and

microstructure. We have found that this method could be a promising way to enhance hole mobility in Cu_2O-based materials without the requirements of high temperature and/or a special working atmosphere. As shown in Figure 1, we were able to increase the film conductivity of as-deposited Cu_2O films (prepared at high-rate of $>$150 nm/min) almost by a factor of two just by applying a single laser pulse with a length of 375 ms.

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HiPIMS coatings for sub-micro tools for AI applications

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Miniaturization is a clear trend even for objects around us in our daily lives. That's why cutting tools are becoming smaller and more precise. It started with watchmaking medical/dental applications. Reactive sputtering was always the technology of choice for tools below 1 mm. Today's driver is AI technology. It makes tools for drilling computer boards a high-volume application for extremely small diameters in the 0.25 mm range.

HiPIMS is the most suited coating technology for sub-micro tools since it gives smooth coatings without any droplets, a dense and fine-grained morphology of the film together with low intrinsic stresses.

There are different approaches to HiPIMS, and this talk will give an overview of the various technologies and how new developments such as chopped HiPIMS, pulse synchronisation and positive kick paves the way for bringing HiPIMS into industry.

More knobs to turn – HiPIMS gives effective control over the plasma. The settings of the HiPIMS pulses allow to tailor the energy precisely to micro tools. This avoids antenna effects and defects because of over-etching the fine geometry. Precision machining with micro tools requires super sharp cutting edges. The unique HiPIMS feature of synchronizing the pulses at the cathodes with the HiPIMS Bias supply is the key to actively managing the intrinsic stresses in the film.

HiPIMS with its unique combination of favourable properties opens new business opportunities in the growing market of precision and sub-micro machining. A very good example of how the academic research in reactive sputtering supports the European industry when the traditional bread-and-butter business for the car industry is declining.

Exploring different modes of operation of multipulse HiPIMS

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Three different modes of operation for using HiPIMS with a titanium target at argon buffer gas were examined in terms of plasma diagnostics and coating properties. In all three modes of operation, a 1 kW average power was kept constant. The first mode of operation involves splitting a strong single HiPIMS pulse into multiple evenly distributed weaker pulses by proportionally dividing the period. The second mode of operation is based on the first one, but the pulses are grouped together in a short burst, or pulse package, where the overall period is conserved. Comparing the second mode of operation to the first enables the identification of the effect of pulse grouping. In the third mode of operation, the pulses are also grouped into the pulse package, however, the period is extended proportionally to the number of pulses in the pulse package. This allows for the creation of short bursts of energetic pulses separated by very long off–times while maintaining the duty cycle. In this way, it is possible to determine whether the grouping of the strong pulses into a pulse package is more beneficial for the deposition than the negative effects induced by prolonging the off-time. Plasma diagnostics revealed that grouping the pulses into packages in the second mode of operation resulted in a higher ionised metal flux fraction on the substrate compared to first strategy, which led to stronger ion bombardment of the growing coating, resulting in denser coatings and changes in the crystalline microstructure. The third mode of operation does not increase significantly ionised metal flux fraction but influenced the texture and grain size of the growing films.

December 13th

Thin-film ceramics for energy applications

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This invited presentation gives an overview of our research on selected thin-film-ceramics for energy applications in the field of protective coatings and energy harvesting. Protective coatings made by energy-efficient reactive sputter deposition for applications in electrochemical cells and nuclear fuel rods are surveyed. Dense, hard coatings in the CrNbN system ranging from nitrides to supersaturated metal solid solutions offer wear resistant coatings that also have potential to withstand the harsh conditions nuclear fuel rods are exposed to in a reactor. In particular, Cr-rich coatings with 2-6 at. % and 10 at.% exhibit notably dense microstructures with relatively high hardness, properties of the essence for protective coatings. For thermoelectric devices, I present an overview of our work on multicomponent CrN-, ScN-, and $Ca_3Co_4O_9$-based thin films. We have developed methodology for highly textured as well as nanoporous virtually phase-pure $Ca_3Co_4O_9$ thin films. These can further be deposited on flexible mica substrates, enabling flexible inorganic thermoelectric thin films that withstand repeated bending. They can also be made as free-standing films and as nanoporous materials for reduced thermal conductivity. CrN exhibits n-type conduction with a high power-factor enabled by a high electron concentration thermally activated from N vacancies, and alloys can be made of rocksalt-$Cr_{1-x}Sc_xN$. Multicomponent alloying of ScN and CrN in alloys such as CrMoVWN or combinations thereof offer further possibilities for tailoring the thermoelectric properties and growth conditions.

Engineering Ti-Doped Ta_3N_5 Photoanodes via Reactive Magnetron Co-Sputtering for Enhanced Solar Fuel Applications

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Tantalum nitride (Ta_3N_5) is a promising semiconductor for photoanodes in solar fuel generation, but its performance is often limited by inefficient charge carrier transport and significant carrier trapping, caused by a high density of defects that introduce deep-level electronic states within its bandgap. In this work, we employ reactive magnetron co-sputtering combined with ammonolysis to improve the charge transport properties of Ta_3N_5 photoanodes with Ti doping. By systematically exploring sputter power variations, substrate pre-treatments, choice of interlayer materials, and substrate types, we optimize the synthesis process to achieve controlled Ti doping, with concentrations ranging from 0 to 3 at.%. This is enabled by carefully managing the sputter rates and intentional Ti target poisoning [1].

Comprehensive characterization, including structural, compositional, optical, electrical, and photoelectrochemical methods, reveals that Ti^{4+} ions substitute Ta^{5+} lattice sites, thereby introducing compensating acceptor states, reducing concentrations of nitrogen vacancies, and reduced Ta^{3+} states, and suppressing trapping and recombination. Consequently, we demonstrate that Ti doping decreases the conductivity immensely by lowering the charge carrier density but simultaneously increases the mobility of free charge carriers due to reduced recombination at nitrogen vacancies [1].

Building on these findings, we investigate various substrate conditioning techniques and interlayer depositions using reactive sputtering to further optimize light harvesting and charge transport to the counter electrode. Additionally, we explore the use of transparent substrates that can withstand the high-temperature conditions required for reactive sputtering and ammonolysis.

Overall, this work provides valuable insights into the precise engineering of Ti-doped Ta_3N_5 photoanodes, significantly improving their performance as advanced photoanodes for solar fuel applications. These results lay the foundation for further enhancements in the design and fabrication of high-efficiency photoelectrochemical systems.

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Superconducting NbN thin films deposited by reactive magnetron sputtering

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NbN is an interesting material for superconducting metallization of semiconductor devices thanks to its high critical temperature of about 16 K. For integration in devices, the thin films need to be deposited on Si substrates, preferably without substrate heating. This contribution addresses the reactive sputter deposition of NbN in order to identify optimum microstructural parameters for films with good superconducting performance. Thin films were deposited on Si and SOI substrates from a Nb target in an Ar+N_2 mixture using dcMS and HiPIMS. A typical dependence of the critical temperature, T_C , on the N_2 gas flow is shown in Figure 1a. However, this graph does not provide sufficient insights for optimization of the deposition process. We have identified two parameters that are crucial for the film performance.

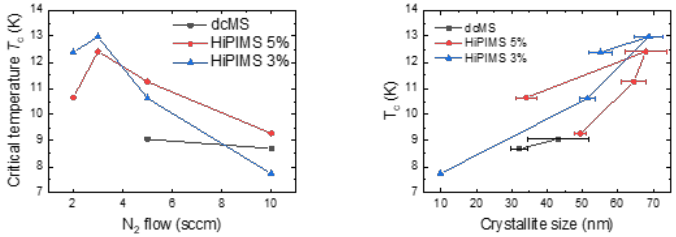

Figure 1: (a) Critical temperature T_C of 100 nm NbN thin films deposited by dcMS and HiPIMS as a function of the N_2 gas flow. (b) T_C as a function of the crystallite size. The lines connect the points according to the increasing N_2 flow.

- The film morphology is correlated with the resistivity at room temperature. An underdense columnar microstructure developing in dcMS is detrimental to the resistivity. Metal flux ionization

in HiPIMS reduces the shadowing effect and results in a substantially higher film density and lower resistivity. A significantly lower overall oxygen content is also observed as a result of reduced oxidation during air exposure. Furthermore, the dense films exhibit a sharper superconducting transition.

- The T_C is directly related to the crystal quality, measured as the crystallite size, Figure 1b, and the microstrain. All films exhibited the desired δ-NbN fcc structure. However, the crystallite size was almost 70 nm for the films with the highest T_C as compared to 50 nm achieved in dcMS. The best performing films also exhibited the smallest amount of microstrain.
- In summary, we show a deposition process for synthesis of dense films on Si without substrate heating. The highest T_C achieved in this work for films 100 nm thick was 13 K. For thinner films of 20 nm, the T_C was 9.5 K.

This research was funded within the QuantERA II Program under Grant Agreement No 101017733, by the Swedish innovation agency Vinnova, project 2024-00436, and by Olle Engkvist Foundation, project 213-0276.

The study of defect reduction in optical coatings produced using reactive process feedback control

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Electro-optic thin film coatings are critical in many everyday technologies. The coatings manipulate light in response to an electrical input, making them essential in a wide range of devices and applications such as in smartphones, eyewear and imaging devices.

In the fabrication of electro-optic coatings, defect control is critical for ensuring optimal device performance and precise optical and electrical properties. Even small defects in the thin film structure can lead to significant issues, especially in high-precision applications.

This study investigates the use of feedback control methods, in combination with a rotating magnetic array, to reduce coating defects during reactive sputtering processes. The rotating magnetic array not only enhances target utilization but also reduces arcing on the target surface by distributing the plasma more evenly across the target. These effects lead to more stable sputtering conditions and minimize issues such as micro-arcs. Real-time feedback control of key process parameters - such as reactive gas flow - dynamically adjusts conditions to further mitigate defects.

In this study we produce aluminium nitride coatings on 150 mm silicon wafers using a 300 mm wide circular target. The final defect count was analysed across the wafer surface for a range of defect sizes. Defect counts are investigated using optical methods and counting software to determine the final defect count. The results show that combining feedback control with the rotating magnetic array significantly improves the defect count when compared to industry standard methods to produce aluminium nitride coatings. A key discovery was the importance of tuning the feedback control loop, where the setpoint of control was a crucial parameter in the reduction of small and large defects.

How to converge feedback control and measure double hysteresis ?

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The transition region of the hysteresis curve in reactive magnetron sputtering is an important yet inherently unstable mode, posing significant challenges for achieving stable control. This mode offers opportunities to deposit coatings with unique properties such as a specific optical index or thermochromic behavior, and achieving stability requires effective feedback control [1,2].

Applying feedback control introduces additional complexities due to various factors influencing stability and convergence, such as reactive gas pressure changes [3], target erosion [4], feedback loop adjustments [5], arcing [6], and oxide deposition [7]. Some of these factors may also cause process drifting, particularly when the discharge voltage [1] or an optical emission line intensity [8] are used as feedback inputs. Our study systematically investigates these processes through time-resolved analysis and by linking them to the hysteresis framework, demonstrating, for instance, how oxide deposition shifts the hysteresis curve to higher voltages and influences the predicted rate of oxygen flow adjustment in feedback control as a function of process variables, such as pumping speed. These insights not only support an optimized method for achieving efficient feedback convergence but also enable a more detailed investigation of the transition mode.

A detailed understanding of target properties is essential for examining the transition mode, particularly given the Reactive Sputtering Deposition (RSD) model's prediction of history-dependent transition states [9]. More specifically, this model indicates that the process behavior differs depending on whether it originates from a metallic or poisoned state. A previous study by Schelfhout et al. [10] has measured double hysteresis to demonstrate this history dependence through an indirect method using IV-characteristic analysis. However, this approach is less suited for direct application in practical coating production. We present direct

measurements of double hysteresis using feedback control, offering an approach that better aligns with industrial processes.

Accurate double hysteresis measurements using feedback control require maintaining converged feedback control at each process point while ensuring measurements are timely enough to account for slow and irreversible changes like target erosion. To address these requirements, we detail a procedure to measure double hysteresis under feedback control, ensuring both convergence and efficient timing. These results provide the first demonstration of double hysteresis measurement under feedback control and highlight its dependence on key parameters, including discharge current and argon pressure.

This work strengthens the link between theoretical predictions and practical industrial requirements for reliable coating processes, improving stability and performance in deposition settings.

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Exploring the Link Between Plasma Gas Speciation and the Properties of TiO_xN_y Thin Films

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The properties of reactively sputtered titanium oxynitride (TiO_xN_y) thin films are highly sensitive to the reactive gas (O_2 and N_2) environment during the deposition. Thus, by carefully regulating the ratios of oxygen and nitrogen in the plasma, it becomes possible to tailor the material's optical, electrical, and structural characteristics.

In this work TiO_xN_y was deposited from a metallic titanium target using a pulsed DC power supply and in a reactive environment composed of an $Ar/O_2/N_2$ gas mix. The amount of N_2 was held constant at 3 sccm during the deposition and the amount of O_2 was varied to obtain a hysteresis loop (Figure 1a.). The addition of nitrogen into the plasma closes the gap between the forward and reverse hysteresis cycles, and therefore, provides more control over the process. The gas speciation was monitored in-situ using the Gencoa Optix system and plasma intensity was measured using optical emission monitoring (OEM). A range of material characterisation techniques were performed on films deposited at different OEM setpoints, such as XRD, XPS, Dektak stylus profilometry and sheet resistance and uv-vis spectroscopy to evaluate their optical and physiochemical properties.

By utilizing the Gencoa Optix for in-situ plasma monitoring (Figure 1b), a clearer connection can be made between plasma gas speciation and the optical and physiochemical properties of TiO_xN_y thin films. Understanding these relationships is crucial for optimizing the performance of these films in applications like optical coatings, photocatalysis, and electronic devices. This study provides new insights into how reactive gas species influence the tuneable properties of TiO_xN_y films, enabling enhanced process control and greater flexibility in material design. In this work it was found, for example, that the TiO emission line at 777 nm

was only easily detectable once the OEM setpoint was beyond the toe of the hysteresis curve.

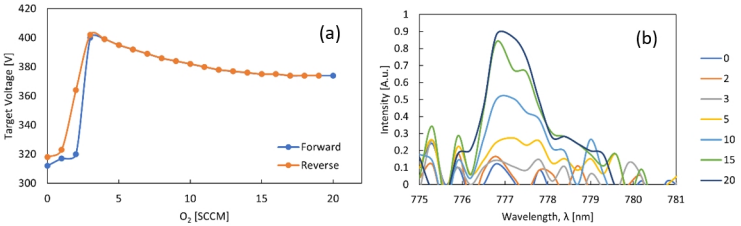

Figure 1: Shows the hysteresis voltage (a) and the change in intensity for the TiO emission line at 777 nm during the forward cycle for the reactive sputtering of TiO_xN_y (b). The legend shows specific O2 mass flow rates in sccm.

Specially architectured thin films for sensing applications

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One of the most important goals of nanotechnology is to engineer nanostructures and nanopatterns, paving the way for a society reliant on intentionally designed nanomaterials. This includes specialized domains such as nanoelectronics, nanomedicine, nanomachining, and various other applications based on nanoscale technologies.

Controlling the organization of molecular building blocks at the nanometer scale is of utmost importance, not only for advancing scientific understanding and generating valuable knowledge, but also for pioneering the development of next-generation technological devices with electrical, optical, chemical, or biological improved responses.

Physical Vapor Deposition (PVD) techniques offer great potential for developing nanoarchitectured thin films (nanostructures and nanopatterns) over specific areas of surfaces, machine parts and/or devices. This enables the preparation of solid-state features approaching the molecular length scale. Despite the well-established reliability of PVD techniques, severe requirements of processing are involved, including growing features and nucleation constraints, but also the lack of dimensional variation in the specific systems that are being developed, approaching manufacturing limitations.

In this presentation, the recent developments on specially grown nanostructured thin films will be explored regarding the versatility of the reactive magnetron sputtering technique to tailor the electrical, mechanical and optical properties of different thin films' systems and thus their potential in targeted applications, namely for physical, chemical and biological sensing.

Beyond a comprehensive demonstration of the significant variations in some basic properties of known thin film systems concerning their basic characteristics, this talk will highlight the emerging role of Glancing

Angle Deposition (GLAD)-based nanostructures as a versatile platform with extensive sensing capabilities.

In recent years, GLAD-fabricated thin films for sensing applications can replace conventional nanomaterials due to their broad scope, ease of fabrication, controlled growth parameters, and hence, sensing capabilities. The morphological features tailored at the nanoscale exhibit heightened sensitivity, and boast the optical, electrical, thermal, mechanical and tribological properties.

Phosphide and phosphosulfide thin films by reactive sputtering

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Despite the success of Group III phosphides as optoelectronic semiconductors, phosphorus-containing thin-film materials are still largely unexplored. This is unfortunate, because phosphorus is one of the most versatile elements in the periodic table, as it can take a range of positive and negative oxidation states giving rise to many possibilities for designing new semiconductors.

Using the example of copper as the metal, I will show two types of chemically diverse phosphorus-containing compounds that can be grown by reactive sputtering using a simple metallic Cu target and PH_3 reactive gas as the phosphorus source. First, two distinct binary copper phosphides (Figure 2) are accessible by reactive sputtering depending on the phosphorus chemical potential during growth. One is Cu_3P [1], a high-conductivity semimetal that is natively p-type doped by Cu vacancies. The other is CuP_2 [2], a 1.5 eV band gap semiconductor with homoelement P-P bonds that is of interest for thin-film photovoltaics. While Cu_3P had been grown in thin-film form before with other techniques, CuP_2 had not been grown before as a thin film by any technique.

A second interesting family of materials is obtained when phosphorus and copper are combined with a more electronegative species such as sulfur ("phosphosulfides"). There are hardly any reports of thin-film phosphosulfides in the literature [3]. "Hybrid" reactive sputtering of a Cu target in PH_3 under a beam of evaporated cracked sulfur enabled thin-film synthesis of the Cu_3PS_4 compound (Figure 2). Cu_3PS_4 is a 2.3 eV band gap semiconductor with remarkably high minority carrier lifetimes, which could find applications as a light emitter.

It is instructive to note that phosphorus is in different oxidation states in all these three compounds: -3 in Cu_3P, -1 in CuP_2, and +5 in Cu_3PS_4.

This case study emphasizes that reactive sputter processes can be powerful tools for the synthesis of new thin-film materials from exotic chemistries.

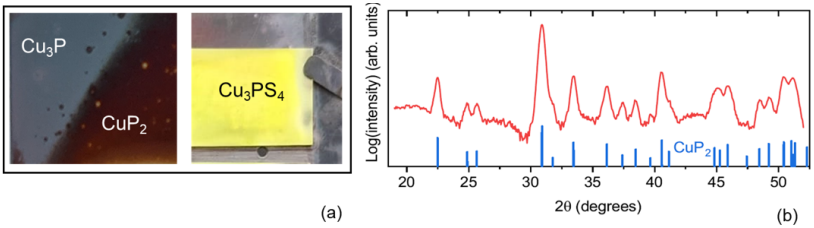

Figure 2: (a) Optical images of two Cu phosphide films with different Cu/P ratios and a Cu phosphosulfide film (Cu_3PS_4) obtained by reactive sputtering. (b) X-ray diffraction pattern on CuP_2, showing a polycrystalline single-phase film [2]

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Deposition of $BiVO_4$ Thin Films by Reactive Magnetron Co-Sputtering for Visible Light Photoelectrochemical Water splitting

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Over the last decades, hydrogen gained interest among the industrial and research communities as a "green" source of energy. Photoelectrochemical (PEC) water splitting appears to be a promising strategy for hydrogen production by using an abundant source of hydrogen (water) and a renewable energy source (solar energy). In PEC water splitting, photocatalysts immerged in an electrolyte are used as photoelectrodes to harvest the energy from a light source and convert it to drive the overall water splitting reaction. However, the practical viability of this process still depends greatly on solving multiple material challenges. Several materials attracted considerable attention over the past few years to perform the water oxidation reaction and to be used as a photoanode in a PEC water splitting cell. $BiVO_4$, in particular, possesses promising intrinsic properties, such as a low band gap, favourable band edges positions and good stability in aqueous environment. Nevertheless, some limitations are yet to be overcome, such as high electron-hole pair recombination rates and poor electrical conductivity.

Here, $BiVO_4$ thin films are produced using reactive magnetron co-sputtering from 2 metallic targets (bismuth and vanadium) in an argon and oxygen atmosphere. This configuration allows the optimisation of the properties of the deposited coating (chemical composition, morphological parameters…). Several characterisations are performed to identify the thin films produced (SEM, EDX, XRD, TEM, Raman spectroscopy…). The photoelectrochemical properties of the deposited samples are tested in a PEC water splitting cell, using a three-electrodes configuration with an Ag/AgCl reference electrode and a Pt mesh as counter electrode.

The future of RSD and reactive sputtering

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No one likes to be the last speaker on a meeting as everyone gets tired, hungry and wants to go home. So, this talk is not intended to be a real scientific contribution but merely aims to discuss a few interesting trends linked to reactive sputtering. Frankly speaking, on most questions I don't even have the answer but at least it is fun to think about it and go home with perhaps new insights.

From the interesting presentations given during this edition of the RSD conference, I have learned many things. The application of the technique to generate fascinating coatings and to develop new thin film materials shows that reactive sputter deposition is still very much alive. Key questions in this research field are both fundamental, application driven and connected new trends:

- How will AI and machine learning assist in improving our knowledge about this technique?: I recently read a paper about the use of machine learning for the prediction of the sputtering yields of elements. The approach shows some interesting ideas, but does it provide new physics? Most of the conclusions can also be made after reading the paper you all have read "Theory of Sputtering. I. Sputtering Yield of Amorphous and Polycrystalline Targets", by P. Sigmund. I don't want to sound old fashioned but we should use AI and machine learning not to invent the old in another way, but to explore new ideas to tackle questions regarding reactive sputtering.

- Is reactive sputtering sustainable? Or better, are coatings sustainable?: A question I often ask to my students during a class on thin film deposition is to estimate the mass of Ag consumed per year by an average large area coating set-up for architectural glass. They come very quick to conclusion that although the typical thickness of the coating is only 10 nm, the plant converts

approximately 1 tonne of silver into these layers. Will we every be able to recycle the metals we sputter deposit?

- Fundamental research regarding reactive sputtering is my hobby. After more than 20 years of research I can now answer the questions "Why is the deposition rate during poisoned mode so low?" In a few slides I will explain the main idea, but isn't surprising that this same question was the start of the RSD conference series.

So, to end, the talk will give a short overview of the history of this conference with the intention to start the debate on its continuation, and format.

Abstracts – Posters

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Testbourne is a supplier of high purity metals, alloys & compounds. We can offer an extensive selection of materials available in fabricated forms such as sputtering and arc targets, evaporation materials, powders, wire, rods & sheets. Testbourne supplies industries such as Architectural & Automotive glass, Hard Wearing & Decorative, Electronics, Electro-optics and research establishments for over 40 years. At our instrumentation department we represent brands like, Ceramtec (connectors, feedthroughs etc.), Inficon (quartz sensor crystals) and RD Mathis (Evaporation boats, Crucibles). For more information please look at our internet site or contact us.

Testbourne offers a reclamation services on all precious metals. Used precious metal targets either bonded or un-bonded, melted pieces and chamber scrapings may be reclaimed. After refining of the spent material we will either issue a credit or off-set against a new order.

Silicon Dioxide Coatings by Reverse Pulse HIPIMS

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Transparent oxide layers such as silicon dioxide play a vital role in a wide variety of applications in modern architecture from photovoltaics to low e glazing as well as in displays and optical sensors. Utilising High Power Impulse Magnetron Sputtering (HIPIMS) it is possible to deposit high density and high hardness layers. In this study the effect of utilising reverse voltage on the coating properties and arc likelihood were examined.

Utilisation of the constant current mode in the pulses prevented process instabilities usually linked to current runaway in reactive processes.

SiO_x was deposited from pure silicon targets in a reactive Ar-O_2 atmosphere. Peak power density was 0.5 A/cm^2, the pulse duration was up to 20 μs. OES observation of Ar neutral lines (Ar I) showed that plasma persisted to more than 150 μs after the pulse switch off.

Arcing rates were significantly reduced using reverse pulses due to the discharging of the target surface. The delay between the end of the pulse and application of the reverse voltage had a strong influence on arcing rates. Applying a reverse voltage after allowing the plasma near the target to decay slowly after switch-off, resulted in inefficient target neutralisation and higher arcing rates. Application of reverse voltage immediately at the end of the pulse utilised high density plasma to neutralise the target and eliminate arcing. SiO_x films deposited without additional heating or substrate biasing had a refractive index of 1.43-1.48, similar to bulk glass. A transparency of 97% and k-value of 10^{-3} for 200 nm thick films indicated low defect density achieved at the lowest arc rate. The nanohardness of 1 μm thick films was 10$\pm$1 GPa

and Young's modulus 77±9 GPa, representing a 10% increase against the glass substrate.

Real-time in-situ sheet resistance measurements to study silver thin film nucleation during magnetron sputtering

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Applications such as low-emissivity window coatings [1] and biosensors [2] require the growth of flat metal layers on substrates with a weak film/substrate interaction, such as on oxides and 2D-material. This weak interaction can however be a drawback, as it is a decisive factor for pronounced 3D morphology [3]. Hence, to be able to control the shape and microstructure of such thin metal films requires further fundamental understanding of the mechanisms in the early growth stages.

In this study, the degree of 3D clustering during growth is examined under various deposition conditions by measuring the nominal film thickness at the percolation threshold. This is achieved by monitoring the sheet resistance during nucleation of thin silver films on soda-lime glass substrates, using an in-situ four-point probe. From the drop in sheet resistance, the nominal film thickness at the percolation threshold is determined. Silver is deposited by direct current magnetron sputtering at room temperature, varying the target-substrate distances between 9.0 and 22.0 cm and the vapor arrival rate between 0.23 and 3.83 monolayers/s.

A power-law relationship is observed between the inverse vapor arrival rate and the percolation threshold thickness, with a power-law exponent between 0.08 and 0.28, depending on the target-substrate distance. Test particle Monte Carlo simulations indicate a significant flux of high-energy particles at the substrate, primarily due to argon ions reflected from the cathode. To further investigate the impact of this flux, experiments were repeated at a higher discharge voltage to enhance momentum transfer to the substrate. The resulting power-law exponents were consistently higher, ranging from 0.11 to 0.36.

An exponent of 1/3 corresponds to growth with a substantial rate of surface diffusion and coalescence processes, while lower exponents indicate reduced coalescence, with an exponent of 1/7 suggesting a lack of completed coalescence [3]. At larger target-substrate distances, slower deposition rates allow more time for complete coalescence, resulting in higher power-law exponents. Comparing results from the original and higher discharge voltage conditions suggests that increased momentum transfer in the latter delays percolation, likely promoting surface diffusion and favoring faster reshaping during island coalescence, ultimately leading to a more pronounced 3D morphology.

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Exploring Engineered Artificial Minerals for Lithium Recovery from Recycling Slags: Insights from Thin Film Experiments

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The growing demand for Li-ion batteries creates the need for sustainable recycling strategies. Current pyrometallurgical recycling processes recover the more noble elements (Co, Ni, Cu) while neglecting the slag elements (Li, Mn, Mg, Al). An approach for recovering these elements is modifying the slag's chemistry or solidification conditions to form Engineered Artificial Minerals (EnAMs) that are enriched in target elements and separable from the residual slag. Identifying suitable EnAMs and conditions to maximize their formation is critical, and high-throughput experimentation with sputtered materials libraries is an appropriate strategy.

This poster presentation is about a combinatorial phase constitution study of the thin film system Mg-Mn-Al-O (i.e., a slag subsystem) and the fabrication of Li-Al-O films.

Through high-throughput characterization of a reactive sputtered thin film materials library, spinel solid solution is observed over a wide compositional space of the Mg-Mn-Al-O system (Figure 1a), with Mn present in oxidation states 2+ and 3+, which is not a suitable EnAM phase [1]. This led to the idea of substituting $Mn^{2+/3+}$ with Mn^{4+}, so that in slags, the promising EnAM Li2MnO3 potentially forms, as no spinel-like compounds with Al^{3+} and Mn^{4+} are currently known. As a first step to evaluate this hypothesis with thin film materials libraries,

the fabrication of $Mn^{4+}O_2$ thin films by sputtering a MnO_2 target was optimized using reactive deposition conditions to compensate for the MnO_2 reduction that occurs during inert sputtering.

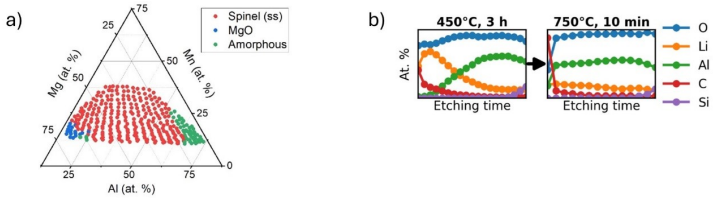

Figure 1: (a) Phase constitution in the thin film system Mg-Mn-Al-O. (b) Effect of Rapid Thermal Annealing on the XPS depth profile of a reactive sputtered Li-Al-O thin film on a Si substrate.

Investigating the formation of possible Li-containing EnAMs with combinatorial thin films requires good crystallinity in the films. However, reactive sputtered Li-Al-O films are observed as X-ray amorphous and develop inhomogeneous depth profiles during annealing, with Li tending to migrate towards the film surface due to its volatility. It is shown that this effect can be counteracted by using Rapid Thermal Annealing instead of conventional annealing (Figure 1b) [2].

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Reactive Superimposed HiPIMS / RF Deposition

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We investigated a superposition of RF and HiPIMS [1] on a single magnetron and studied the influence of pressure, target powers and reactive gas flow on process stability, peak currents and pulse form for the deposition of AlN and Al_2O_3.

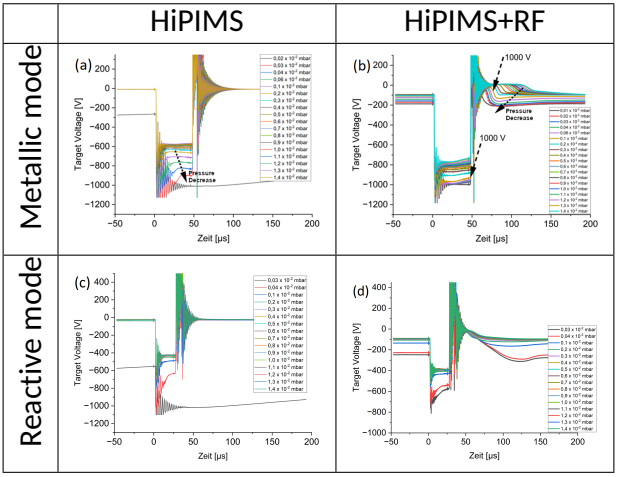

Figure 1: Measured HiPIMS voltage forms for varying pressure and oxygen flow, with and without RF-superposition; Al-deposition (45µs pulse) respective Al_2O_3-deposition (25 μs pulse). Applied power: 1 kW HiPIMS / 500 W RF

For that purpose, we modified a custom-built sputter coater (FHR SV 400) and connected a SPIK3000A-EF-05 HiPIMS Generator (Melec) and a CEASAR 1325 RF Generator (Advanced Energy) to the same source [2]. The outputs of both power supplies can be applied individually, continuously superimposed or synchronized during the off-times of the HiPIMS pattern. The HiPIMS signals were recorded using an oscilloscope (see Figure 1). For Al_2O_3 we found that a) addition of oxygen significantly decreased the peak voltage and the effective DC-voltage in the HiPIMS-off time; b) the temporal evolution of the dc-bias is altered upon oxygen

gas feed c+d) superposition of RF and HiPIMS allows for stable operation at lower pressures than with HiPIMS only.

We further observed that the deposition rates of the individual processes simply added up to a total deposition rate in the superimposed processes. Similar findings were made for AlN. Results on transparency and hardness will also be presented.

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Accurate Reporting of Time-of-Flight Measurements with Gated Mass Spectrometry

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The quality of high-power impulse magnetron sputtering (HiPIMS) deposited films can often be improved through the effective use of metal-ion synchronization (MIS) [1-3]. However, effective synchronization requires precise measurements of the time-of-flight (ToF) of ions, such that an accelerating bias can be properly synchronized. These measurements are commonly done using time- and energy- resolved mass spectrometry but require calibrations of the transit time of ions inside of the mass spectrometer to accurately report the ToF. The transit time can be calculated by estimating the travel length in varying parts of the spectrometer (e.g. from orifice to detector) and accounting for the interactions of ions with varying electrostatic optics (such as the extractor, energy filter, mass filter, and dynode). The errors associated with these estimations can lead to nonphysical values in a HiPIMS process, such as negative ToFs, or metal ions arriving to the substrate before process gas ions. As a result, many groups emphasize that their calibrations are estimations, or relevant only at sufficiently large time steps [4,5]. **Here we report a practical approach to determine the transit time in the spectrometer experimentally, which was already successfully employed for multiple projects in our group** [2,3]. We use a bipolar HiPIMS power supply to synchronize a gating pulse to the front end of a HiDEN Analytical EQP-300 mass spectrometer (Figure 1). The orifice of the mass-spec (50 μm) was placed at a 12 cm working distance. ToF was then measured by applying a +70 V bias to repel ions, and a 5 µs gating pulse of -30 V to accept them. To prevent interference of the driven front end (kept at +70 V) with the HiPIMS plasma, a grounded shield is placed in front of the mass-spec head with a 2 mm opening. The gate was synchronized to the HiPIMS pulse by providing a trigger signal, and data was collected at 5 μs intervals by adjusting the time delay of this pulse. The time-of-flights of Ar^+, N^+, and Al^+ ions measured in this way are compared to those calculated using mass spectrometry flight tube equations.

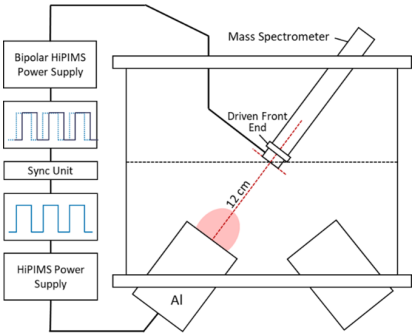

Figure 1: Schematic of sputter system used in ToF measurements. A HiDEN Analytical EQP-300 mass spectrometer was used and its driven front end was synchronized to a 5 μs gating pulse.

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New thermal spray ceramic NiOx(CrOx) targets for sputtering functional layers in low-E glass coatings

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Low-emissivity (low-E) glass plays an important role in keeping buildings and cars sustainable and comfortable in the changing global climate. This type of glass is coated with a multilayer thin film stack that reflects infrared (IR) radiation while remaining transparent to visible light, thus keeping heat in or out without darkening the room. Typically, the IR reflecting layer is a thin (10 nm) silver film, which is in turn supported by various seed, barrier, dielectric, and protective layers. Each layer is commonly prepared by magnetron sputtering from their respective target materials. In this work we will focus on recent improvements in thermal spray ceramic rotatable $NiO_x(CrO_x)$ targets.

Over the past decades, our company has been developing several ceramic alternatives for sputtering processes that have historically used metallic targets in a reactive gas ambient. This is the case of the $NiO_x(CrO_x)$ rotatable targets presented here, as an alternative to Ni(Cr) targets. Sputtering layers from these ceramic targets has many advantages, such as lack of hysteresis, less crosstalk and better control over layer composition. For example, Figure 1 shows no hysteresis, i.e. more stable operating point, for NiO_x targets compared to metallic Ni targets under certain conditions.

Introducing a nickel oxide seed layer has been proven to allow control over the texture of the silver layer[1], while a nickel chromium oxide layer is often used as protective barrier layer in the stack, as it has a high resistance to oxidation[2]. The characteristics of these sputtered layers such as phase formation, optical properties and composition will also be explored in this study.

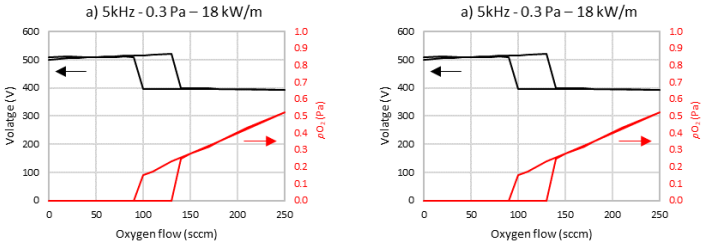

Figure 1: Working curves for AC magnetron sputtering of (a), a metallic Ni target and (b), a ceramic NiOx target.

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Combinatorial Sputter Synthesis and Characterization of (La-)Co-based Thin-Film Spinel and Perovskite Materials Libraries

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Compositionally complex spinels and perovskites are considered potentially stable and active catalysts for electrochemical applications. Through combinatorial synthesis via reactive co-sputtering and characterization of thin-film material libraries, the structural and functional properties along continuous compositional gradients are investigated and correlated. For Co-based systems single phase spinel forms for a wide compositional range.

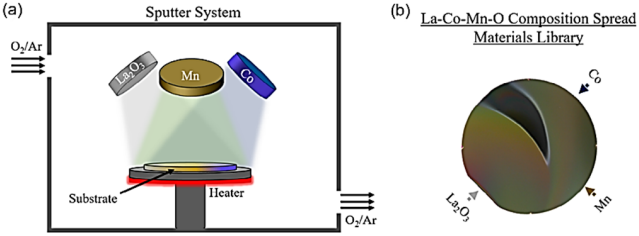

Figure 1: a) Schematic drawing of the reactive sputter process on heated substrate to fabricate the La-Co-Mn-O materials library. b) photo of the materials library, sputter target positions are indicated by arrows and the respective elements [1]

This was investigated for the systems Co-Ni-O and Co-Mn-O, which were later extended to Co-Mn-Ni-O, Co-Mn-Fe-O and Co-Mn-Fe-Ni-O. The fabricated thin-film materials libraries have shown a phase pure spinel structure in nearly every measurement area, except for Mn-rich areas, where also Mn_2O_3 peaks were detected by XRD. Contrary to the expected continuous composition gradient, single-phase regions with homogeneous composition (La_2O_3 or stoichiometric La-perovskite) form during reactive co-sputter deposition of La-based perovskite. This effect of self-organized thin film growth can be used to synthesize

perovskite (ABO_3) thin-film libraries covering a wide range of chemical compositions for the B-site elements in phase-pure perovskite regions. This approach facilitates the discovery and tailoring of chemical compositions for desired functional properties. The synthesized material libraries were screened for their electrocatalytic activity in the oxygen evolution reaction, which led to the identification of a region with interesting catalytic activity in the La-Co-Mn-O system. In-depth characterization of this region revealed a unique columnar-grown microstructure with a large catalytic surface and excellent stability during electrocatalytic measurements. [1]

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NiMo-C coatings synthesised by reactive magnetron sputtering as a catalyst for the hydrogen evolution reaction in an acidic environment

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Leading research on hydrogen production for energy purposes is currently focused on water electrolysis, particularly on developing electrodes that are efficient and durable in both acidic and alkaline environments. In this context, a primary objective is to substitute noble metals with alternative, more cost-effective and accessible materials that exhibit minimal differences in catalytic properties. One potential solution is the utilisation of binary systems that are based on an amorphous carbon matrix and contain either metallic (such as nickel or cobalt) or carbide precipitates (such as MoC, WC, NbC) in the nano-scale range [1,2,3]. The presence of the carbon matrix protects the catalytically active centres against corrosion while simultaneously limiting electrical conductivity and, thus, the effective charge transfer [3].

In the current work, we present the initial investigation into the utilisation of NiMo-C coatings, synthesised via reactive magnetron sputtering of the $Ni_{80}Mo_{20}$ alloy in an argon/acetylene atmosphere, as prospective catalysts for HER (Hydrogen Evolution Reaction) operating in an acidic environment. The evolution of coatings structure, chemical and phase composition with increasing carbon content (from 5 up to 75 at.%) were investigated by X-ray diffraction, X-ray photoelectron spectroscopy and transmission/scanning electron microscopy. These studies were further supplemented by the measurement of electrical conductivity using atomic force microscope working in the conductive mode. The results obtained have shown that microstructure of deposits evolves from nano-crystalline, through amorphous to quasi amorphous

with nano-columnar structure (for the carbon content in the range of 20-75 at.%). The nanostructure observed consists of metallic columns perpendicular to the surface surrounded by amorphous carbon shell. This configuration is anticipated to facilitate high electrical conductivity along the columns. The surface of these coatings is distinguished by the presence of discrete nano-areas with high conductivity, exhibiting a diameter of approximately 20 nm. It is hypothesised that these areas, predominantly comprising nickel, will serve as catalytic centres for hydrogen during the electrolytic decomposition of water.

The electrocatalytic properties of the coatings were evaluated by comparison with the catalytic capacity of platinum and graphite under identical experimental conditions. The overpotential (η) and the Tafel slope, which is a kinetic parameter that indicates the rate of HER, were selected as the primary parameters for the assessment of electrocatalysts' activity. It has been found that the medium carbon coatings show optimal properties in this direction, i.e. high corrosion resistance in an acidic environment and good HER performance. Even at the highest carbon content, the Tafel slope does not increase substantially, what is attributable to the nano-columnar coatings' structure.

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Optical and photochromic properties of yttrium oxyhydride thin films deposited by reactive magnetron sputtering

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Yttrium oxyhydride (YHO) thin films have shown potential as smart materials due to their reversible photochromic effect, achieving a photochromic contrast (ΔT_{vis}) of up to 50% under UV/blue light exposure [1]. However, the precise structure of YHO and the underlying mechanism of photochromism are not yet fully understood. YHO thin films are synthesized through the oxidation of yttrium hydride (YH_2). Parameters such as sputtering pressure and film thickness critically affect thin film optical and photochromic properties, with low sputtering pressures yielding metallic, oxygen-containing YH_2 films, while higher pressures result in transparent, photochromic YHO films.

To further investigate and enhance YHO photochromic performance, double-layer YHO/MoO3 coatings were deposited. Given the mobility of hydrogen in YHO [2], it was hypothesized that combining YHO with MoO_3 could create a synergistic effect, as hydrogen from YHO might intercalate into the MoO_3 layer, partly reducing Mo cations and forming molybdenum bronze. Thin films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), spectroscopic ellipsometry (SE), UV-Vis-NIR spectroscopy, nuclear reaction analysis (NRA), and a custom-built device for measuring photochromic properties.

Results show a photochromic contrast improvement from 40% with YHO alone to 55% for the YHO/MoO_3 thin films, along with an increased coloration rate (see Fig. 1). The photochromic response of the double-layer coatings can be fine-tuned by adjusting the thickness and deposition parameters of MoO_3, such as sputtering pressure and reactive gas flow. Both XPS and NRA indicate the formation of molybdenum

bronze through Mo reduction and an increased hydrogen content in the upper MoO_3 film, respectively. Additionally, scalability through large-area roll-to-roll deposition of YHO was explored, supporting potential applications in smart windows and other optoelectronic devices.

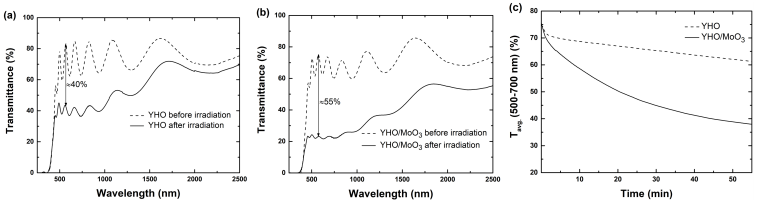

Figure 1: Transmittance spectra (250–2500 nm) of (a) YHO and (b) YHO/MoO_3 coatings before and after 20 hours of UV-A light irradiation, indicating the photochromic contrast values; (c) transmittance decrease in the 500–700 nm range during darkening.

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Embedding Fe Nanoparticles into CrN Films For enhancing thermoelectric properties

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Nanostructured materials and nanocomposites have shown great promise for improving the efficiency of thermoelectric materials. Herein, Fe nanoparticles were embedded into a CrN matrix by combining two physical vapor deposition approaches, namely high-power impulse magnetron sputtering and a nanoparticle gun. The combination of these techniques allowed the formation of nanocomposites in which the Fe nanoparticles remained intact without intermixing with the matrix material. The electrical and thermal transport properties of the nanocomposites were investigated and compared to a monolithic CrN film. The measured thermoelectric properties revealed an increase in the Seebeck coefficient, with a decrease of hall carrier concentration and an increase of the electron mobility which could be explained by energy filtering by internal phases created at the NP/matrix interface. The thermal conductivity of the final nanocomposite was reduced from 4.8 W $m^{-1}K^{-1}$ to a minimum of 3.0 W $m^{-1}K^{-1}$. This study shows prospects for the nanocomposite synthesis process using nanoparticles which can be used beyond improving the thermoelectric properties of coatings and could be extended to variable applications.

Enhancing Charge Transport in Metal (Oxy-)Nitrides for Efficient Solar Fuel Generation

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Solar water splitting offers a promising pathway to generating carbon-neutral solar fuels by converting sunlight directly into chemical energy. A critical challenge in advancing this technology is the scarcity of suitable photoanode materials that meet all three essential requirements: long-term chemical stability, high photocarrier extraction efficiency, and appropriate bandgap. Nitrides and oxynitrides are particularly attractive as photoanode candidates due to their tunable properties based on cation and anion compositions; however, their thin film synthesis remains challenging due to the need for harsh processing conditions.

Among these materials, Ta_3N_5 and $LaTiO_2N$, both with bandgaps of 2.1 eV, have attracted significant attention. Nonetheless, their practical application is constrained by poor charge carrier transport. To address this issue for Ta_3N_5, we employ reactive co-sputtering to synthesize Zr-doped Ta_3N_5 thin films, systematically investigating the influence of Zr incorporation on the compound's crystal structure, defect density, and (opto-)electronic properties. Enhanced hole extraction is specifically targeted through careful control of Zr doping levels and sputtering parameters. For the perovskite-type oxynitride $LaTiO_2N$, the high recombination rate of photoexcited carriers remains a significant challenge. By reducing defect concentrations within $LaTiO_2N$ thin films through optimized reactive sputtering and tailored annealing protocols, we aim to significantly enhance charge transport, pushing towards higher solar-to-hydrogen conversion efficiencies.

Conformal Coverage of Complex Topographies in a Reactive Process using Remote Plasma Sputtering

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With the progression towards higher aspect ratios and finer topographical dimensions in many micro- and nano-systems, it is of increasing importance to be able to conformally deposit thin films onto such structures. Sputtering techniques have been developed to provide such conformal coverage through a combination of coating re-sputtering and ionised physical vapour deposition (IPVD); the latter by use of a secondary plasma source or a pulsed high target power (HiPIMS).

This paper reports on the use of an alternate remote plasma sputtering technique in which a high-density magnetised plasma is used for sputter deposition. The remote plasma sputtering is an inherently continuous IPVD process (not needing a secondary discharge) and through the use of substrate bias the arrival characteristics of sputter flux ions and plasma ions can be controlled [1].

Through the reactive deposition of Al2O3 onto complex structures, scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX), results demonstrate that applying a negative substrate bias during film growth can result in conformal coverage of complex topographies with Al_2O_3 film growth on surfaces obscured from the initial sputter flux.

This work demonstrates that selective deposition can be achieved on trench structures. Deposition of Al_2O_3 onto 5:1 aspect ratio structures has shown that through control of the substrate bias the coating thickness on the vertical and horizontal planes can be varied. For low or zero substrate bias, the majority of the coating will deposit on the horizontal planes. Employing a substrate bias > -267 V results in the majority of the coating being deposited on the side walls. This occurs through the attraction of ionised sputter flux and re-sputtering of the thin film on the horizontal surfaces due to ion bombardment. With further optimisation of the process parameters a uniform film thickness on all surfaces of the high aspect ratio structures can be achieved.

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