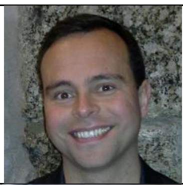
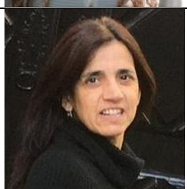
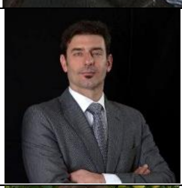
VÁCUO 2021
Workshop

November 9, 2021
Instituto Pedro Nunes, Coimbra

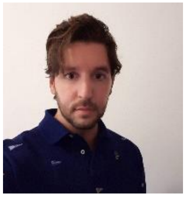
The **VÁCUO 2021 Workshop** aims to bring together national and international experts in a more intimate meeting that promotes discussion on deposition methods, materials synthesis and characterization techniques using vacuum technologies. The 2021 edition will be held on Tuesday November 9 between 2-6 pm, at the Instituto Pedro Nunes Auditorium, R. Pedro Nunes, 3030-199 Coimbra. The topics of the Workshop will focus on vacuum technology and applications, functional coatings for the industry and fundamental research, with applications in the field of tribology, biosensors, electronic devices, solar cells and nanostructured surfaces.

<u>Programme:</u>

14:00–14:05 **Foreword:** Carlos Tavares, *SOPORVAC*

	14:05-14:25 **Cr based sputtered decorative coatings for automotive industry** *Edgar Carneiro* Centre of Physics, University of Minho, Guimarães
	14:25–14:45 **Advanced photoanode for dye-sensitized solar cells prepared by atomic layer deposition of ultrathin TiO_2 shield on mesoporous SiO_2 scaffold** *Fátima Santos* LEPABE, Faculdade de Engenharia, Universidade do Porto
	14:45-15:05 **Production, Characterization and Application of Reactive Multilayer Films** *Ana Sofia Ramos* CEMMPRE, Dept. of Mechanical Engineering, University of Coimbra
	15:05-15:25 **Boosting MEMS production with ALD** *Ernesto Barrera* PhotonExport, Barcelona, Spain
	15:25-15:45 **Electrical and structural changes induced by ion implantation in MoO_3 lamellar crystals** *Daniela Pereira* INESC-MN, Instituto Superior Técnico, Lisboa

COFFEE BREAK
15:45-16:10

	16:10-16:30 **Suspended graphene films with millimeter dimensions transferred by vacuum sublimation** *Alexandre Carvalho* i3N/Aveiro, Departamento de Física, Universidade de Aveiro
	16:30-16:50 **Magnetron sputtered Au:TiO_2 thin films as LSPR-based optical biosensors for DNA samples** *Diogo Costa* Centre of Physics, University of Minho, Guimarães
	16:50-17:10 **Ultrafast nonlinear optics spectroscopy on nanostructures and functionalized surfaces** *Ana Cristina Silva* CeFITeC, Universidade NOVA de Lisboa
	17:10-17:30 **Tribological properties of DLC coatings produced by DOMS in Ar-Ne discharges** *Fábio Ferreira* CEMMPRE, Dept. of Mechanical Engineering, University of Coimbra

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Cr based sputtered decorative coatings for automotive industry

Edgar Carneiro [1*], Nuno M. G. Parreira [1], Todor Vuchkov [2,3], Albano Cavaleiro [2], Jorge Ferreira [4], Martin Andritschky [1] and Sandra Carvalho [2]

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The present work aims to study the impact of O and N addition on Cr sputtered coatings onto plastic (polycarbonate, PC) used on automobile parts, as a promisor alternative for auto parts metallization while eliminating the usage of toxic hexavalent chromium. The coatings were deposited by DC magnetron sputtering from a single pure Cr target in reactive atmosphere (N^2 and/or O^2). The deposition of the coatings was performed keeping the total pressure constant close to 1 Pa by tunning Ar pressure while the reactive gases was added. The target current density was kept at $J_W = 20\ mA \cdot cm^{-2}$. Structural characterization revealed a mixture of α-Cr, δ-Cr, β-Cr2N, CrN crystalline structures and amorphous oxides. Coating hardness ranged from 9 GPa for CrON coating to 15 GPa for CrN coating. All deposited coatings showed a particularly good interface adhesion and adjusting the amount of O and N it was possible to tune the optical properties of the Cr-based coatings as desired. The promising results open future industrialization of sputtered Cr based coatings on automotive industries.

Advanced photoanode for dye-sensitized solar cells prepared by atomic layer deposition of ultrathin TiO_2 shield on mesoporous SiO_2 scaffold

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Nowadays, Dye-sensitized solar cells (DSSC) are emerging as one of the most attractive indoor photovoltaic technology to address the challenge of sustainable powering wireless sensor nodes for IoTs and low power consuming electronics [1]. Light with weak intensity is predominantly absorbed in a thin layer of the photoanode; few photoelectrons are injected into TiO_2 and most of them are captured by trap states[2], slowing electron transport and promoting recombination. Conventional mesoporous TiO_2 photoanode made from nanoparticles display a high density of intraband traps due to high surface area and a large number of grain boundaries[3]. Consequently, developing new strategies for efficient transport of low flows of electrons in the photoanode is imperative for DSSCs operating under weak light.

In this study, a new composited photoanode for DSSCs was prepared by atomic layer deposition (ALD) of a thin TiO_2 layer over a mesoporous SiO_2 scaffold. Silica scaffold of *ca*. 30 μm thickness and average pore sizes of *ca*. 21.0 nm was prepared from terpineol-based paste formulations. The efficiency of the photoanode in DSSC strongly depends on the uniformity of the TiO_2 coating. This was addressed by fine-tuning of the ALD process parameters. Precursors pulsing and carrier gas purging times, a number of ALD cycles were optimized for complete coverage of the inner surface of the SiO_2 scaffold. The titanium isopropoxide (TIP) monolayer chemisorption on silica was found to fit well with the shrinking core model; it allowed to compute the optimum deposition pulse time of TIP to fully coat the silica scaffold [4]. A photoanode composed from ca. 7 nm of TiO_2 shield on mesoporous SiO_2 scaffold allowed to attain ca. 6 % of PCE at 1 Sun. The photoanodes displayed high optical transparency, improved electronic mobility, and reduced back electron recombination, making them promising for implementation in DSSCs operating under weak light conditions.

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Production, Characterization and Application of Reactive Multilayer Films

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Reactive multilayer thin films/foils are composed of tens, hundreds, or thousands of alternating individual layers of reactants having a large negative enthalpy of mixing, which results in highly localized heat release once reaction is initiated. For certain systems and designs these exothermal reactions can become self-sustained, attaining high temperatures [1]. Therefore, multilayers with nanometric period (bilayer thickness) can be used as highly localized heat sources, which make them attractive for several applications. In particular, alternating nanolayers of Al and Ni, that constitute one of the most extensively studied reactive multilayer system, have been successfully used for joining applications [2,3]. For some years now, reactive multilayer foils are being used as local heat sources to melt braze alloys – Reactive Brazing. Taking advantage of the improved diffusivity and reactivity, metallic multilayer films have been used to enhance the diffusion bonding process allowing sound joints to be obtained at less demanding conditions – Reaction Assisted Diffusion Bonding (RABD). Reactive multilayer thin films are deposited by magnetron sputtering directly onto the materials to be joined, ensuring excellent contact between base and filler materials. The possibility of providing an interface with intermediate properties is another advantage of the RABD process, particularly important for dissimilar joining. The use of Al/Ni, Al/Ti and Ni/Ti multilayer thin films allowed sound joints, without pores or (micro) cracks, to be processed at lower bonding temperature and/or time. Although so far reactive multilayers have been mostly used as localized heat sources to promote/enhance joining, they could also have potential for self-healing applications. In this case, the reactive multilayers are deposited onto W wires and the heat released by the multilayers' reaction should promote the melting of a repairing material.

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Boosting MEMS production with ALD

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Main Benefits for using ALD for MEMS:
MEMS (Microelectromechanical systems) are found in day-to-day applications, such as car airbag accelerometers and microphones, or in the background of many invisible yet crucial applications, such as in micro-scale energy harvesting or various pressure sensors. MEMS combined with ALD makes for rugged and functional MEMS technology.

MEMS manufacturing industry utilizes the electrical and mechanical properties of silicon and other materials in various types of devices and applications. ALD is especially well suited for protecting and functionalizing layers in three-dimensional devices and features with high aspect ratios.

There is no general ALD solution for MEMS, as MEMS devices and processes vary a lot.
The goal of this paper is to highlight the possibilities that ALD brings to MEMS design and manufacturing. ALD can bring advantages by Simplifying processes, enabling coatings on challenging structures, improving material performances, lower Temperature depositions, Fine-tuning material properties by mixing and laminating.

MEMS Process steps by ALD:

- Etching – high selectivity masking materials at low T
- Patterning – aspect ratio tuning by sidewall depositions
- Passivation – all around coatings to challenging structures
- Electrical contacts – metal and conductive nitrides
- Functionalization – SAMs and optical coatings
- Sacrificial layers – very well controlled thickness
- Bonding – very smooth films enabling direct bonding
- Encapsulation – good barriers against moisture and chemicals
- Membrane stress tuning – low T films with controlled stress

Results:
Etch mask, selectivity up to 1:100000
Trenches in Si ethced in the ICP afett patterning Al2O3 by lift-off 2 um resist mask. (Courtesy of VTT Finland).

Protection / Passivation of PZT:
ALD thin film suscesfullly impelemnted n MEMS fabrication and usedd to proctec PZT layer during subswquent processing steps and to protect from the ambiant air. (Courtesy of T. Lisec, FhG ISiT, EU project EPAMO)

Coating of challenging structures:
Conductive TiN layer deposited to channel with 200 nm gap have achieved an Aspect ratio of 1:2500 (Test structure by Pillar Hall Courtesy of VTT)

Electrical and structural changes induced by ion implantation in MoO_3 lamellar crystals

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Molybdenum oxide (MoO_3) is a wide band gap semiconductor, with interesting structural properties for several applications such as biosensors, gas sensors, solar cells and lithium ion batteries. Changes in the oxidation state of MoO_3, for example by the creation of oxygen vacancies, can lead to different behaviors, which can range from semiconductor to metallic [1,2]. The possibility of tuning the electrical properties by controlling the concentration of defects, is a useful and valuable tool for design and optimization of new devices.

In this work, we use oxygen ion implantation to modify the electrical and structural properties of orthorhombic MoO_3 lamellar crystals [3,4]. High resolution X-ray diffraction (HRXRD) measurements show that the defects created during implantation induced a significant expansion of the *b* lattice parameter which increases with increasing fluence. These structural changes are accompanied by a high and reproducible increase of the electrical conductivity from fluences above $\sim 1\times 10^{15}$ cm^{-2}, suggesting this effect is related to the most damaged regions in the sample with higher density of oxygen vacancies and/or defect complexes behaving as electron donors or, by the existence of new phases more conductive than the α-MoO_3 orthorhombic phase. Complementary, micro-Raman spectroscopy measurements confirmed both the creation of oxygen vacancies and the existence of new molybdenum suboxide minority phases for fluences above $\sim 5\times 10^{14}$ cm^{-2}.

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Filmes de grafeno suspensos com dimensões milimétricas transferidos por sublimação em vácuo

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Enquanto membrana atomicamente fina, o grafeno potencia aplicações que exijam simultaneamente uma massa reduzida, resistência mecânica e transparência ótica.[1] Alguns exemplos são os sensores de pressão, [2] microfones,[3] janelas transparentes de baixa absorção,[4] até áreas mais exóticas como a sequenciação de DNA.[5] Contudo, a obtenção de membranas suspensas atomicamente finas obriga a um complicado processo de transferência do grafeno a partir do substrato usado no crescimento por deposição química em fase de vapor (CVD), tipicamente cobre, para outro substrato perfurado. Este processo recorre tipicamente à deposição de uma camada de suporte polimérica, com a sua posterior remoção em meio líquido, o que limita o tamanho das suspensões obtidas devido à ação de forças de tensão superficial.[2,6] Neste trabalho, é apresentada uma alternativa de transferência de grafeno baseada num material de suporte sublimável, o antraceno, que possibilitou a obtenção de filmes de grafeno de 10 camadas sobre cavidades circulares com 4 mm de diâmetro, ultrapassando o recorde registado na literatura.[7] Este resultado proporciona um maior rendimento do processo de transferência, viabilizando novas aplicações, tendo sido desenvolvido um microfone que revelou uma excelente resposta específica.

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Magnetron sputtered Au:TiO_2 thin films as LSPR-based optical biosensors for DNA samples

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Biosensors based in Localized Surface Plasmon Resonance (LSPR) reveal potential as label-free optical transducers to produce low-cost and portable biosensors, with real-time monitoring capability. As such, plasmonic nanoparticles (e.g., Au and Ag) are extensively studied to develop highly sensitive and reliable optical sensors. Changing the size, distribution, shape, and composition of the nanoparticles allows tunning the absorption band that arises from the LSPR phenomena. However, LSPR transducers are fundamentally sensitive to differences in the refractive index of the medium surrounding the nanoparticles, thus not selective to a specific analyte. To overcome this, functionalization strategies are applied to immobilize biorecognition elements, such as antibodies, aptamers, DNA or RNA strands, among others, providing selectivity to the optical transducer. Plasmonic bands are also found in magnetron sputtered thin films composed of noble metal nanoparticles dispersed in a dielectric matrix. The optical response of the thin films can be tune by means of post-deposition treatments, which causes the crystallization and growth of the nanoparticles, as well as the surrounding media. In this work, the deposition and post-deposition annealing conditions of plasmonic Au:TiO_2 magnetron sputtered thin films were optimized to obtain the best optical response. This feature was evaluated by measuring the optical response of the thin films in contact with two media with different refractive indexes: deionized water (η= 1.3325 RIU) and a 20% (w/w) sucrose solution (η= 1.3639 RIU). The produced spectra were analyzed using NANOPTICS software and allowed to determine the refractive index sensitivity (RIS) of each thin film sample. Afterwards, relying in the well-known relation between thiol groups and Au nanoparticles, thiolated single-stranded DNA probes were immobilized in the selected thin films. For this study, a microfluidic system and custom-made optical system were designed and employed. Results show the successful functionalization of the thin film with the SH-DNA probe and detection of the complementary DNA strand.

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Ultrafast nonlinear optics spectroscopy on nanostructures and functionalized surfaces

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Ultrafast nonlinear optical spectroscopies have become important tools in surface and interface analysis where they can provide information on optical, electronic, magnetic and vibronic properties, reaching a state of very high surface sensitivity. Nanostructures with their large fraction of surface to volume are interesting subjects for studies with interface sensitive optical techniques. The basic principles of optical Second-harmonic generation (SHG) are reviewed. Applications of optical SHG and SHG spectroscopy to characterization of nanostructures, and semiconductors surfaces and interfaces, nano crystals, metallic quantum wells, and 2D structures, will be presented.

Tribological properties of DLC coatings produced by DOMS in Ar-Ne discharges

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Diamond-like carbon (DLC) films have been the paramount solution to reduce friction during operation of internal combustion engines for the automotive industry because they offer excellent surface properties, including very low coefficient of friction (<0.2) and wear rate (<10^{-16} m^3/Nm), even under high load/pressure. In previous work, the authors have shown that a recently developed variant of HiPIMS, called deep oscillation magnetron sputtering (DOMS), was suitable for the deposition of hydrogen-free DLC coatings with structural properties comparable to those of the DLC films deposited by another state of the art deposition processes. In this work, the tribological properties of the DLC films deposited by DOMS with increasing bias voltage and different Ar/Ne discharge plasmas are characterized and correlated with the hardness of the films in order to identify the wear mechanisms. It is shown that increasing the substrate bias changed the wear mechanisms from layer-by-layer removal at a lower bias to a mechanism consisting in the successive formation of fine particles which agglomerate at the outer parts of the wear track at higher substrate bias. On the other hand, adding Ne to the discharge gas resulted in a significant decrease of the specific wear rate, reaching a minimum value of 4 x10^{-17} m^3/Nm, while the friction coefficient of the films remained close to 0.15, i.e., within the range of typical values for DLC films tested in relatively humid conditions. Thus the tribological properties of the DLC films deposited in this work are very interesting for many applications in the automotive industry, such as for the replacement of the CrN coatings nowadays standardly deposited onto the piston rings of internal combustion engines.