

Plasma Surfaces and Thin Films:

Thin Film Coatings for Electro-optics

10 September 2024

Manchester Metropolitan University,
Manchester, UK



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The effect of different photocatalysts on nitrogen and oxygen plasma chemistry in a dielectric barrier discharge plasma

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The use of photocatalytic materials in plasma systems has the potential to enhance the selectivity and yield of desired products. This work presents the diverse effect of bismuth vanadate, BiVO₄, zinc oxide, ZnO, and Titanium dioxide, TiO₂ on nitrogen and oxygen plasma chemistry and the formation of end products. Photocatalysts were produced as thin film coatings using reactive magnetron sputtering on the dielectric alumina plates, Al₂O₃, and barium titanate beads, BaTiO₃, which were then incorporated within a dielectric barrier discharge (DBD) plasma reactor. The surface properties of the catalysts were examined using state-of-the-art surface analytical equipment including Scanning Electron Microscopy, X-ray Photoelectron Spectroscopy, Raman Spectroscopy and Atomic Force Microscopy to assess the topography, chemical composition, and crystalline structure of the catalyst coating [1-3]. Ex-situ FTIR analyses of end products were carried out to qualify and quantify the generated end products. Oxygen concentration was varied between 0 and 100 %, while an applied voltage of around 6 kV and a total flow rate of 1 slm were used. Results show that ZnO coatings resulted in the formation of nitrogen dioxide, NO₂, and nitrous oxide N₂O. While with annealed BiVO₄, ozone, O₃, and N₂O were the main end products. Annealed TiO₂ resulted in the formation of O₃ and, Dinitrogen pentoxide, N₂O₅. In the case of no coating, the main end products were O₃, N₂O₅, NO₂ and N₂O. These results prove the importance of the properties of the plasma facing surface on plasma chemistry and the formation of end products in the DBD. They also demonstrate the potential of tailoring plasma processes to suit the requirement of different applications.

[1] S.C. Capp et.al, J. Environ. Chem. Eng, 9, 106046 (2021).[2] Z. Abd-Allah et.al Plasma Processes and Polymers, 13, 649-653(2016).[3] D.A.G. Sawtell et.al, Plasma Processes and Polymers, 15, 1700051 (2018).

Plasmonically Activated Antimicrobial Surfaces Based on Transition Metal Nitride Films Deposited by High Power Impulse Magnetron Sputtering

Ehiasarian A

TiN and NbN are some of the most plasmonically active and environmentally robust thin film materials. Their plasmonic activity promotes a high photocatalytic antimicrobial function, however thin films suffer from high optical losses due to the segregation of O impurities at (111) – oriented grain boundaries. Densification through constant-current High Power Impulse Magnetron Sputtering (HIPIMS) deposition can improve the optical properties, however the influence of film texture and plasma chemistry are not known. In TiN, time-resolved optical emission and energy resolved mass spectroscopy revealed a gas-rich ignition phase which developed into a metal-rich phase where the metal component is continuously pumped while the plasma density remained constant. A steady metal-dominated state was reached for pulse durations above 100 μ s. Films deposited during the ignition stage had a strong (111) texture while those deposited during the “pumping” and “steady state” regimes contained a strong (200) component. The high momentum of Nb ions promoted the (200) component as the preferred orientation and increased the grain size of the films. Patterning by colloidal masking and reactive ion etching produced nano pillars with diameters of 10s of nm.

All films exhibited a strong plasmonic behaviour with low optical losses, as determined from the imaginary component of the electric permittivity obtained from ellipsometry, and a resonant wavelength in the infrared spectrum. Significant light activation of phonons and production of hot electrons was detected by Raman spectroscopy in patterned surfaces compared to the flat as-deposited films.

Nano-patterned surfaces under light activation achieved moderate antimicrobial kill rate of 1-1.6 log for *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) and were superior to benchmark coatings of TiCN, CrCN, TiO_x and TiO_xN_y as well as patterned Si.

Plasma-enhanced pulsed laser deposition of photocatalytic thin films

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Plasma-enhanced pulsed laser deposition (PE-PLD) is a novel thin film deposition method which employs RF plasmas and laser-ablated plasma plumes to produce semiconductor thin films. A high-powered, pulsed laser ablates material from a metal target into a plasma plume, which interacts with non-metal ICP species to form metal compound material that deposits onto a substrate. PE-PLD has been shown to produce high-quality metal oxide thin films with many applications, including photocatalysts which use solar energy to produce hydrogen fuel from water splitting. PE-PLD remains an active area of research, particularly understanding its underlying plasma physics and chemistry, such that thin films can be created according to specific criteria rather than empirical observation. This talk centres around the suitability of PE-PLD in producing metal oxynitride thin films for photocatalysis.

From modelling the laser ablation of different photocatalytic metals using the code POLLUX, the electron temperature and mass density of the plasma plume both increased with the atomic number of the material, whilst the mass density of the material had no observed effect on the electron temperature or particle density of the plume.

From measuring the absolute ground-state densities of atomic O and N plasma species for a range of low-pressure O₂/N₂ plasma mixtures using the TALIF diagnostic, the relative flow input of O₂ and N₂ was shown to be the parameter with the greatest control over the O:N atomic density ratio, allowing it to change by up to a factor of 100.

The structure and chemical composition of deposited metal oxynitride thin films were analysed with different diagnostics, showing a consistent lack of nitrogen present on the films and lack of visible light absorption, highlighting many areas of improvement for PE-PLD in producing oxynitride films, such as understanding interactions between O₂/N₂ plasma species and their effect on deposition.

Deposition of Functional Films onto Powder Substrates via Magnetron Sputtering

Kelly P

Fine particulates, ranging in size from sub-micron to 100s of microns, are ubiquitous in manufacturing and processing, and in many cases, it is highly desirable to apply a functional film to the particles to modify their properties and performance. Magnetron sputtering is a well-established technique for the deposition of high quality metallic and ceramic coatings onto a wide range of substrate materials and forms. It is not generally suitable, though, for the coating of such particulates. This paper, however, describes the use of an oscillating bowl feeder mechanism to manipulate particulates under two co-planar magnetrons, such that uniform coverage of the particles is achieved. Coatings of Ti, TiO₂, Sn, SnO₂, MoS₂, Pd and Ni have been all deposited by this technique and the resulting particles have been characterised by SEM, EDX and XRD. Specific functional properties, such as catalytic efficiency or drug release rate have been investigated and are reported.

Atmospheric Pressure Plasma Synthesis of Individual Metal Atoms

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Introducing low-dimensional materials has revolutionized materials science, enabling the development of materials with highly desirable properties. However, reducing dimensionality to synthesize atomic-scale materials is extremely challenging, particularly at atmospheric pressure. In this study, we develop a non-equilibrium plasma method at atmospheric pressure to synthesize individual atoms. Atomic-scale electron microscopy confirms the deposition of individual bismuth (Bi) atoms on graphene oxide layers. Using Optical Emission Spectroscopy (OES), we demonstrate that the interaction between plasma-generated species and a metal wire can lead to the formation of individual atoms and clusters. These results suggest that atmospheric pressure plasma is a straightforward and effective method for synthesizing individual atoms, with potential applications in catalysis and quantum sensing.

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 150mm silicon wafers using a 300mm 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 in the final coating.