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Radiation Physics



“Alexandru Ioan
Cuza” University



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Conference

Invited lectures

Plasma technologies in Industry 4.0: Opportunities for Plasma Scientists

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Industry 4.0 (often referred to as the “fourth industrial revolution”) refers to the concept of factories in which machines are augmented with wireless connectivity and sensors, but in a broader sense also to the development of production processes that are compatible with the major concept. The compatibility is achievable in practical cases only where the processes are reliable enough and any deviations from prescribed parameters manageable. Furthermore, the applied technological processes should be compatible with major trends and policies such as environmental sustainability, low carbon fingerprint etc. Plasma technologies are definitely among technologies that fulfil such trends. They are ecologically benign and enable appropriate surface finish by treatment of products in the continuous mode. Several examples of plasma technologies will be presented. The surface finish of many products is not adequate, so it has to be tailored to meet specific needs. Plasma technologies allow for an almost arbitrary surface finish of virtually any material. The key surface modifications are nano-structuring and functionalization, which is achievable by using plasma created in different gases and by different discharges. In order to achieve the goals of Industry 4.0, the classical approach is insufficient, so plasma reactors should be equipped with sensors of plasma parameters, particularly meters of reactive particles fluxes. Even more challenging is the technology of selective etching of polymer composites where both plasma parameters and products’ temperature should be kept in the range of prescribed values in order to benefit from the optimal surface finish. Finally, it will be shown that charged particles play a minor role in many advanced technologies, where the main reactants are molecular fragments, including atoms. Synergy with UV/VUV radiation will be stressed, too.

Growth and characterization of the magnesium based thin films using Laser-induced Thermionic Vacuum Arc technology

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Nowadays, the great challenge is coming from the technological transfer for advanced nanocomposites growth for the development of innovative production technologies, as well as the variation of the nature of the components, in order to obtain multifunctional materials with given unique characteristics.

Specifically, magnesium-based nanocomposites, owing to their remarkable properties of the coating surfaces such as wear resistance, roughness, low friction coefficients are worth to be investigated in different combination and forms. Multi-component thin films as well as single thin films were deposited during the last years using Thermionic Vacuum Arc (TVA) technology. TVA is a versatile deposition method combining anodic arc and electron gun systems for the growth of thin films. [1]

The aim of this work is to present the results of the magnesium based nanostructures: MgX – X=Ag;Zn:Ti and pure magnesium deposited by a new concept, using at maximum the offered performances by TVA technology and by Laser-beam effect, namely Laser-induced Thermionic Vacuum Arc (LTVA). The plasma/surface interactions combined with photonic processes and growth mechanisms are relevant for finding the best combination of the magnesium based complex nanocomposites with tailored properties. The focus is related mainly to the TVA stability at the transition from electron bombardment to the TVA arc plasma ignition and running simultaneously for both systems (arc discharge and laser) and their synergistic effects in a special configuration.

The surface morphology, structure, wettability and mechanical properties of the deposited Mg-based thin films were investigated using transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), and free surface energy (FSE) by surface energy evaluation system, accompanied by the nanoindenter equipped with Berkovich indenter, respectively. The results revealed that the grains in the Mg film deposited with the LTVA system were smaller than formed in the TVA; a promising result for future application of the LTVA. Selected area electron diffraction (SAED) technique provided the crystalline characteristics of the second element in magnesium matrix, adding interesting information on the binary nanostructures.

[1] R. Vladoiu, M. Tichy, A. Mandes, V. Dinca-Balan, P. Kudrna, *Coatings*, 10 (2020) 211.

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Romanian participation in EUROfusion research

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**See the author list in Joffrin E et al., Overview of the JET preparation for deuterium-tritium operation with the ITER likewall, 2019 Nuclear Fusion 59 112021*

The ‘European Consortium for the Development of Fusion Energy’ (<https://www.eurofusion.org/>), is Europe’s fusion research laboratories consortium. EUROfusion funds research activities in accordance with the Roadmap to the Realization of Fusion Energy (<https://www.euro-fusion.org/wpcms/wp-content/uploads/2013/01/JG12.356-web.pdf>) with the aim of achieving fusion electricity. The main objectives are related to the preparation for the experiments on ITER (www.iter.org) and to the development of concepts for the DEMO fusion power demonstration plant (<https://www.euro-fusion.org/newsletter/demo-and-the-road-to-fusion-power/>).

Romanian participation in EUROfusion includes the nuclear fusion research activities carried out by several research institutes and universities, in various work packages.

Theoretical and modelling research activity has been focused on various topics, such as: i) modelling Wall Touching Kink Modes and Vertical Displacement Events and implementation into the JOEK-STARWALL code, ii) development of an almost fully analytic, dynamic, interpretative, multi-mode model describing the evolution of the NTMs, iii) assessing the importance of the new concept of hidden drifts on W transport, iv) contribution to the extending of the atomic and nuclear data in support of AMNS library.

The participation in the plasma facing components topics mainly addresses to the development of advanced techniques for fuel retention assessment, tools for removing fuel and dust from the wall surface and mixed material deposition. The research activities related to materials development for DEMO are related to the development of various interface materials and joining technologies for the divertor heat sink components, as well as for thermo-physical properties characterizations.

The work on developing superconducting magnets comprises two main research directions. The possibility of incorporating high temperature superconducting tapes in magnets for fusion applications has been explored by the enhancement of the electrical transport properties of YBCO-based high temperature superconducting tapes. Advanced X-ray micro-tomography examination analysis has been performed to support the development of a more complex model of multifilamentary superconducting wires.

The Romanian team has coordinated the activities for developing of a new tangential gamma-ray spectrometer for fast ion measurements in deuterium and deuterium–tritium plasmas of JET. Design and construction activities are performed for the development of the JT-60 Thomson Scattering diagnostics and Vacuum Ultra-Violet spectrometer diagnostics. Various numerical tools for plasma diagnostics, comprising gamma, neutron and hard X-ray tomography, bolometry, JET fast-visible camera image processing and time series methods for assessing the efficacy of instability pacing techniques, have been also developed.

Efficiency of the secondary electron emission in magnetized discharges

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The secondary electron emission (SEE) process is a key factor at plasma-surface interface. It is essential for the optimal operation of plasma-based applications, from laboratory experiments to fusion reactors or high-energy accelerators. It can be an advantage or an issue. In magnetized discharges, the secondary electrons are forced to move on helical trajectories, some of them being returned to the emissive surface. Thus, the efficiency of the SEE process may be severely reduced by the presence of the magnetic field. In particular configurations, the SEE is completely suppressed.

A three-dimensional Monte Carlo (MC) simulation method has been used to study the role of a magnetic field B on the SEE process, including a set of other relevant parameters: the magnetic field tilt with respect to surface normal, θ_B , the electron reflection coefficient on the surface, R , the most probable speed of the secondary electrons, v_S , and the electric field in front of the surface, E . The secondary electrons have been emitted under a cosine angular distribution, with a maxwellian distribution in energy. They were moving in a low background pressure, on collisionless trajectories.

As a result of the numerical simulations, an analytical solution has been proposed for the effective SEE yield [1], $\gamma_{eff} = \gamma \frac{\cos\theta_{BE}}{1-R(1-\cos\theta_{BE})}$, where γ is the SEE yield without magnetic field, $\theta_{BE} = \theta_B(1 - A\cos\theta_B)$, and $A = \frac{2E}{Bv_S}$. A very good agreement has been found between the MC simulations and the analytical formula (Fig. 1).

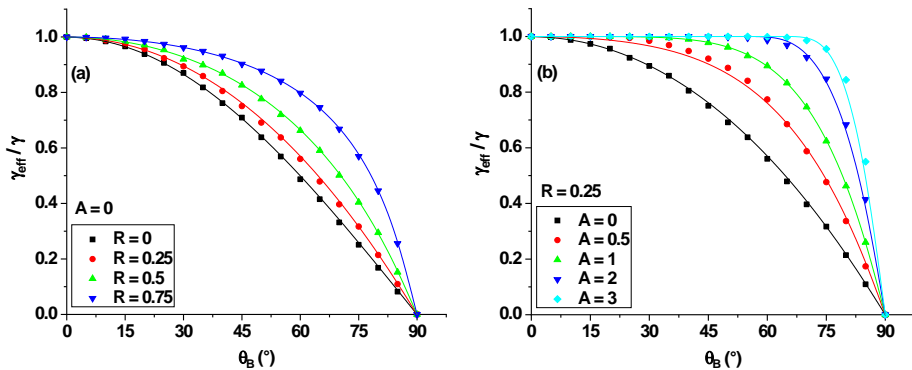


Fig. 1. The effective SEE yield as a function of the magnetic field tilt θ_B , for different values of the reflection coefficient R (a) and of the parameter A (b). Symbols correspond to MC simulations, full lines correspond to the analytical solution.

[1] C. Costin, Scientific Reports **11** (2021) 1874

Keywords: secondary electron emission, magnetized plasma, Monte Carlo simulation, analytical solution

Advanced diagnostics, thermal coupling and fundamental studies of atmospheric pressure discharges

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Non-equilibrium plasmas such as nanosecond or microwave discharges at atmospheric pressure have demonstrated high reactivity and important potential of applications. Understanding their energy and thermal coupling, kinetics and transport phenomena relies on advanced diagnostics and is essential for the optimization and development of plasma technologies.

Diagnosing the confined microwave discharges generated in micro-capillaries or jet configurations and the transient nanosecond discharges at atmospheric pressure is particularly challenging. With the plasma radius below mm, exhibiting large collisional broadening and dominant quenching phenomena, the sensitivity and selectivity of spectroscopic methods are severely reduced, and the uncertainties of techniques such as laser induced fluorescence are significantly increased. Moreover, the presence of strong gradients of temperature, pressure and species, which is accompanied by fast kinetics and complex transient dynamics requires highly resolved spatial and temporal diagnostics.

The energy coupling in these discharges is also complex. It is time dependent and exhibits intricate branching in the plasma chemistry, gas heating, thermal radiation, conductive and convective heat transfers, EM leak and light generation.

Here, examples of advanced diagnostics used for investigations of the power balance, thermal coupling and fundamental mechanisms in these confined high-pressure discharges will be presented. The use of high intensity light sources, such as ultrafast lasers with their inherent Stark detuning and Rabi oscillations, and the diagnostic advantages such as quench-free and photolytic-free regimes will be discussed. The heat transfer control and the possibility to tailor key discharge properties in capillary microwave plasmas will be shown.

Keywords: thermal coupling, power balance, laser diagnostics, nanosecond discharges, microwave capillary discharge

Characteristics of surface-wave microwave discharges and their application possibilities

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The surface-wave microwave discharge generated with the help of a surfatron wave launcher is a very flexible system, since discharge can be ignited in a wide pressure range from mbar up to atmospheric pressure. Both at low and atmospheric pressures, the species concentrations in the discharge and afterglow region can be easily tuned with the system parameters, initial gas mixture compositions and flow rate. In the case of low pressure, a flowing Ar/N₂-O₂ surface-wave afterglow is particularly well adapted to provide a low gas temperature plasma free of charged species (avoiding ion bombardment damage of biological material), and rich in reactive oxygen and nitrogen species (e.g. O and/or N atoms, O₂(a) and NO molecules) and a high intensity of UV radiation finely tuned with the help of the initial gas mixture composition and the gas pressure [1]. These characteristics make this system favourable for surface functionalization, structuring and sterilization, nanoparticle production and the study of plasma chemical processes [2].

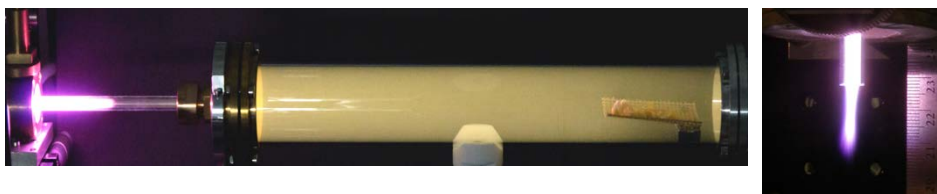


Figure. Surface-wave microwave discharge at low and atmospheric pressure ignited in a 5 mm and 4 mm I.D. quartz tube, respectively, in Ar/N₂-O₂ mixture using 1 slm flow rate. At low pressure the discharge is connected to a 44 mm I.D. afterglow tube.

At atmospheric pressure the system is well adapted for liquid treatments, since the plasma plume that is in contact with the liquid can be easily tuned with the initial gas mixture composition and the treatment distance. By tuning the plasma composition, i.e. the concentration of electrons and nitrogen and oxygen content species at the plasma-liquid interface, the creation of NO₂⁻, NO₃⁻ and H₂O₂ species in the liquid can be controlled, and plasma-activated liquids with very different compositions can be produced [3].

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Keywords: surface-wave microwave discharge, afterglow, plasma-activated liquid

Preheating and quenching in CO₂ microwave plasma reactors

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With increasing global interest in renewable energy technology, storage of electrical energy has become particularly relevant. The chemical industry must likewise be transformed to rely only on sustainably generated electricity to convert common molecules such as CO₂ into chemical building blocks or fuels. Plasma-driven conversion has great potential for upscaling, since no rare materials are required in construction or upkeep, while also being compatible with the intermittent nature of sustainable electricity sources. Using plasma powered by microwaves, high power densities, and thus high throughputs, can be obtained. Moreover, since the plasma is essentially electrode-less, degradation of the reactor over time is virtually non-existent.

Typical energy efficiencies for CO formation obtained in CO₂ microwave plasma reactors approach 40%, with the remaining 60% of the input power going towards heating of the gas [1]. In theory, energy efficiencies of ~ 50% should be attainable for the thermal conversion of CO₂, and > 50% if non-thermal conversion pathways can be accessed [2]. Moreover, optimal efficiencies are currently found at reduced pressures of ~ 150 mbar, demanding additional power input for expansion of the gas. For industrial application of CO₂ microwave plasma the ability to operate at atmospheric, or supra-atmospheric, pressure would be beneficial since it would reduce complexity of the system and improve the coupling with subsequent processing steps, such as gas separation of CO, O₂ and CO₂ from the output gas. In this contribution, we will look at recent theoretical and experimental work performed at DIFFER on the topic of heat recycling and rapid quenching of output gas from the reactor, with the aim of maximizing energy efficiency and moving the operational window towards higher pressures. To this end, a new technique for measuring gas temperatures in a CO₂/CO/O₂/O mixture will also be discussed [3].

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Atmospheric Plasma Immobilization of Antimicrobial Zeolite Loaded Silver Nanoparticles on Medical Textiles

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1. Introduction

Nosocomial infections, in particular problematic chronic wounds, are a ubiquitous general concern. This apprehension was acuted by the prevalence of multidrug resistant bacteria and emergence of Pandemics. Therefore, the development of novel and highly effective antimicrobial wound dressing comprising marginal or absent cytotoxicity to the patient is crucial. Plasma plays a key role in improving the functionalization of surfaces, in particular of textiles [1]. Thus, in this work we used atmospheric double dielectric discharge (DBD) plasma activated woven polyester (PES) functionalized with silver nanoparticles (AgNPs), enzymes as antimicrobial agents, immobilized using mordenite (MOR) zeolites and polysaccharide-based matrixes to mitigate cytotoxicity.

2. Methodology and results

MOR was used with the objective of improving the concentration, stability, and immobilization efficiency of AgNPs and enzymes in the functionalized fabric. Therefore, a solution combining the AgNPs, and/or antimicrobial enzymes was prepared. Afterwards, this solution was mixed with a polysaccharide matrix, consisting of alginate or chitosan. Woven PES surface was activated using DBD and was impregnated with the prepared formulation. The antimicrobial activity of the functionalized fabrics was characterized using bacteria commonly associated to nosocomial infections as well as a virus that is a potential surrogate of severe acute respiratory coronavirus 2 (SARS-COV-2). The antimicrobial tests performed comprised the evaluation of antimicrobial efficacy when in contact with the composites during 1 to 2 hours, by adapting the following standards: AATCC TM100-100 and ISO18184. The microorganisms used were *S. aureus*, *E. coli*, and bacteriophage MS2. The formulated composites containing alginate as matrix displayed a high antibacterial activity (higher than 99.999 %) which was stable for over than 15 days of storage. However, it did not exhibit any antiviral activity. The alginate composites also did not hinder the activity of protease, which may have an important antifouling activity. Whereas, the composites containing chitosan exhibited a highly effective antimicrobial activity against the bacteria and the virus (higher than 99.9999 %) when zeolite was present in the formulation.

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Keywords: Atmospheric plasma, textile, nanoparticles, antimicrobial, zeolite, enzym

Nitrogen fixation by pulsed electrical discharges: from backgrounds to application

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Non-thermal plasmas operating at high pressure attracts increasing attention for a variety of fields as a green and sustainable approach in comparison with conventional chemical processes. Recently, atmospheric nitrogen conversion or nitrogen fixation (NF) has got renovate attention. The aim of NF is to overcome limitations of current industrial processes, their high-energy consumption and environmental impact. Plasma-based NF is a promising approach to convert atmospheric nitrogen into simple nitrogen compounds such as ammonia or nitrogen oxide. The N₂ fixation by electrical discharge into fertilizers or other chemical valuable products is of key importance for the industry as an alternative route to the Haber-Bosch process. In plasma-assisted nitrogen fixation, the synthesis can be conducted at the ambient atmosphere using nitrogen from the air with or without a catalyst. The obvious challenge on the way of high-energy efficiency is the energy-expensive process of molecular nitrogen dissociation. Among different mechanisms of dissociation, a vibrational excitation of the molecular nitrogen followed by dissociation can provide high energy efficiency required for NF.

Here we overview the kinetics of nitrogen fixation in ns-duration pulsed discharge in presence of plasma/liquid interface. An atmospheric pressure ns-pulsed discharge of 10 ns duration with a liquid electrode is investigated as a source of non-thermal plasma operating in air and capable of effective N₂ fixation into valuable products. The kinetics of the O atoms, OH and NO radicals production and recombination are studied by the laser-induced fluorescence spectroscopy and methods of time-resolved optical emission spectroscopy. The effect of the liquid electrode on the discharge properties is deeply analyzed. The mechanisms of the discharge sustaining and pathways of nitrogen fixation in NO_x products are revealed. The effect of the discharge frequency of 10 Hz in a single-mode and a burst mode at a repetition frequency of 100 kHz is also investigated in terms of the efficiency of NO products formation. Based on time and space resolved absolute density determination, the role of OH radicals in NO production is determined and discussed giving insights on future of nitrogen fixation by pulsed plasmas.

Plasma degradation of antibiotic contaminants in water

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Water contamination with antibiotic compounds raises great concern both due to their detrimental effects on various aquatic species [1, 2] and, even more importantly, due to their already established role in promoting and spreading antimicrobial resistance [3, 4]. Limited efficiency of conventional water treatment methods for the removal of antibiotic pollutants has triggered the research on alternative solutions, i.e. the advanced oxidation processes [5,6]. Among them, non-thermal plasma is undeniably one of the most promising approaches, which has already demonstrated its potential for the removal of a number of antibiotics belonging to various classes [7].

The talk will be focused on several key performance indicators: the antibiotics removal, the mineralization, the energy cost and the toxicity of the treated solution. One of the greatest challenges of plasma treatment is related to efficiency enhancement, which is largely influenced by the plasma reactor design and by the experimental conditions, i.e. gas and solution composition, input energy, solution properties, eventual addition of catalysts etc. The effect of these parameters will be addressed in some detail.

The degradation mechanism is another essential issue that will be discussed. Although it is generally agreed that plasma degradation of antibiotics is dominated by hydroxyl radical attack, evidence of contributions from other plasma-generated species is available. The detection of reaction products and assessment of degradation pathways are equally important, in view of the safety of the treated effluent. Determining the toxicity of plasma-treated water, discriminating between toxicity induced by the degradation products and eventual effects of the plasma-produced oxidants, as well as concluding whether the treated solutions retain their biological activity still represent key research challenges. Besides improving the fundamental knowledge, research into these aspects might also offer practical benefits, related to tuning plasma parameters in the hope of a more efficient, safe and selective water treatment process.

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Oral presentations

DBD plasma treatment of polymeric foils used for flexible printed electronic circuits

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1. Introduction

The printed electronic circuits represent a response to the current society needs in terms of adaptable electronics being used in different domains such consumer electronics, automotive industry, healthcare or robotics.

The substrate is often made of polymers, which, in the majority of cases, are non-polar materials that exhibited low surface energy, and consequently in order to improve the adhesion and/or wetting of a layer at its surface supplementary manufacturing processes have to be employed. The low adhesion of ink at the polymer's surface is one of the main technological drawback in printing technologies. Among other methods (ultrasonic baths, vacuum coating, low-pressure plasma), the DBD plasma treatment applied in atmospheric conditions represents a convenient and relatively easy to apply method.

2. Experimental setup and results

The surfaces of different polymeric (such PET, BOPET or polycarbonate) foils have been pre-treated in atmospheric conditions using a DBD reactor following the immediate and long storage effect of plasma on the surface. The DBD reactor consist of two coplanar circular stainless-steel electrodes having a diameter of 60 mm and a distance of 7 mm between them. The dielectric consist in a 3 mm glass sheet on which the foil is placed while the system is supplied using a 40W, 10 kV, 10 kHz power supply. The foils have been treated for different periods, ranging from 0.2 to 30 s. The effects of the applied plasma treatment have been evaluated considering the surface properties such wettability, surface tension or adhesion forces.

The wettability has been evaluated based on the static water contact angle (WCA) measured for different treatment times, immediately after the treatment as in terms of treatment persistence for up to 100 h of storage after the treatment. For all the tested substrates, there is a decrease of WCA for all the considered treatment times. The WCA is increasing starting with the first hours after the treatment, without reaching the initial WCA of the untreated foil, not even after 100 h of storage.

The surface tension has been evaluated using dedicated test inks. The results showed an increase of the surface tension for all the tested foils, from initial values of under 38 or 36 mN/m to over 44 mN/m, even for short treating times of 0.2 ms. The surface tensions remains at the same values obtained immediately after the treatment, even for storage times of 100 h.

The adhesion forces have also been evaluated using atomic force microscopy (AFM). The results obtained for two different foils (one made of PET and one of polycarbonate) indicate a dependence between the treatment time and the adhesion force.

The adhesion of conductive ink to the polymer substrate has been evaluated considering hardness standardise tests. The results showed an improvement of the ink adhesion to the surface for the foils that have not been previously heat-treated.

Atmospheric plasma fluoridation for application in dentistry

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Dental caries is a chronic, multifactorial disease, which involves tooth demineralization caused by biofilm-produced organic acids. Preventive care (e.g., fluoride-containing compounds and water and salt fluoridation) showed good results regarding the management of tooth decay. Fluoride (F⁻) enhances calcium and phosphate diffusion into the tooth, reducing the enamel demineralization and increasing the remineralization process [1,2].

Despite these advantages, when ingested in high concentrations (e.g., water, toothpaste, fluoride supplements), chronic fluoride toxicity was observed. Therefore, considering the drawbacks of fluoride intake [3,4], and the need of regularly topical fluoride applications to ensure an optimal amount of F⁻ [5], new approaches are required.

In this contribution we report atmospheric pressure plasma treatments adapted for the improvement of F⁻ retention into hydroxyapatite samples. The fluoridation experiments were conducted using fluoride-containing compounds or Ar/N₂/SF₆ treatments, applied on plasma-activated surfaces (Ar or Ar/N₂). SF₆ plasma treatments were performed in a controlled environment, while the other experiments were conducted in open air. The samples were treated using two plasma sources: a Dielectric Barrier Discharge (DBD) source and a plasma jet/torch, using a radiofrequency power supply (13.56 MHz). The gas flows, the powers, and the source-sample distances were adjusted depending on the plasma source, with higher values for the plasma torch and lower values for the DBD source.

Scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and X-ray Photoelectron Spectroscopy (XPS) were used to evaluate the surface morphology and the chemical composition. The results indicate that plasma treatments improved F⁻ retention, with the highest relative atomic percentage of 12 % (XPS) for SF₆ treatments. Moreover, biological tests proved the antibacterial effects of the treated samples, which is relevant for dental applications.

Keywords: plasma jet, dental applications, fluoridation, atmospheric pressure plasma;

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Modification of mechanical properties of polydimethylsiloxane surface by low-pressure plasma treatment

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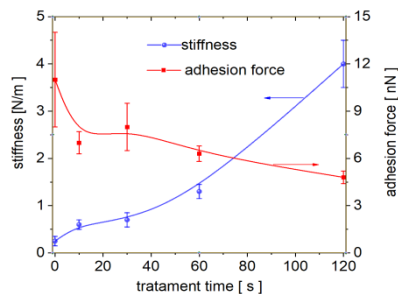
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1. General

Polydimethylsiloxane (PDMS) is an elastomer material often used for fabrication of nano- or micro- structured surfaces for biosensors, micro fluid devices or functional surfaces inspired by nature [1]. PDMS with variable surface stiffness can be obtained by using various values for mixing ratio with the curing agent, curing temperature and curing time. All these parameters control the number of linking sites between PDMS monomers, thus determining friction, adhesion and elasticity properties of the material surface [2].

In the present work we study the effect of plasma treatment on the elastic modulus, surface adhesion and surface friction of a PDMS moulds obtained by curing PDMS mixed with curing agent in ratio of 10:1 for 24 hours at 60°C. The PDMS samples (1 cm × 1 cm × 1 mm) were treated by negative glow plasma of a d.c. glow discharge in Ar at low pressure (0.2 Torr) for various durations (from 10 s to 2 minutes). The modification of PDMS surface properties induced by plasma was investigated by atomic force microscopy (AFM) technique. The mechanical stiffness and adhesion of PDMS surfaces was characterized by AFM indentation performed with a silicon nitride AFM probe with a sharpened tip, while the frictional property was investigated by lateral force microscopy (LFM) technique. The as-fabricated PDMS samples exhibited a very soft and sticky surface due to many surface dangling polymer chains. The plasma treatment determines a strong increase of surface stiffness (Young modulus) and a decrease of adhesion force (Fig. 1). This is an effect of a drastic increase of density of polymer linking sites generated by plasma radicals on the PDMS surface. As result of plasma treatment, the friction behavior of PDMS is also changed drastically. The topography and friction images showed that plasma treatment determines increase of surface roughness and friction nonuniformity in first 30s of treatment followed by a decrease in roughness and increase in friction uniformity afterwards.

Fig. 1 Evolution of PDMS surface stiffness and adhesion during plasma treatment



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Computer-aided analysis of the theories of stochastic transport

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The complex physics of turbulence has many aspects that are not clarified, in spite of a huge research effort during more than one century in several important domains as fusion and space plasmas, fluid and atmosphere dynamics. Significant advance was obtained in the last decades, especially based on the fast development of the numerical simulations, while the improvement of the theoretical approach was much less marked. Even the basic process of stochastic transport, which is much simpler than the self-consistent treatments, has not a satisfactory theoretical description in the regimes with strong nonlinear effects.

We analyze the main theoretical methods applied to the stochastic advection of particles in two-dimensional incompressible velocity fields, Corrsin approximation [1] and the decorrelation trajectory method [2]. We have developed numerical simulations for the evaluation of the hypotheses of these theories rather than of their results (as done by many authors). This includes a new method for generating Gaussian random fields with space dependent average and dispersion for the calculation of the conditional averages that appear in these theories. The possibilities of improving these methods are discussed. Properties of the trajectories that could be used as new theoretical hypotheses are found.

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Keywords: statistical physics, stochastic transport, turbulence, fusion plasmas

Direct numerical simulations of the turbulent transport of heavy impurities in tokamak plasmas

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We present the statistical approach of direct numerical simulations (DNS) [1] and its applications in the study of heavy ions turbulent transport in fusion devices. Understanding the transport of heavy impurities (especially tungsten) is of paramount importance for the confinement and control of the plasma. Highly ionized impurities, if they reach the core of the plasma, become a dangerous channel of power loss. Our transport model includes the ExB drift, the polarization drift, the parallel acceleration [2], collisions or variable magnetic fields [3]. From a computational perspective we show that DNS is a fast and reliable tool for the investigation of turbulent dynamics. From a physical point of view, we offer interpretations and quantitative descriptions of transport coefficients, mapping their dependency on the properties of the turbulent fields.

Keywords: tokamak, turbulence, impurity, simulation

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Simulation of plasma disruptions triggered by Vertical Displacement Events in ITER

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It is well known that due to the nonlinear evolution of the MHD instabilities, the plasma discharge might be terminated in the form of a major disruption. It is expected that in ITER tokamak a limited number of major disruptions will definitively damage the chamber with no possibility to restore this tokamak. The understanding of how currents flow to the plasma facing surfaces during plasma disruptions has been considered by us as the key basis for our plasma disruption modelling. Consistently, we have developed a wall model that covers both eddy currents, excited inductively, and source/sink currents due to current sharing between the plasma and the wall. For the determination in space and time of the surface currents we have deduced a set of equations that we have solved in their weak form by minimizing the correspondent energy functionals and by using a Finite Element approach [1, 2, 3]. We have found that: a) the determination of the geometry of the wetting zone as well as the power deposition to edges of the protective plates in ITER is an essential step in disruptions simulation and b) the vertical disruptions have to be considered as fast equilibrium evolution rather than as an inertial MHD (plasma inertia force is 8-9 orders of magnitude smaller than electromagnetic forces in JET disruptions and irrelevant on time scale [4]).

Our code for simulation of the electromagnetic wall response during Vertical Displacement Events and Wall Touching Kink Modes received the status of open source license, to be used by the EUROfusion community. Our approach has been implemented successfully into the JOEKE-STARWALL code [5].

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Keywords: fusion plasmas, modelling and simulation, tokamak, MHD instabilities

Laser ablation technique for obtaining Be nanoparticles

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The FPW (First Plasma Wall) of ITER (International Thermonuclear Experimental Reactor) will consist of 8-10 mm of bulk beryllium (Be) [1]. Be particles can be expelled from the FPW during plasma operation due to ELMs [2] and imperfect plasma confinement. These Be nanoparticles (NPs) can produce severe damage due to its deposition on remote areas, changing the inner wall properties but also on diagnostic equipment.

In this work, we describe a method to obtain beryllium nanoparticles by laser ablation of a solid target immersed in liquid medium [3]. The scope of our work relates to the broader study of chemical and physical properties of Be particles and dust, in view of their future practical implications in the functioning of tokamak-based nuclear fusion reactors. Our approach of using laser ablation in liquid medium in order to produce NPs has several advantages: a) safe for the human body and the environment; b) has low cost; c) easy to implement in order to expand current research in this relatively unknown area. Be dust has been obtained following the irradiation of a beryllium bulk target immersed in water, acetone or heavy water, respectively, using a Nd:YAG laser source providing ns pulses. In order to argue the obtaining of Be NPs, scanning electron microscopy (SEM) was used for morphological and surface analysis. Colloid analysis by dynamic light scattering supported the SEM analysis findings in terms of particles size. The average NPs size for Be target laser ablated in water is between 40 and 85 nm and in heavy water around 70 nm. Surface X-ray photoelectron spectroscopy (XPS) was used to perform chemical analysis on Be dust existence and investigate their chemical bonds with other species. All colloidal solutions had high atomic contribution of Be (over 50%). The sample obtained in heavy water is distinguished from the rest by a high percentage of Be (66 at %) and small contribution of oxygen and carbon. This means that heavy water is the most promising liquid medium for obtaining Be NPs by laser ablation process.

Keywords: nuclear fusion, PFC, Be NPs, plasma laser ablation

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On diagnosing the magnetospheric plasma state from auroral tomographic observations

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Auroral tomography is a technique used to infer the spatial structure of auroral optical emissions from observations provided by an array of all-sky cameras. The analysis of tomographic data for stable auroral arcs allows for an estimation of the flux of energy carried by the accelerated precipitating magnetospheric electrons feeding the arc. We discuss here a method developed to infer the properties of magnetospheric plasma at the origin of the observed auroral arc. The method is designed for stable, quiet aurora and is built on a quasi-electrostatic magnetosphere-ionosphere coupling model. The method is based on a parametric description of magnetospheric interface generators from Vlasov equilibrium solutions derived for magnetospheric plasma interfaces having properties similar to kinetic tangential discontinuities. For each instance of the generator solution, we estimate the field-aligned potential drop between the magnetosphere and the auroral ionosphere. Furthermore, we can calculate the field-aligned current density, the flux of precipitating energy, and the height-integrated Pedersen conductance. The diagnosis of the magnetospheric plasma state is performed with a minimization procedure. We estimate an error in the least-square sense by comparing the numerical results with observational data. This way we find which generator model produces auroral arc properties that best fit the observations. The physical characteristics of the selected model provide an estimation of the real magnetospheric plasma state at the origin of the observed auroral arc.

A Perspective on the Scaling of Magnetosheath Turbulence and Effects of Bow Shock Properties

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Planetary magnetosheaths, like other heliospheric plasmas such as the solar wind, are naturally found in a turbulent state. Power spectral density (PSD) with a specific power-law scaling is typically interpreted as the trademark of turbulence. In fully developed turbulence, three characteristic power-law regimes, separated by spectral breaks, are observed: (i) the driving (or energy containing) range; (ii) the inertial range, dominated by nonlinear turbulent interactions which transfer the energy over multi-scales; and (iii) the dissipation range.

At inertial-range scales, where energy transfer dominates energy injection or dissipation, power-law scaling is expected for all moments of fluctuations, i.e., structure functions [1]. The power-law exponent of the PSD, α , is directly related to the second-order structure function exponent, ζ_2 , through the relation $\alpha = -(\zeta_2 + 1)$ [2]. A novel data analysis method AMPA - automatic Multi-order Power-law-fitting Algorithm (Tam & Chang, 2011) allows a rigorous determination of the scale ranges (usually chosen arbitrarily) and scaling exponents considering a global multi-order linear fit of the structure functions in the log-log representation.

We apply AMPA on magnetic field measurements provided by the Cluster spacecraft in the magnetosheath of Earth and analyze the scale behavior of the fluctuations for two events recorded behind quasi-perpendicular magnetosheath. An analysis of simultaneous solar wind data from the Advanced Composition Explorer suggests that the scale behavior of the magnetosheath fluctuations might be controlled by the structure of the bow shock; solar wind turbulent fluctuations are only partially destroyed while they are carried across the bow shock.

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Energetic Processing of Interstellar Dust Analogs Obtained in Dielectric Barrier Discharge

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Throughout the years, various carbon based materials have been studied as candidates for a series of absorption bands observed in the diffuse interstellar medium. So far, plasma based techniques have been proven as viable methods to synthesize these carbonaceous dust analogs. Our carbon dust analogs are synthesized using a dielectric barrier discharge operating in He and 15% hydrocarbons gas mixture [1]. The deposition method leads to the synthesis of an amorphous carbon “fluffy” structure with infrared signals corresponding to the signatures extrapolated from observational spectra of carbon rich regions in the interstellar medium.

While our product is a noteworthy carbon dust analog candidate, further energetic processing is necessary to fully understand the processes that interstellar medium particles undergo and the consequences it has on its morphological and chemical properties. To that extent, the dust analogs were irradiated using 3 MeV and 5 MeV protons and alpha particles at the 3 MV Tandetron™ accelerator at the “Horia Hulubei” R&D in National Institute for Physics and Nuclear Engineering – IFIN-HH [2].

This study focuses on the effects induced by energetic processing in the carbonaceous samples. The morphological changes, observed via Scanning Electron Microscopy, involve structure alterations due to etching and local phase transitions of the carbon flakes characteristic of these samples. The chemical structure is investigated using Fourier Transform Infrared Spectroscopy, performed in Attenuated Total Reflection mode. The absorption bands specific to astrophysical data of carbon-rich regions (3.4 μm, 6.87 μm, 7.27 μm) are still present post-irradiation, exhibiting an asymptotic decrease of the relative band intensity. Furthermore, based on the data from deconvolution of CH₂, CH₃ and C=C relevant absorption bands, we discuss the evolution of sp²/sp³, CH₂/CH₃ and H/C ratios, as well as the extent to which these results are in agreement with other carbon analogs and data extrapolated from infrared observational spectra.

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Vorticity Dynamics in Laser-induced Plasma

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Outline: Laser induced-breakdown has generated interest for a variety of practical applications: laser ignition of engines, sources for laser-induced breakdown spectroscopy, waveguides for remote standoff detection etc. A typical nanosecond laser breakdown event is governed by two processes: (1) multiphoton ionization (MPI): generates seed electrons; (2) electron avalanche ionization (EAI): grows the plasma through electron impact reactions. During EAI, the plasma kernel reaches high temperatures ($\sim 30,000$ K) and high pressures (~ 100 bar). Finally, the subsequent recombination phase is governed by the plasma kernel collapse into a toroidal structure comprising of two counter rotating vortex rings which generate significant air entrainment and cooling¹. Current study focuses on the physics governing the post-discharge vorticity dynamics. Experiments and numerical simulations are used to identify the main mechanism of vorticity generation (i.e. baroclinic torque) and propose ways to control its rotation direction using two overlapped pulses.

Methodology: Laser ignited kernels were visualized inside a high-pressure cell using OH* chemiluminescence². Numerical simulations were performed using a Riemann solver developed in-house based on the Navier-Stokes equations with species transport³.

Results: Shown below are kernel dynamics observed during the laser ignition of propane-air mixtures using two overlapped pulses (UV+NIR).

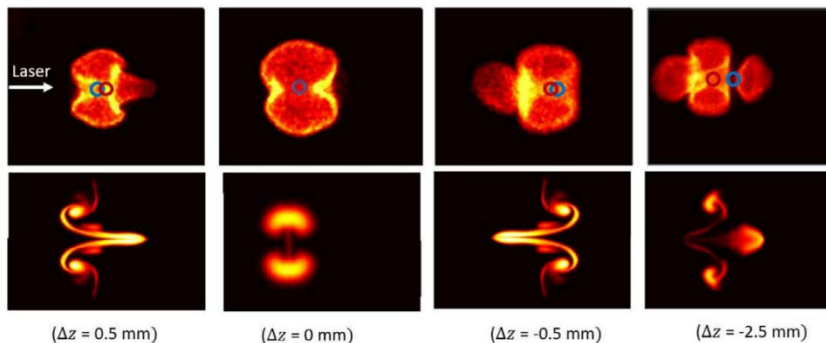


Figure 1: (Top) OH* Chemiluminescence images of the toroidal structure formed during the early ignition stages of a propane-air mixture. (Bottom): Numerical modeling results of the same experiment

Keywords: Plasma Instabilities, Laser Breakdown, Laser Ignition, Plasma Modeling

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Insight plasma structuring and oxidations processes during pulsed laser deposition

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In situ analysis on transient plasmas generated by ns-laser ablation of Ag in various background gases (Ar, O₂ and N₂) were performed by angular and time-resolved Langmuir probe technique and space- and time-resolved Optical Emission Spectroscopy (OES). The work was focused on understanding the complex dynamics of ablation plasma by tailoring the ionic energy distribution during the deposition process. A distinctive feature of the study is the focus on the floating regime of the probe as time-of-flight measurement tool. Plasma multi-structuring is seen for a wide range of pressures with each feature corresponding to an ionization state of the Ag ions, results confirmed by OES investigations. These results are discussed in the framework of multiple double layer formation during plasma expansion. Complementary, OES allowed for the spatial and temporal monitoring of visible and UV emission of the plasma. The nature and pressure of each gas influences the emission in a unique manner. Particular attention is given to the case of Ag in O₂ where we observed Ag_xO_y molecule formation and its impact onto the plasma energy. For each of the investigated conditions used, thin films were deposited and their properties linked with the ones of the plasma. This link is the main purpose of our complex approach as real time measurement and comprehensive control of pulsed laser deposition of thin films remains one of the main goals of the community.

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Characterization of carbon ions in excimer laser-generated plasma using the electrostatic energy analyzer

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The ion dynamics in laser-produced plasmas has been a topic of constant interest in the last decades due to various applications, from ion implantation to surface cleaning and patterning or thin film growth by pulsed laser deposition. Features like energy distribution, average charge state or ion relative abundance allow their optimization and development.

We bring here further insights on the carbon plasma expanding in vacuum by studying the positively charged particles using an electrostatic energy analyzer. KrF excimer laser ablation of graphite with intensities of $\sim 10^8$ W/cm² led to generation of spatially separated C¹⁺ and C²⁺ ions. Their individual energy distributions as well as global plasma parameters are evaluated for various laser fluences. This was varied in different manners, *i.e.* by changing the pulse energy or the focusing condition, and significant differences are inferred. By a multi-peak deconvolution procedure, the parameters characterizing each group of ions are deduced [1,2].

The global plasma parameters and their dependences on the laser pulse energy, *i.e.* on its fluence, were obtained by assuming a shifted Maxwell-Boltzmann distribution function. It resulted that the thermal velocity is slowly varying with the pulse energy. The opposite was obtained for the center-of-mass velocity, which is significantly increasing with the pulse energy. We remarked that, when a rectangular laser spot is used, the way how the laser fluence is adjusted (through the spot area by changing the focusing lens position or through the changing of the laser pulse energy) significantly influences the resulting plasma parameters.

Using a retarding electrical field to repel part of ions, the temporal recorded traces gained a multi-peaks shape, which is in correspondence with the C²⁺ and C¹⁺_{fast} ions from the fast plasma structure. The C¹⁺_{slow} ions belonging to the slow plasma part were evidenced as a hump in the tail of the temporal trace.

We also studied the influence of the laser pulse energy on the production of different types of ions, by using a multi-peak fitting procedure of the recorded signal. By increasing the fluence, we observed a significant increase of C²⁺ and C¹⁺ fast ions production, while the amount of singly charged ions in the slow plasma part is less changed. Also, the thermal velocities associated with each group of ions were evaluated. Developments of such simple experimental procedures are further possible, by comparison with theoretical calculations of fractional populations in the frame of different plasma models.

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Magnetron sputtering gas aggregation cluster source operated in plasma jet mode

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In this work, we present our first results regarding the operation of a magnetron sputtering gas aggregation cluster source in plasma jet mode (MSGA-jet), pointing to the experimental conditions triggering the transition from the classical MSGA mode to the MSGA-jet one [1]. In this new operation mode, the plasma expands through the exit aperture of the MSGA source in the deposition chamber. To the moment, we observed the MSGA-jet mode when using RF (13.56 MHz) discharges, the jet development being favored when the MSGA cluster source is operated with larger diameter exit apertures. For a specific diameter of the exit aperture, the MSGA cluster source can be operated in both classic and jet modes, depending on the position of the cathode inside the cluster source (the MSGA-jet is obtained shortening the aggregation lengths). It might also be obtained by increasing the pressure or the power applied to the magnetron discharge.

We note that both operation modes lead to the generation of nanoparticles. Here, we will present the possibility to obtain metallic core-shell nanoparticles by injecting a precursor gas into the plasma jet. To this purpose, we use a tungsten (W) magnetron target, Ar as sputtering gas (injected in the MSGA chamber), while C₂H₂ is used as precursor gas (injected in the plasma jet in the immediate vicinity of the exits aperture). The nanoparticles obtained in both MSGA and MSGA-jet mode were investigated regarding their morphology, structure and chemical composition using electron microscopy (SEM and TEM) and X-ray photoelectron spectroscopy (XPS).

Supplementary, the plasma jet is characterized by a low processing temperature, allowing deposition of the nanoparticles on the substrates susceptible to thermal damage. Thus, we successfully deposited W nanoparticles on PMMA foils using the MSGA-jet mode, without affecting the polymer surface.

Considering these findings, the MSGA-jet mode appears as a promising tool for application in nanotechnology.

Keywords: plasma jet, magnetron sputtering gas aggregation, core-shell nanoparticles.

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Plasma-based synthesis of mesoporous films of vanadium and vanadium-oxide nanoparticles

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Nanoparticles (NPs) and nanoparticles-based nanomaterials receive steadily growing interest in the scientific community. The popularity of NPs is foremost due to their unique physico-chemical properties and high surface-to-volume that make them highly attractive for use in various technological fields. This is especially true in the case of metal-oxide NPs that were reported to be advantageous, for instance, for bio-detection, photo-catalysis or as antibacterial agents. Naturally, the critical step is the controlled and reliable synthesis of nanoparticles with the required functionality.

The main aim of this study is to investigate the possibility of producing metallic vanadium nanoparticles using a plasma-based gas aggregation source (Fig.1) as well as to test the possibility to convert such synthesized nanoparticles into metal-oxide ones by an additional annealing step. It is shown that this fully solvent-free synthesis strategy allows for the production of vanadium NPs with a mean diameter of 30 nm (see Fig.1) at a deposition rate reaching several layers of NPs per minute. Furthermore, XRD and XPS analysis of produced NPs proved that they are fully transformed into crystalline V_2O_5 particles upon annealing at open-air at the temperature of 550°C. The change in the chemical structure of nanoparticle films is furthermore accompanied by alterations in the morphology of individual NPs and their optical properties with the formation of an optical band gap with an energy of 2.58 eV. Finally, the possible utilization of these materials will be discussed.

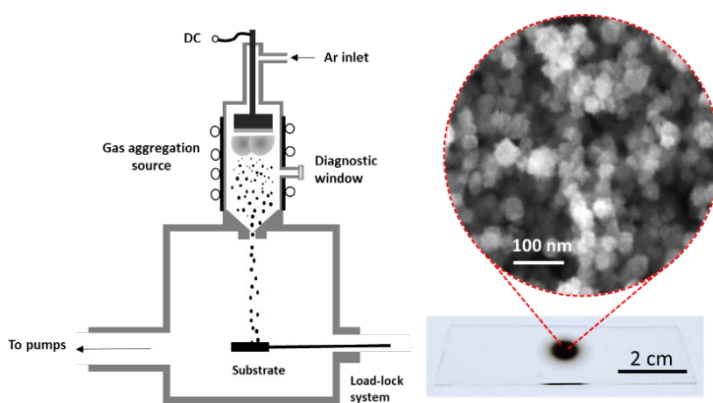


Figure 1. Experimental set-up (left) and photography of a glass slide with a spot of vanadium NPs together with a SEM image of nanoparticle film (right)

Copper and stainless steel plasma coated woven fabrics for EMI shielding

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Electromagnetic Interference shielding by means of flexible materials has found many applications in nowadays radiation polluted environment, mainly for protection of human's health and electronic devices [1]. There are two main technologies to impart electric conductive properties to textile fabrics according to [2]: insertion of conductive yarns into the fabric structure and coating of the fabric. Copper plasma coating on fabrics with inserted conductive yarns of silver and stainless steel was found to improve electromagnetic shielding effectiveness (EMSE) with 8-12 dB on the frequency domain 0.1-1000 MHz [3]. This study aims to assess shielding properties of woven shields with inserted metallic yarns both by plasma coating with stainless steel and copper.



Fig. 1 – Steel and Copper plasma coating on woven fabric with stainless steel yarns



Fig. 2 - Steel and Copper plasma coating on woven fabric with silver yarns

Two types of cotton woven fabrics with inserted conductive yarns were used: a fabric with stainless steel yarns in warp and weft direction with specific mass of 143 g/m² and a fabric with silver yarns in weft direction with the specific mass of 114 g/m², both with a distance of 4 mm between the metallic yarns.

These fabrics were coated in magnetron plasma with stainless steel and copper layers of 400 nm thickness on both sides of the fabric. Figure 1 & 2 present experimentally determined EMSE, according to ASTM ES07. Most effective results of an additional 20 dB in the frequency range of 0.1-10 MHz were achieved by the stainless steel coating on the fabric with stainless steel yarns, a fact to be explained by the improved electric connections on the fabric surface due to the similar metallic content.

Keywords: magnetron plasma, fabrics, EMI shielding, copper, stainless steel, silver

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Cold atmospheric pressure plasma assisted inactivation of bioaerosols containing bacteria, purified SARS-CoV-2 RNA and SARS-CoV-2

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Bioaerosols consist of airborne particles (0.001-100µm) originated biologically from plants and animals and can contain living organisms such as viruses, bacteria, and fungi. Moreover, higher bioaerosols concentrations may be observed in indoors generally associated with human activities that could spread diseases such as influenza, allergies, and respiratory syndromes [1]. In this field, cold atmospheric pressure plasmas (CAPs), thanks to their blend of bioactive agents (electrons, ions, UV rays and electromagnetic fields), enables the production of reactive oxygen and nitrogen species having antimicrobial properties, related to oxidation of cell membrane, protein molecules and DNA [1]. Several studies demonstrated the CAP assisted inactivation of bioaerosols [2]. In this work, experiments aimed at evaluating bioaerosols inactivation by a direct DBD plasma source are presented. The plasma source is driven by a high voltage generator (AlmaPULSE, AlmaPlasma s.r.l.) and it consists of two symmetrical electrodes both covered by a dielectric layer with an interelectrode gap of 2 mm, allowing the flow of bioaerosol through the plasma discharge. A suspension of *Staphylococcus epidermidis*, purified RNA of SARS-CoV-2 and SARS-CoV-2 virus was used to produce bioaerosols and inactivation experiments were done for different average power of the plasma discharge. The DBD showed the ability to reduce the bacterial load to a Log R~3 and fully degrade viral RNA and viruses. These preliminary results demonstrated the possibility to use a CAP generated by a dielectric barrier discharge to inactivate bioaerosols containing *S. epidermidis*, RNA of SARS-CoV-2 and viruses.

Keywords: DBD plasma source, bioaerosols, inactivation, SARS-CoV-2

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Laser-synthesized Si nanoparticles in liquid environment for biomedical applications

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Silicon nanoparticles (NPs) bears a high current interest due to the high number of potential applications in various fields, such as optoelectronics, solar cells, bioimaging and immunotherapy [1]. Among various methods used for their fabrication (electrochemical etching of monocrystalline silicon, thermal destruction of silicon-rich oxides, hydrothermal decomposition of different Si-contained organic precursors, etc.) the Pulsed Laser Ablation in Liquid (PLAL) is one of the most versatile [2]. When medical applications are envisaged, PLAL becomes the primary choice due to the wide range of experimental parameters available for tailoring their features (size distribution, shape, surface chemistry, etc.) and to the easy integration (surface functionalization with no need of surfactant in the synthesis of stable colloidal NPs solutions).

In this paper we report the use of PLAL to obtain silicon nanoparticles functionalized with Concanavalin A (Con A) protein for medical applications in immunotherapy. Laser ablation of a silicon wafer was performed in liquid media by using the first harmonic (1064 nm) of a Nd:YAG laser. The fabrication process was optimized by varying the laser fluence, the number of pulses used for the ablation, and the liquid media (distilled water, ethanol, etc.). Afterwards, the fragmentation of the obtained NPs in solutions was performed by using the third harmonic (355 nm) of the Nd:YAG laser, aiming to reduce their size and to modify their surface chemistry. Various analyzing techniques were applied to determine the structural (TEM, XRD, Raman), optical (UV-VIS absorption spectroscopy, photoluminescence) and chemical (XPS, FTIR) properties. Hydroxyl functional groups induced directly from PLAL were further used for the surface functionalization of NPs with Con A proteins through various chemical binding methods.

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Study of a Dielectric Barrier Discharge in Contact with Water

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Numerous investigations on plasma-liquid interaction have been published in the literature using various reactor geometries [1, 2]. However, studies on the basic configuration showed in Fig. 1 consisting of a pin-to-plane dielectric barrier discharge (DBD) in contact with water are still lacking. We therefore performed the electrical diagnostics of such a discharge, operated in a.c. regime with sinusoidal high voltage, focusing mainly on the power injected into the plasma in relation with several experimental parameters.

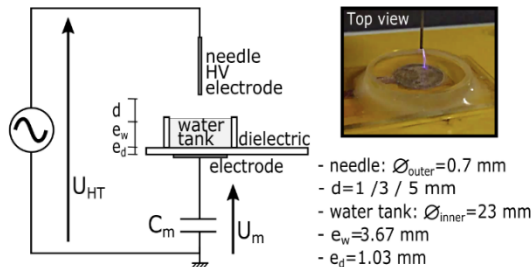


Fig. 1 Scheme of the experimental setup with the main dimensions and a picture of the discharge device.

The power dissipated in the discharge was determined by the method known as the Q-V diagram or Lissajous figure. It was found that the injected power increased linearly with the amplitude of the applied voltage, while without water the evolution followed a second-order polynomial. In both cases, the discharge power was approximately proportional with the frequency. For the DBD above water, the power was about three times higher than that without water, for the same air gap. It was observed that changing the discharge gap from 1 to 5 mm led to a slight increase in power, by only 15%. The water conductivity also influenced the power injected into the plasma only to a small extent. Thus, the variation in water conductivity over four orders of magnitude determined a fluctuation in discharge power of $\pm 20\%$. We believe that these observations, especially those concerning the effect of conductivity and of the distance between the electrode and the surface of the water, are valuable for the use of a pin-to-plane DBD in water treatment.

Keywords: dielectric barrier discharge, discharge over water, injected power.

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Hybrid Non Thermal Plasma/Heterogeneous Catalysis Process for emerging contaminant mineralization in water

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Many emerging pharmaceutical contaminants present in water have low biodegradability and are persistent to conventional wastewater treatments. In this context, Advanced Oxidation Processes (AOPs) have been extensively studied because of their degradation efficiency of organic molecules. Among AOPs, Non-Thermal Plasma (NTP) which has the capacity to efficiently produce reactive species ($\cdot\text{OH}$, O_2^- , H_2O_2 , O_3 , etc.) able to oxidize the most of organic micropollutants. However, the use of NTP reactors alone (Dielectric Barrier Discharges, coronas) lead to a very low mineralization rate [1, 2].

In order to improve the mineralization rate, NTP/Fenton-like hybrid process was developed in this work. The efficiency of Non-Thermal Plasma process was investigated with and without catalyst coupling. A homemade catalyst, iron (III) supported on glass fiber, was immersed in the liquid to be treated in a Dielectric Barrier Discharge (DBD) reactor with multiple-needles-to-plate configuration for paracetamol mineralization (Fig. 1). The plasma discharge was generated above the polluted liquid by injecting dry air as working gas. Chemical analyzes were performed to evaluate the paracetamol removal, to detect the produced species, and to evaluate the mineralization rate. The pH, conductivity, and concentrations of H_2O_2 , O_3 , NO_3^- , and NO_2^- were also measured. Electrical diagnostics allowed to evaluate the injected power and finally the Energy Yield (EY). The synergetic effects of plasma/catalysis coupling process led to a complete degradation of paracetamol with 54% in mineralization rate after 60 min of coupling treatment with an initial concentration of $25 \text{ mg}\cdot\text{L}^{-1}$ unlike plasma alone which did not lead to any mineralization. Moreover, EY was enhanced by a factor of two.

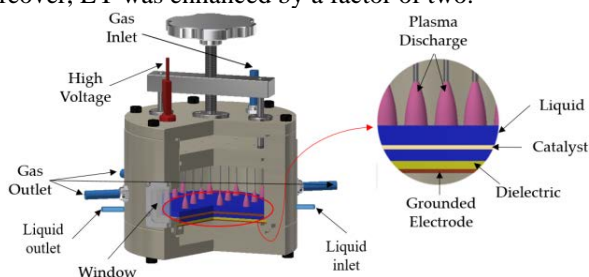


Fig. 1: scheme of DBD reactor for plasma/heterogeneous catalysis coupling

Keywords: Non-Thermal Plasma, Fenton-like, paracetamol mineralization

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Regular contributions

Topic 1

Processes in plasmas, modelling and simulation. Space plasmas

Kinetics of ozone generated by surface dielectric barrier discharge micro-plasma in small chambers

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Recent experimental investigations of molecular species produced by surface dielectric barrier (SDBD) micro-plasma in a small volume of atmospheric air enclosed by a discharge chamber revealed that the humidity in the gaseous discharge medium increases with drastic consequences on the generation of oxygen and nitrogen reactive species [1].

In this work, a computational model accounting for plasma generation, diffusion and consumption of O_3 is developed based on 2D continuity equation (diffusion approximation) in planar geometry. The model considers a discharge chamber (10 cm \times 10 cm) with the SDBD device placed at 2 cm and 8 cm from the top and bottom surfaces of the chamber, respectively (see Fig. 1). The O_3 is produced in the small volume of micro plasma of SDBD in air by dissociation of O_2 under collisions with energetic electrons [2]. The ozone diffuses and is consumed in volume reactions with OH radicals [3] and in surface reactions at inner walls of the discharge chamber [4]. The OH radicals are produced in plasma by water molecule dissociation under electron collisions [2] and by reaction between excited atomic oxygen produced in plasma and water molecules [5]. The time evolution of $[O_3]$ distribution in the discharge chamber shows that while the SDBD is running a large gradient of $[O_3]$ is generated by plasma and the discharge chamber gets filled with O_3 through diffusion (Fig. 1). After the SDBD has been cut off the spatial distribution of $[O_3]$ evolved towards homogenization. The presence of water vapour in the discharge chamber affects drastically the kinetics of ozone during discharge and post discharge times. In simulation, density of water vapour in the discharge chamber is considered to increase linearly during the discharge.

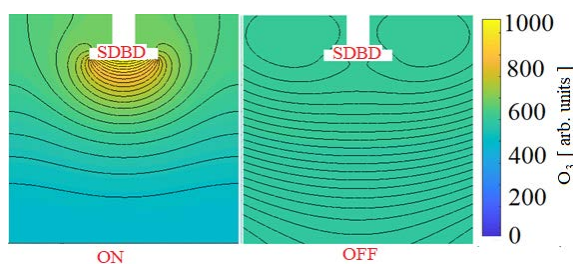


Fig. 1 Distribution of ozone concentration in the discharge chamber before and after the SDBD was cut off.

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Global Ionospheric Response to a Periodic Sequence of HSS/CIR Events During the 2007–2008 Solar Minimum

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In this study, we investigate the global ionospheric impact of high-speed solar wind streams/corotating interaction regions (HSS/CIR). A series of 10 such events are identified between December 1, 2007 and April 29, 2008, characterized in the frequency domain by the main spectral peaks corresponding to 27, 13.5, 9, and 6.75 days. The spectra of solar wind magnetic field, speed, and proton density, as well as those of the geomagnetic indices AE and SYM-H, are solely dominated by these features. By contrast, the ionospheric NmF2 and to a lesser extent the hmF2 spectra have a much more complex structure, with secondary peaks adding to or replacing the main ones. We argue that this is evidence of the nonlinear nature of the magnetosphere-ionosphere coupling, highlighted particularly in the NmF2 ionospheric response. Additionally, we show that hmF2 is more closely correlated than NmF2 to all parameters describing the solar wind and geomagnetic activity. Finally, the ionospheric response shows a higher correlation with Bz than any other solar wind parameter, and higher with SYM-H than AE, indicating that for the low-frequency part of the spectrum, high-latitude Joule heating and particle precipitation play a secondary role to that of prompt penetration electric fields in dictating the ionospheric response to geomagnetic activity, in the case of this sequence of HSS/CIR events.

Keywords: Solar Wind - Magnetosphere - Ionosphere Coupling, Ionospheric response to geomagnetic events Ionosonde data Spectral and statistical analysis

Velocity shear effects on the structure and stability of tangential discontinuities in collisionless magnetized plasmas

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Plasma discontinuities are sharp transitions of the particle and electromagnetic field properties at the interface between two different magnetized populations. In a tangential discontinuity, the normal components of the magnetic field and plasma bulk velocity vanish. The study of tangential discontinuities plays an important role to better understand the interaction between the solar wind and planetary magnetospheres. Indeed, such plasma structures have been detected in the solar wind and at the magnetopause by in-situ space probes. Moreover, the propagation of solar wind discontinuities and their interaction with Earth's magnetosphere is extremely important for space weather science. In this paper we present the results of 1d3v electromagnetic particle-in-cell simulations of two tangential discontinuities characterized by perpendicular velocity shear. The setup corresponds to a plasma slab streaming across a background population. The infinitesimal discontinuities evolve rapidly towards finite-width layers. The two discontinuities are asymmetrical, their scaling being determined by the sign of the perpendicular gradient of the plasma bulk velocity. Three-dimensional velocity distribution functions are computed in different locations across the discontinuities, at different time instances, for both electrons and ions. We emphasize the space and time evolution of the velocity distribution functions inside the transition layers and discuss their deviation from the initial Maxwellian distributions. The ion velocity distribution function inside the discontinuities is a mix between the isotropic Maxwellian of the background plasma and the displaced Maxwellian of the plasma slab. The numerical results obtained show similar features with the theoretical solutions provided by Vlasov equilibrium models.

Keywords: tangential discontinuities, velocity shear, particle-in-cell simulation, velocity distribution function, solar wind

Using PIC simulations for inertial fusion plasma modeling

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Inertial nuclear fusion is one of the main methods of implementing the controlled nuclear fusion on low power reactors. Inertial nuclear fusion is implemented on the laser driven installations of the special target with D-T core. Laser radiation ensures efficient target compression and heating, two key conditions necessary for efficient fusion reactions. The physical phenomena that take place in the inertial fusion devices are quite similar with the high intensity laser-matter interactions and can be studied using the same numerical techniques and theoretical approaches. In this work we studied the interaction of laser radiation with specific targets and described phenomena that strongly influence plasma compression. We simulate and compare three types of targets: simple nuclear fuel pellets, nuclear fuel pellets coated by a carbon shell and gold coated nuclear fuel pellets. We use EPOCH PIC code for numerical study of target ionization, plasma compressing and heating, growing of instabilities and generation of long-range correlations. We study the influence of laser power and pulse duration on the stability of plasma and radiation pressure in the target. As well we describe the plasma-laser interaction for different types of targets to choose the optimal configuration for predictable and efficient nuclear reactions. We conclude that nuclear fuel pellets coated with heavy elements shell are better confined and respectively are much appropriate for controlled nuclear fusion reactions.

Nonlinear diffusion of charged particles in pseudo-three-dimensional magnetic turbulence

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The motion of charged particles in the presence of magnetic turbulence, such as the solar wind or in the interstellar medium is an important but yet unsolved problem in astrophysics. Even though there exists a general agreement about the quasilinear regime of the parallel diffusion in the case of fluctuations parallel to a large-scale field, this is not the case for perpendicular diffusion in the nonlinear regime where the results vary between Bohm diffusion and sub-diffusive regimes due to the trapping of particles in the turbulent potential landscape [1]. We discuss the anomalous cross-field charged particle transport in a three-dimensional magnetic field turbulence with a large-scale mean magnetic. The turbulent magnetic field is modelled as a divergence-less field generated by Gaussian potential and the equations of motion include the ExB drift and an additional factor parallel to the gradient of the stochastic potential. We obtain the diffusion coefficient and the transport regimes as a function of the parameters of the model in the framework of the semi-analytical approximation called the Decorrelation Trajectory Method [2]. We show that the decorrelation produced by the gradient velocity interacts nonlinearly with the trapping effect. In the case of frozen turbulence, the transport is subdiffusive, while for a finite decorrelation time, we obtain diffusive transport in the nonlinear regime. The asymptotic diffusion coefficient is a result of the competition between the temporal decorrelation and the decorrelation produced by the gradient velocity.

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Keywords: diffusion, turbulence, astrophysical plasma

Effects of intermittency on turbulent transport in tokamak plasmasL. Pomârjanschi¹, D.I. Palade², M. Vlad²¹*Faculty of Physics, University of Bucharest, Romania*²*National Institute for Laser, Plasma and Radiation Physics,**RO-077125, Măgurele, Romania*¹*ligiapomarjanschi@gmail.com*

Turbulence plays a major role in the dynamics and confinement of tokamak plasmas. The turbulent electric field E is able, mainly via the $E \times B$ drift, to transport plasma across magnetic surfaces toward the walls. In the Scrape-off Layer region, in contrast to the core of the plasma, the turbulence is also characterized by intermittent phenomena: blobs, Alfvén modes, or ELMs. In this work we investigate how the turbulent transport is affected by these intermittent features. We compute semi-analytical estimations of the transport coefficients which are confirmed with the use of two different statistical approaches: the Decorrelation Trajectory Method (DTM) and the direct numerical simulation method (DNS). We have found that the intermittent phenomena, which depart the turbulent field from Gaussianity, tend to inhibit the transport with a linear dependence on the excess kurtosis of the potential. The characteristic susceptibility is strongly dependent on the correlation time τ_c with a peak value close to the time-of-flight τ_{fl} .

Keywords: tokamak, turbulence, intermittency, transport

Effective Larmor radius, conformal symmetry and the seeds of Edge Localized filaments

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In this work we argue that a particular parameter which occurs in the most common plasma dynamics – the drift waves – has a deep meaning and a fundamental importance in the tokamak plasma regimes: (1) fixes an upper limit to the H-mode; (2) return the 2D plasma to conformal invariance, and (3) clarifies the origin of filamentation of the pedestal.

A layer of strongly sheared plasma rotation at the edge of the tokamak plasma is considered to be the distinctive signature of the high confinement regime. The drift wave turbulence is reduced both by the sheared velocity tearing apart the eddies of correlated convection and by the change of the linear excitation. For the latter, a particular parameter seems to be highly relevant, the ratio between the diamagnetic ion velocity and the electric poloidal velocity. We show that this allows to define an effective Larmor radius which, when this ratio is close to 1, radically changes the conditions of linear excitation of drift waves and instead favors poloidal flow, which is observed experimentally.

We examine the subtle effect of the very large effective Larmor radius: the return of the Lagrangian invariance of the ion polarization drift and the loss of connection between the vorticity and the density (suppression of Ertel’s theorem). The plasma becomes an ideal 2D Euler fluid, with conformal symmetry, no intrinsic space scale. This regime is substantially different of the Hasegawa-Mima dynamics [1].

We show that a large effective Larmor radius for the ions allows excitation of electronic vortices. Their exclusive condition of existence is to have a section smaller than the ion Larmor radius. Or, this is precisely what is provided by the diamagnetic velocity being close to the poloidal electric velocity (i.e. virtually infinite effective Larmor radius of ions). These vortices can be considered the seeds of the filaments of the Edge Localized Modes, the events that suppress the edge transport barrier. The connection with the “peeling-balloonning” instability is also discussed.

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Scaling laws of 2D incompressible turbulent transport

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One of the fundamental problems of turbulence is the relation between the Eulerian characteristics of velocity fields and the Lagrangian characteristics of flows – represented by transport coefficients. The case of two-dimensional incompressible turbulence is particularly important in the description of magnetically confined plasmas, astrophysical plasmas, incompressible fluids, magnetic field lines wandering, etc. The resulting transport is known to be anomalous with supposedly universal scaling laws. In the present work we intend to take under scrutiny the dependency between the scaling exponents and the Eulerian properties of turbulence using the direct numerical simulation method (DNS), a state-of-the-art statistical approach to stochastic transport. The results are confirmed qualitatively using an approximative statistical approach, the Decorrelation Trajectory Method (DTM). We find that the exponents are weakly dependent on the turbulence spectra ($\sim 10\%$). We proceed to a quantitative characterization of these dependencies.

Keywords: turbulence, transport, incompressible, scaling

Topic 2

**Gas discharge physics, plasma sources and
diagnostics, dusty and laser plasmas**

Diagnosis of Be-D magnetron plasma operated in bipolar HiPIMS

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One of the main issues for future next generation fusion devices and ITER is represented by the management of in-vessel tritium retention. Tritium (T) retention into PFC can lead to a series of safety, cost and breeding issues, that can seriously impact the reactor lifetime. The allowed retention in ITER will be 700 g of T to avoid the release of high quantities of radioactive gas in case an accident occurs [1]. If this imposed licensing limit is reached, the reactor will be shut down. Based on extrapolations from experimental data and performed simulations for steady state operations in ITER it is expected that the largest part of the in-vessel tritium inventory will be retained in beryllium (Be) co-deposited layers [1]. In this context it is important to produce Be co-deposited layers in the laboratory frame and to study their fuel retention and release properties. Deuterium is usually used in experiments as a proxy for tritium due to supply and safety issues. Producing suitable Be co-deposited layers represents a challenge due to the fact that they have to be similar with the ones produced in fusion reactors. This contribution aims to perform electrical characterization of the Be-D plasma operated in bipolar HiPIMS regime in order to gain a better understanding of the parameters that govern the process, for future deposition procedure optimization. Measurements of I-V characteristic of the pulses, temporal distribution of plasma potential and ion saturation current respectively, were performed to establish in what manner the controllable parameters (target voltage amplitude, pulse configuration, length of the bipolar pulses, pressure inside the chamber) can impact the plasma density, ion current to the substrate and ion energy respectively. It was observed that the ion current to the substrates (in this case a biased planar probe) increases with the negative pulse width up to 20 μ s. In relation to the pressure, the ion current decreases quasi-linearly with its increase (0.7-3 Pa range) due to ion scattering at the collision with neutral gas particles. Also, the increase of the width of positive voltage applied on the target immediately after the negative pulse also indicate a gain in regards to the ion current. In terms of ion energy, the main interest process parameter is represented by the positive pulse voltage amplitude. Measurements performed showed that during the negative pulse only, the ion energy has a value \sim 5 eV, however this energy can be fine-tuned between 5-200 eV by changing the amplitude of the positive pulse. The research performed here could help in the future with the improvement of reference coatings similar in composition with the layers resulted during the functioning of a fusion reactor with Be as a PFC.

Keywords: bipolar HiPIMS, Be-D plasma, plasma characterization

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Characterization of a filamentary argon plasma jet by optical emission spectroscopy

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Outline: we present a spectral characterization of a stable argon plasma jet functioning in ambient atmosphere. The studied plasma jet (~40 mm) is generated at 13.56 MHz from inside a glass tube with an internal diameter of 4 mm. The discharge is operated with one external electrode on the tube, in the absence of any grounded electrode in discharge proximity. The regime is of interest as the plasma jet consists of a ultra-bright narrow (~0.5 mm) filament coexisting with surrounding diffuse discharge.

Methodology: plasma characterization is performed via 2-D optical emission spectroscopy (OES). The study includes measurements of electron number density obtained from Stark broadening analysis performed on the H_α and H_β lines [1]. A small amount of H₂ (1 sccm) was used for post processing the Stark broadening of H_α (656 nm) and H_β (486 nm) spectral lines. We verified experimentally, by comparing N₂(SPS) spectra with and without H₂ injection, that H₂ addition does not fundamentally change the plasma. Rotational temperature is obtained from the N₂ (C→B) emission spectra around 380 nm and characterizes quite accurately the gas temperature within the discharge since N₂(C) is primarily populated through electron-impact from N₂(X) state [2]. Finally, the electron temperature is determined using the line ratio method applied to 476.6 nm/750.4 nm Argon emission lines [3].

Results: Shown in Fig. 1 below are some preliminary results for the Stark broadening analysis performed on the RF-discharged. Electron number densities ~ 10¹⁵cm⁻³ are obtained near the exit plane of the discharge tube.

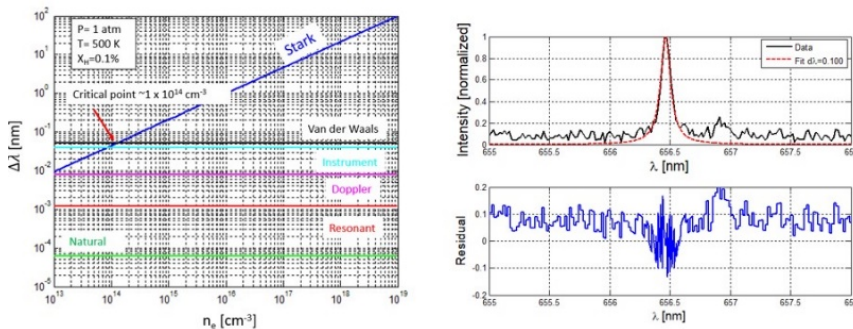


Fig. 1 (Left) Model of line broadening as a function of electron density showing the applicability of the Stark theory. (Right) Fitted H_α allowing determination of electron number density averaged over the tube diameter

Keywords: RF-plasma jet, Optical Emission Spectroscopy, Plasma Diagnostics

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Atmospheric pressure AC high voltage driven plasma jet: vacuum ultraviolet radiance measurements and electrical field diagnostics

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Atmospheric pressure, low temperature, plasma sources are rapidly gaining importance as tools for material worldwide processing, since they are easy to use, technologically simple and environmentally friendly. Applications of these plasmas are widely spread; therefore, it is important to characterize and monitor plasma sources from electrical and optical point of view, in order to fulfill the applications requirements.

Vacuum ultraviolet spectroscopy (VUV) is a powerful technique that can reveal information's about plasma excited species with high energy (up to 10 eV), in the 100-300 nm domain. These high energetic plasma species are of great importance for plasma applications (e.g. etching, decontamination, functionalization, polymerization).

Electrical field determination inside and around electrical discharges represents a continuous concern of the plasma physics community. The usage of an electro-optic sensor (based on the Pockels effect) promises to bring new experimental data on electric field measurements for plasma physicists.

In this report a dielectric barrier atmospheric pressure plasma source (appj), running in helium and argon, is characterized by means of electrical diagnosis, optical emission and vacuum-ultraviolet spectroscopy, as well as electric field diagnosis. We used He and Ar as working gases, at a flow rate of 2 SLM. The applied voltage on the discharge electrodes was around 10 kVpp, at 18 kHz, and 10 W mean power. The discharge configuration consists of a quartz tube (4mm / 6.1 mm diameter), and two 10 mm long copper tape electrodes (9 mm gap). The discharge was driven by a high voltage transformer powered by an amplifier which was controlled by a function generator.

The optical characterization of plasma source revealed a homogeneous discharge, both in He and Ar. The global emission spectra, in the UV-Vis range contains He and Ar lines, along RONS such as: NO, OH radicals, N₂, N₂⁺, O. We further on investigated the emission spectra in the 110-200 nm range using a VUV scanning monochromator (VM 505 + Thorn -EMI 9635 QB detector, Acton Research). In this spectral range we identified the lines of atomic H, O and N, providing powerful VUV radiation with energies up to 9.5 eV. For the Ar appj we observed also the Ar excimer.

For the electrical field measurements, we used an electrical field optical probe (ET5-air Transverse probe) coupled to an eoSenseTM electromagnetic field measurement system (EoSense LF 100U-1, Kapteos). Local electrical field distribution of the plasma was determined, and the values of the E-field was found to be in the range of 10⁵ [V/m].

Furthermore, experiment should be made, like electrical field characterization, surface charge production, ultra-fast photography, in order to have the hole understanding of the plasma source.

Keywords: plasma physics, E-field diagnosis, vacuum-ultraviolet spectroscopy, plasma-bioengineering

Capacitive power supplies for non-thermal plasma multi-reactors scale-up

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1. Introduction

The scale up of Non-Thermal Plasma (NTP) reactors requires a large number of units supplied from the same power supply to work in parallel. The present work aims to demonstrate the feasibility of a number of NTP reactors working in parallel simultaneously.

One problem limiting the use of NTP electrochemical reactors to treat large amounts of water was the difficulty of up-scaling treatment systems. The difficulty lies in the parallel operation of a large number of NTP reactors powered by the same power supply, which requires the parallel operation of several electric discharges, [1]. [2].

The main goal of the present work is to propose several high-performance electrical schematics of pulsed power supplies for NTP reactors system scale-up for industrial applications

2. Experimental

A first variant of the capacitive power supply intended for the treatment of high conductivity water polluted with PFAS, destined to replace the classic pulses generating power supply with rotary spark gap. The proposed power supply schematic consists in a circuit that uses two capacitors that double the secondary voltage of the HV transformer.

The second variant refers to parallel connection of several NTP reactors, which implies their simultaneous operation in the same conditions. This is difficult due to the fact that once an electric discharge is initiated the voltage at the electrodes decreases and cannot ensure the initiation of the discharge in the adjacent reactor. This can be solved by using discharge current limitation using passive elements (L, C) other than electrical resistors, which are consuming electrical power and reduce the efficiency of the multi-reactor system.

The third possibility is to scale up the NTP mini reactor systems consist in using a R-C circuit pulse generator. In this case the pulses are produced by the discharge of a charged capacitor from a direct current power supply through a resistor followed by its discharge directly on the NTP reactor. In this electrical configuration, the reactor plays itself the function of the spark gap.

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Observations of dusty CH₄ plasma with a simple image processing approach

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Hydrocarbon dust-particles are generated in low pressure classical planar radiofrequency discharge (13.56 MHz) in CH₄ gas. The gas is injected with a constant total gas flow rate of 5.6 sccm. The working pressure is 120 Pa and the RF incident power is 80 W. Methane decomposition leads to dust-particle generation within the plasma bulk and to the growth of a coating on the biased electrode. In the plasma, dust-particles become negatively charged and are submitted to forces [1] that keep them in levitation in clouds parallel to the electrodes at the sheath boundaries. The dynamics of the dust-particle cloud localized under the RF electrode modifies the electrical parameters of the discharge [2,3] such as the 4th harmonic of the RF current in this study. Nevertheless, for low disturbances in the cloud it can be difficult to get results from the recorded signal. To overcome this, an image processing approach is proposed. In this work, dust-particles are enlightened by laser scattering and images are cropped thank to a video camera. Prior being collected, images are filtered to only keep laser wavelength (consequently the dust-particle cloud) and cut the emitted light from the plasma. Then image processing is applied to the recorded video and consists in three main steps: dust-particles cloud segmentation, signal integration over the width of the cloud, stacking all processed images. The produced results, figure 1, is a t-x image of the whole experiment where periodical patterns appear.

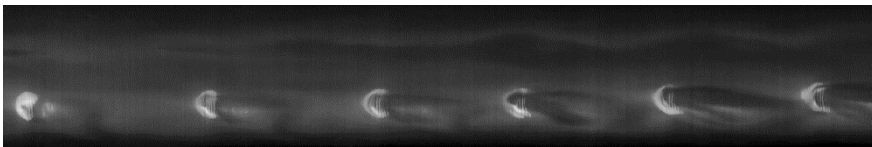


Figure 1. t-x sample over 50 seconds of experiments.

The aim of this work is to show that image processing of recorded videos of the dust-particle cloud allows to highlight and to quantify localized oscillating perturbations within the cloud near the RF electrode. Obtained results with the image processing are then correlated to the experimental time evolution of the 4th harmonic of the RF current. We will show that the image processing of the videos is essential to exploit the time evolutions of the 4th harmonic current.

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Keywords: dusty plasma, image processing, methane, plasma instability.

Acquisition and analysis of leaning input data used in helium APPJ control: effect of voltage and frequency

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Plasma sources are among the most modern and recent technologies used in various fields, including the medical devices. The laboratory results regarding modification of human cells metabolism and bactericidal effects of plasma at atmospheric pressure, as well the possibility of certification as medical devices are promising and drive new research activities nowadays, especially for establishing the control parameters of physico-chemical reactions at the plasma - biological environment interface [1,2].

In this work we present our approach to acquire large data sets that might be used as leaning input data for machine learning. The interaction of a helium atmospheric pressure plasma jet with two liquid media, namely distilled water and saline, was studied experimentally. The operational parameters kept constant were the gas flow rate, the profile and duration of the high voltage pulses, as well as the distance to the target. The amplitude and the frequency of the high voltage pulses were automatically varied in the range of 6 kV - 10 kV and 0.5 kHz – 10 kHz. Both, increase and decrease, of high voltage amplitude was studied, over multiple cycles. Following an accurate timing and using appropriate probes, the applied voltage and the electrical current on multiple paths, were acquired and stored into computer (Fig. 1).

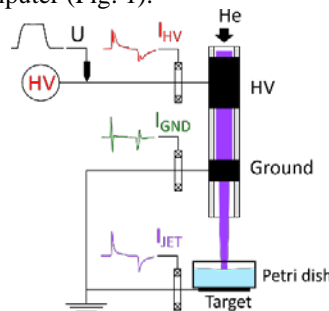


Figure 1. Schematic representation of the experimental setup used to study the He plasma jet impinging liquids

The subsequent analysis of all data sets was performed in order to extract relevant data, such as the values of current peak, charge and time characteristic of the current pulses, as well the variability and statistic parameters.

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The problem of time in Langmuir probe measurements of laser produced plasmas

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Complementary diagnostic tools were implemented in a systematic approach for the analysis of transient plasmas generated by ns-laser ablation of Co, Cu, Ag and Bi targets. The kinetics of the transient plasmas were investigated by angular dependence, spatial-temporal one and time-of-flight analysis. The main focus of our work was to implement a new Langmuir probe measuring regime by using the floating probe approach. Time-of-flight semi-empirical models are used to completely characterize the kinetic and thermal energy of the plasma in the context of pulsed laser deposition.

Multi-peak structures are seen for all metallic plasmas with a good correlation between physical properties of the target (atomic mass, melting point, thermal/electrical conductivity) and the charge density and ion kinetic energies. Each peak is correlated to different single and multiple ionized species (Co, Cu, Ag, Bi), aspect confirmed by optical time of flight where the spatial-temporal separation is also seen. The energetic structuring of the plasma is also observed in the context of time-resolved I-V characteristic for short evolution ($< 1 \mu\text{s}$) times, where strong wave-like behaviour appears. The origin of charged particle oscillation is further analysed in the framework of highly ionized fast expanding plasmas and the impact to the pulsed laser deposition process is discussed. Finally, the nature of the saturation current itself, measured in fast expanding plasmas is discussed and analysed in the light of these new results.

This work was supported by Romanian Ministry of Education and Research, under Romanian Nat. Nucleu Program LAPLAS VI –n. 16N/2019, ELI-RO_2020_12 and PD 145/2020. We acknowledge the Operational Program Research, Development and Education financed by European Structural and Investment Funds and the Czech Ministry of Education, Youth and Sports SOLID21.

Current oscillations recorded by an electrostatic energy analyzer from a Carbon laser-produced plasma

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Detailed characterization of the charged particles dynamics in transient plasmas generated by laser ablation of solid targets received in the past years a high interest, not only for fundamental reasons, but also due to the high number of potential applications, such as ion implantation, nano-processing, biomedicine, or surface cleaning and patterning. It generally consists in measuring the ion charge state, number density and angular distribution, energy, or velocity distributions. These measurements usually imply combinations of electrical time-of-flight methods, using simple Langmuir probes, Faraday cups, electrostatic energy analyzers or mass spectrometers. Our groups have tracked for more than a decade the occurrence of peculiar phenomena, such as plume splitting, charge separation in double (multiple) layers, oscillations in transient plasmas generated by laser ablation in vacuum of various targets [1].

We report here the observation of oscillations in the ionic current recorded with an electrostatic energy analyzer (EEA) at long (17.5 cm) distance from the target. An original signal processing approach based on the short-time Fourier transform (STFT) allows unveiling fine details on the temporal evolution of the oscillation frequencies [2,3].

The temporal traces of the ion current revealed different frequencies for the early and late parts, which were assigned to the fast and slow expanding plasma structures, containing different types of ions. The fast plasma structure oscillates with an average frequency of ~12 MHz regardless of the retarding voltage, while for the slow structure the oscillation frequency exhibits a linear increase, from 3 MHz to 5 MHz. STFT was successfully applied to the recorded signals to infer the temporal variation of the oscillation frequency. The resulting STFT amplitudes confirm that the two oscillation frequencies manifest more significantly when increasing the retarding bias. The time-evolution of the frequency shows different trends for the two plasma structures: decreasing for the fast one and slowly increasing for the slow one.

By investigating the correlation between the currents recorded by the different electrodes of the EEA, we observed that the periodic structures appear in all recorded traces, allowing us to conclude that the oscillatory behavior is an intrinsic manifestation of the plasma plume dynamics, related to the formation of multiple electric double layers.

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Optical emission spectroscopy of a BaSrTiO ablation plasma

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The nature and energy of the species in the ablation plasma plume is important for the growth of high-quality oxide thin films. The purpose of this work is a better understanding of the phenomena governing the growth or modification of the thin films in plasma.

The dynamics of the ablation plasma produced at pulsed electron beam interaction in a pulsed electron beam deposition (PED) setup with a barium strontium titanate oxide target was studied by time-resolved optical emission spectroscopy in argon at a pressure of 10^{-2} mbar. The aim of such optical spectroscopic emission investigations with temporal resolution was the identification of plasma species, the determination of plasma parameters and the identification of the optimal substrate location for the materials to be deposited, and also the correlation of the plasma parameters with the working conditions of the PED device.

The temporal evolution of the spectral lines of target material was investigated together with that of the ionized background gas. Temporal resolution was achieved by changing the iCCD detector gating trigger delay relative to the discharge ignition time and integrating each gate delay on 100 discharges. The time slices were 8-30 steps of 100ns, best suited for the average spectral lines' lifetime and comparable with the duration of the pulsed electron beam current, respectively. A duration of the Ti lines of about 700 ns was observed, correlated with the working gas (Ar) lines. Sr and Ba lines were delayed starting with the peak of the Ar lines intensity at 300-400ns and persisting after the main discharge up to 3 μ s. Peak temperatures of about 1eV for the working gas and about 2 eV for the ablated species were determined using the Boltzmann graph.

Due to similarities between PED and pulsed laser deposition (PLD), these results were analyzed and discussed in the frame of existing ablation models.

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Keywords: ablation plasma, pulsed electron beam deposition, optical emission spectroscopy

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Characterization and optimization of Laser Induced Breakdown (LIB) as an ignition method to generate open field blast waves

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The aim of this work is to characterize the Laser Induced Breakdown (LIB) technique used as a new ignition method able to replace the exploding wire technique to generate blast waves from gaseous charges in open field and to study their effects on structures (buildings, etc.). In particular, we wish to characterize the mechanical energy of the LIB related to the laser energy.

In our experiments, realized in ambient air, we used a pulsed Nd:YAG laser ($\lambda=532$ nm, $\varnothing=9$ mm, $\tau_{FWHM}=5$ ns) with a waist of $2,42$ μm for the focalization and an energy varying between 5 and 200 mJ. The absorbed energy is deduced from a measure from an energy meter (Ophir PE50-DIF-C). To characterize the blast wave, fast imaging and the shadowgraph technique [1] (Fig. 1a) were used to follow the evolution of its radius versus time (Fig. 1b).

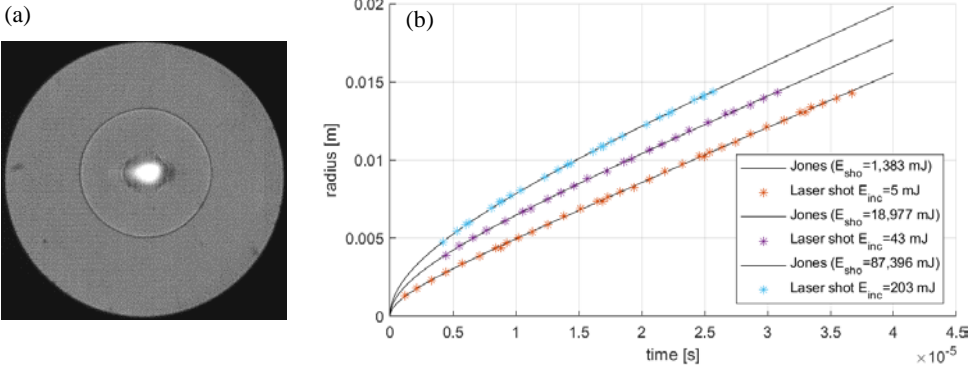


Figure 1: (a) Shadowgram of a blast wave in air;
 (b) Radius of the blast wave versus time: experiment and Jones model.

As N.D. Peters [2] and G.C. Gebel [3] did, we characterize the blast wave by using the Jones intermediate strength blast wave model, a model that better suits the phenomenon than Sedov-Taylor, mainly designed for strong shocks [4]. In this work, the hypotheses of the used model are discussed. The mechanical energy from the blast wave resulting from the LIB can be deduced with the Jones model.

This energy increases with the laser incident energy, over a range between 5 and 200 mJ. The proportion of the blast wave energy associated with the absorbed energy (E_{sho}/E_{abs}) decreases until it reaches an asymptote around 45%. This behavior is especially interesting for the aim of this project because it allows an easy determination of the shock energy from the laser energy.

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Topic 3

Plasma material processing and fusion technology

The role of the deposition on the optical and electrical properties of indium oxide thin films

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Transparent conducting thin films are advanced functional materials widely used for various applications, including electronic and photovoltaic devices which require the typically mutually exclusive properties of transparency and electrical conductivity [1, 2]. Among the common list of different transparent conducting materials, indium oxide (In_2O_3) is one of the widespread materials due to its low resistivity, high transparency and good etchability [1].

In_2O_3 thin films were grown on various amorphous and single crystalline substrates between room temperature and 500 °C by pulsed electron beam deposition method (PED). The parameters during the PED growth (pressure, working gas and substrate temperature) had strong effects on the electrical and optical film properties, without evidencing large differences in the crystalline structure and film stoichiometry. Detailed Hall effect (resistivity, carrier mobility and density) and optical transmission measurements were performed for the films deposited under about 10^{-2} mbar pressure in either oxygen or argon gas at different substrate temperature. The electrical properties of In_2O_3 thin films grown by PED are tunable, spanning over four decades of resistivity values and modifying the semiconductor behavior from a non-degenerate to a degenerate one.

The optical transparency was high in the visible spectral range for all films (>80%, including the substrate contribution), while in the near-infrared range it was dependent on the carrier density. A correlation between the film composition, structure and their optoelectronic properties was made. In addition, X-ray photoelectron spectroscopy measurements showed the correspondence between surface properties and functional properties of In_2O_3 thin films grown by PED. Finally, a discussion about the role of deposition methods on optoelectronic properties of In_2O_3 thin films will be presented in comparison with films grown by different methods (PED, PLD, RF magnetron sputtering, Chemical Vapor Deposition, DC Plasma, Atomic Layer Deposition, etc.).

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Keywords: ablation plasma, thin films, pulsed electron beam deposition

Acknowledgement: This work was supported by the project Romanian National Nucleu Program LAPLAS VI-contract no.16N/08.02.2019

Electrical characterization of CNW architectures in view of gas sensor applications

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Carbon nanowalls (CNW, vertical graphenes) consisting of inter-connected vertical carbon sheets built from small graphene domains with their c-axis oriented parallel to the substrate are promising materials for new electrical devices. The material based on vertical graphene's functionalized and decorated as well their application in gas detection may have a tremendous economic impact.

The main goal of this study was to morphologically and electrically characterize CNW based fabricated architectures [1]. The obtaining of reproducible CNW materials based on vertical graphene, with various morphological and structural properties was already demonstrated at laboratory level. The CNW synthesis is done by PECVD in a radiofrequency plasma jet, based on Ar discharge feed with H₂/C₂H₂ [2].

Electrical measurements were performed on CNWs deposited on Pt electrodes by using a Keithley 2400 source-meter and a Keithley 6517a electrometer, commanded by a computer, at room temperature. For the prepared samples, both forward and reverse current-voltage (I-V) characteristics were acquired in the ranges of -0.5 – 0.5 volts. The current-voltage characteristics proved the semiconductor behavior of such structures by their asymmetry between forward and reverse curves (Figure 1). Such kind of measurements, aiming to the identification and testing of the CNW materials with new properties controlled during synthesis and upon plasma post-treatment, represent very recent approaches (as example the using CNW material in gas detection).

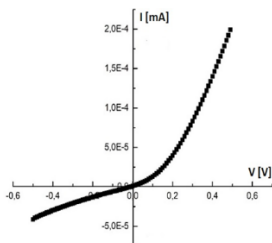


Figure 1. I-V characteristics of CNW acquired at room temperature

Keywords: carbon nanowalls, argon/nitrogen treatment, electrical resistance

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Surface analysis of zinc oxide thin films grown by pulsed electron beam deposition

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ZnO thin films are a widely adopted alternative for replacing doped indium oxide films as electron transport layer in photovoltaic applications. In particular, the crystallographic polarity influences the ZnO optoelectronic properties, because the typical wurtzite has alternating atomic layers of Zn and O ions along the c-axis with cationic (Zn-face) or anionic (O-face) surface termination. The purpose of this work was to show that the polarity of zinc oxide thin films can be controlled by the film growth method.

In this study ZnO thin films were grown by pulsed electron beam deposition (PED) on c-cut sapphire single crystal substrates at different temperatures and gas pressures. Epitaxial thin films with tailorable optical and electrical properties were grown by varying the film deposition parameters, even starting from relatively low substrate temperatures. Hall effect and optical measurements were used in order to study the transport properties, bandgap and transparency of the films.

X-ray photoelectron spectroscopy was performed both for investigating the film surface, the valence-band structure and in particular to determine the polarity of the wurtzite ZnO thin films. These investigations revealed the dominant polar face of ZnO films as function of the growth parameters. The results will be discussed in correlation with the semiconductor electrical properties of the film and the pulsed aspect of the film growth, for example the kinetic energies of species impinging on the substrate determined from previous *in situ* ablation plasma plume diagnostics (ion probe current waveforms).

Keywords: zinc oxide, thin films, pulsed electron beam deposition

Acknowledgement: This work was supported by the project Romanian National Nucleu Program LAPLAS VI-contract no.16N/08.02.2019.

Obtaining Ag-fluorocarbon nanocomposites with controlled properties by combined plasma-based methods

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Nanocomposite materials consisting of polymeric matrix with embedded metal nanoparticles have recently received considerable attention due to their unique physical properties [1]. Such metal-polymer nanocomposites can be used as materials for novel functional applications in optics [2], electronics [3], magnetics [4] and medicine [5].

The present work focuses on the synthesis and characterization of metal-fluorocarbon nanocomposite materials fabricated by combined plasma-based techniques, namely physical vapor deposition (PVD) and plasma enhanced chemical vapor deposition (PECVD). Deposition of metal-fluorocarbon nanocomposites was performed in a spherical vacuum chamber on which the PVD/PECVD plasma sources are mounted at 45° in respect to the substrate holder and 9 cm distanced from it. Silver nanoclusters were obtained by various physical vapor deposition methods, based on magnetron sputtering (MS) in classical configuration or with gas aggregation (MSG) sources. For the fluorocarbon matrix, the synthesis was conducted via magnetron sputtering of polytetrafluorethylene (PTFE) target or by plasma assisted chemical vapor deposition using fluorine containing precursors (SF₆/CH₄).

The successful synthesis of AG NP was evidenced upon topographical and morphological investigations performed by means of Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). The dependence of the chemical composition of the metal-fluorocarbon materials upon the applied RF power and the SF₆/CH₄ ratio was evaluated through Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Photoelectron Spectroscopy (XPS) investigations. Spectroscopic ellipsometry allowed to prove that optical properties of such nanocomposites can be finely controlled via the synthesis parameters of both components.

Keywords: Ag/fluorocarbon nanocomposites, nanoclusters, magnetron sputtering, gas aggregation, PECVD

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Analysis of HCl doped polyaniline thin films by DC plasma polymerization reactor

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Abstract

Thin polymeric films have a great interest in modern technologies with applications from gas sensors, LEDs to photovoltaic panels and electromagnetic shielding due to their mechanical and electrical proprieties, low cost and the ease of production. Polyaniline (PANI) is one of the most common used polymer for its electrical proprieties and stability and wide range of applications. The usual methods of obtaining PANI films are bulk polymerization, interfacial polymerization and electrospinning. The last one is used rarely due to the need for a very high voltage power source. The conductive form of PANI is emeraldine salt, obtained by doping (protonatic) the emeraldine base form of PANI with an acid. This work is focused on the analysis of thin HCl doped polyaniline films obtained by a DC plasma polymerization reactor [1], as a continuation of our previous work that investigated the influence of plasma and deposition parameters on pure PANI films. Several thin film were prepared by doping the aniline monomer with HCl solutions with different concentrations (0.1M, 0.5M, 1M, 2M) and injecting them inside the positive column of a DC glow discharge. The plasma forms free radicals that bond on a substrate placed inside the reactor forming polymer chains. The morphological proprieties were investigated by Scanning Electron Microscopy (SEM) and molecular analysis was performed by Fourier-transform infrared spectroscopy (FT-IR). The average thickness of the deposited samples is around 1 μ m. Conductivity measurements were performed by using a Keithley 2400 SourceMeter in order to investigate the influence of protonation by HCl solution of the thin polymeric films.

Keywords: Plasma polymerization, polyaniline thin films

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TRIBOLOGICAL PROPERTIES OF CHROMIA AND CHROMIA COMPOSITE COATINGS DEPOSITED BY PLASMA SPRAYING

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ABSTRACT

Ceramic coatings can be used in various industrial applications to protect surfaces from wear and abrasion [1-2]. Plasma sprayed chromia coatings are known to have high hardness and high anti-wear resistance in dry and lubricated sliding [3]. Small additions of SiO₂ or TiO₂ can further improve the properties of coatings [4]. The aim of this work was to coat stainless steel substrates using different material powders as well as to compare tribological properties of chromia and chromia composite coatings formed at different plasma temperatures.

Chromia (Cr₂O₃) and chromia composite (Cr₂O₃-SiO₂-TiO₂ and Cr₂O₃-SiO₂-TiO₂-graphite) coatings on stainless steel substrate were formed using atmospheric plasma spraying. An aluminum bonding layer was used to achieve higher adhesion between the coating and the substrate. Plasma jet was created using an air and hydrogen gas mixture. Plasma jet temperatures were varied from 3420 K to 3530 K by increasing the arc current from 180 A to 220 A. The surface morphology was investigated using scanning electron microscopy (SEM) Hitachi S-3400N. The elemental composition of the deposited coatings was determined by energy dispersive spectroscopy (EDS) using Bruker Quad 5040 spectrometer. The phase composition of the coatings was investigated by X-ray diffraction (XRD). The friction coefficient was measured using a CETR-UMT-2 ball-on-flat tribometer with 1 N and 3 N loads. Specific wear rates were estimated from the surface profiles, which were measured using Ambios XP-200 Profiler.

Surface morphology investigation results indicated that addition of SiO₂ and TiO₂ decrease surface roughness. EDS results show that chromium oxide coatings on average consisted of ~52,3 at.% Cr, ~46,9 at.% O and ~0,8 at.% of other elements. The highest intensity peak in the XRD patterns for chromia coatings corresponds to eskolaite (Cr₂O₃) phase. Chromium oxide composite coatings with addition of graphite had an additional (002) graphite peak. Chromia coatings coefficient of friction (COF) was in the range from 0,407 to 0,674. It was obtained that the specific wear rates of coatings were at least 10 times lower compared to the steel.

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Nanoscale tribological properties of diamond like carbon films deposited by different magnetron sputtering techniques

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Diamond like carbon (DLC) thin films are promising candidate coatings for low-friction, wearing-resistant, chemically-inert, bio-compatible surfaces for a wide range of applications. While the chemical bonding (ratio between sp^3 and sp^2 orbital densities) in these film structures depends on deposition techniques and deposition parameters, it also largely affects their tribological properties.

In the present work we investigate comparatively the nanoscale friction and adhesion properties of DLC thin films (about 350 nm in thickness) obtained by three magnetron sputtering techniques, i.e. dc magnetron sputtering (DCMS), unipolar high power impulse magnetron sputtering (s-HiPIMS) and bipolar high power impulse magnetron sputtering (b-HiPIMS) [1]. The DLC films were deposited by sputtering of a graphite target on polished silicon substrates by applying dc voltage (-500 V), unipolar voltage pulses (-900 V in amplitude and 10 μ s in width) and bipolar voltage pulses (negative pulses followed by positive pulses with amplitude of 200 V and 50 μ s in width) in DCMS, s-HiPIMS and b-HiPIMS techniques, respectively. All three magnetron sputtering techniques were performed in Ar gas at pressure of 1Pa and constant mass flow rate of 50 sccm. Raman spectroscopy investigation revealed sp^3 bond contents of 18%, 39% and 51% for the DLC films obtained DCMS, s-HiPIMS and b-HiPIMS techniques, respectively. The adhesion and friction properties of the three DLC coatings were investigated by atomic force microscopy (AFM) working in contact mode with sharp silicon nitride tips. The friction versus load curves were acquired by scanning the DLC film surfaces at constant speed of 1 μ m/s under variable normal loading force. The results (Fig. 1) showed a linear dependence of friction force on the externally applied loading force for all films, which determined friction coefficient values of 0.16, 0.21 and 0.19 for films obtained by DCMS, s-HiPIMS and b-HiPIMS techniques, respectively. The surface distribution of friction force for the DLC film obtained by DCMS showed a heterogenic diamond/graphite structure with nano-domains of very low friction, which are attributed to graphite.

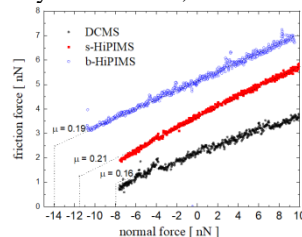


Fig. 1 Friction force versus loading normal force for the DLC films deposited by the three magnetron sputtering techniques

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Copper-based transparent heat reflecting plasma coatings for energy saving applications

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Increasing attention was devoted in the past years to development of energy saving applications, in the context of conventional energy resources depletion. In this regard, several studies highlighted the importance of using transparent heat reflectors with high performance and low manufacturing costs. One simple solution consists in developing a transparent heat reflector based on a multilayered plasma coating characterized by various spectral characteristics. According to the International Energy Conservation Code (IECC), the multilayer structure has to be fully integrated into energy efficient windows, doors, skylights and other fenestration products. Also, this targeted spectrally selective coating must be capable to exhibit a good transparency in the visible spectral range along with a high reflectivity in the near-infrared range.

The aim of the present study is to obtain a multilayer structure with high thermal performance comprising only few alternating dielectric (SiN_x) and metallic layers (Cu). The SiN_x monolayers were deposited by RF magnetron sputtering technique from a Si target in an Ar/ N_2 gas mixture, while the Cu monolayers were obtained by using either DC or HiPIMS magnetron sputtering techniques of a Cu target. The optical properties of metallic layers were evaluated and compared in terms of initial performance (ex-situ) and stability over time.

The deposited mono- and multilayers were analyzed by spectrophotometry measurements in order to derive two important parameters which accounts for glazing's solar heat gain (g factor) and thermal losses (U factor). These parameters were evaluated for two types of window structures, comprising 2 and 3 glazings with 4 mm thickness which are individually separated by a 12 mm extra space filled with various types of gases (i.e. Ar, Kr, Xe).

Summarizing, the obtained Cu based transparent heat reflectors can be considered a viable solution for rising the architectural attractiveness in terms of the cost-effectiveness, stability over time and process reproducibility.

Keywords: transparent heat reflectors, optical coatings, multilayer design, magnetron sputtering.

Acknowledgement: This work was supported by a grant of the Ministry of Research, Innovation and Digitization, CNCS/CCCDI – UEFISCDI, project number 105TE/2020, within PNCDI III, and Core Program, Project no. 18N/2019.

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Plasma Functionalized Carbon Nanowalls as Microporous Layers in PEM Fuel Cells

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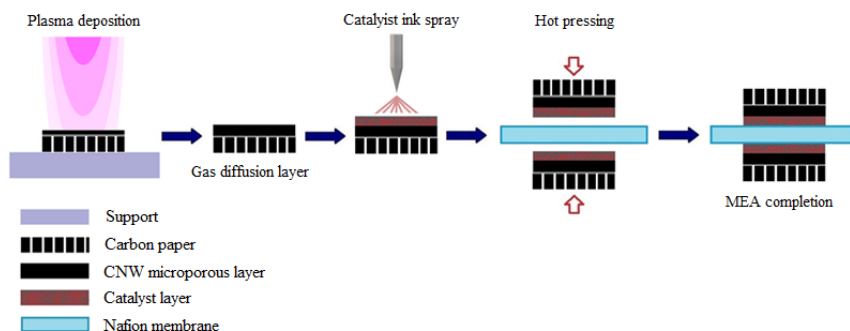
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Abstract

The cathode microporous layer (MPL), as one of the key components of the proton exchange membrane fuel cell (PEM-FC), requires specialized carbon materials to realize the two-phase flow and inter-facial effects. In this respect, we designed a novel MPL based on super-hydrophobic nitrogen doped carbon nano-walls (CNW). Employing radio-frequency plasma enhanced chemical vapor deposition techniques directly on carbon paper we produced high quality microporous layers at competitive yield-to cost ratio with distinctive MPL properties: high porosity, good stability, considerably durability, high hydrophobicity and substantial conductivity [1].

Platinum-ink, serving as fuel cell (FC) catalyst, was directly sprayed on the MPLs and incorporated in the FC assembly by hot-pressing against a polymeric membrane [2]. The integrated PEM-FCs were tested in a single cell PEM-FC on a BT-112 Single Cell Test System, showing power performance comparable to industrial quality membrane assemblies (330 mW·cm⁻²), with elevated working potential (0.99 V) and impeccable fuel crossover for a low-cost system resulting from a highly scalable, inexpensive, and rapid manufacturing method.

Graphical abstract



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Acknowledgements

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Structural and magnetic specificity in Fe-B intermetallic thin films obtained by thermoionic vacuum arc and magnetron sputtering

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Iron (Fe)- boron (B) thin films are intensively studied due to their special properties and the wide range of fields in which they are used. Boron is the element that facilitates the increase of the hardness and the wear resistance of the material in which it is inserted. In combination with iron, boron is an effective glass former, helping to form amorphous structure at relatively low concentrations. The major difficulties in the case of Fe-B thin films are the control of the preparation of films of well-defined concentrations and the possibilities of accurately determining these concentrations. Thus, the present study closely analyzes the morpho-structural and magnetic properties of thin Fe-B films having thicknesses of 90-100 nm, focusing on the comparison between the films obtained by two preparation methods, namely: (i) Thermoionic Vacuum Arc (TVA) and (ii) Magnetron Sputtering (MS). Structures with different B concentrations between 5% and 50% atomic were obtained. Morpho-structural properties were highlighted using the X-ray diffraction (XRD) method supplemented with X-ray reflectometry (XRR) and Scanning electron microscopy (SEM). Elementary determinations were made by both X-ray dispersive spectroscopy (EDX) and Rutherford backscattering spectroscopy (RBS). At the same time, the magnetic properties of the Fe-B layers were carefully investigated by Kerr magneto-optical effect, SQUID magnetometry and Mössbauer spectroscopy. It was highlighted that the films become amorphous for B concentrations of over 10% atomic. With much more highlighted ferromagnetic characteristics in the case of films obtained through TVA. Mössbauer conversion electron spectroscopy (CEMS) showed the orientation of the Fe spins in the plane of the film while MOKE experiments performed for various orientations of the magnetic field in the plane of the film showed a dependence of the angular distribution of the light magnetization axes strongly dependent on the B concentration.

RF Magnetron Sputtering Co-deposition and Characterization of Hydrogen Permeation Barrier Oxides in Tungsten & Beryllium Metallic Reinforced Matrix Composites

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Over the last decade, the fusion research community undergone extensive studies for controlling the tritium inventory in the future nuclear fusion reactors, proven not to be a straightforward task due to several material constrains. For example, the Demonstration Power Station (DEMO) will integrate inner walls made from low activation steel, such as martensitic steel that has a high hydrogen permeability [1]. Therefore, highly impermeable materials (e.g. metals, oxides) applicable as hydrogen permeation barriers (HPB) are considered the most valid candidates for mitigating and controlling the tritium inventory.

Here, we report the deposition of HPB-like layers by RF magnetron sputtering system. Taking into consideration that an ideal HPB layer exhibits an appropriate homogeneous, dense and defect-free configuration, the deposition process was followed by a complex characterization consisting in structural, mechanical and topological measurements.

Thus, single thin layers of W, Be and oxides (Al_2O_3 , Cr_2O_3 , Er_2O_3 , SiO_2) and co-deposition configurations (50:50 wt.%) as reinforced matrix composites (W, Be) - (Al_2O_3 , Cr_2O_3 , Er_2O_3 , SiO_2) were deposited on stainless steel (316-low carbon) substrates.

Therefore, from the SEM images, one could observe significant surface morphology improvement for the W, Be - Cr_2O_3 co-deposition comparing with the pure oxide; while the AFM measurements showed us a mitigation of the RMS roughness factor for all co-depositions. Scratch test determined an improved adhesion characteristic for Be - Er_2O_3 , while other co-depositions presented a weaker adhesion property in comparison with pure oxides.

These results are highly relevant for choosing the proper material configuration what could be categorized as HPB, furtherly studied in laborious permeation yield campaigns.

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Keywords: hydrogen permeation barrier, oxide, tritium inventory;

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A plasma method to decorate carbon nanomaterials with tungsten particles for sensor applications

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Carbon nanostructures (such as carbon nanowalls/carbon nanofibers) are among the most studied materials. By attaching metal nanoparticles (particularly tungsten), we can enhance physical and chemical properties, needed for new applications, namely as gas sensors.

In this contribution we present the possibility of synthesize hybrid architectures by combining carbon nanostructures [1] with tungsten particles. The experimental technique consists of two steps: one based on plasma enhanced chemical vapor deposition, for obtaining the carbon nanomaterials [1], and the second in which we decorate de carbonic material with W particles created via erosion of the discharge electrode [2], by using a radiofrequency (13.56 MHz) atmospheric pressure plasma jet.

The hybrid structures were investigated by SEM, EDS, XPS methods. From the SEM images we could observe that the W particles have spherical and cauliflower-like particles and that they cover the CNW prevalent on the edges, in contrast to other deposition methods [3]. In XPS spectra we could observe that the tungsten particles prevalent combines with oxygen and nitrogen from ambient atmosphere, resulting wolfram oxides and nitrides. Also, we could observe specific bonds of carbon, such as C-C or C-Hx and W(CO)₃.

In further study we focus on using the tungsten decorated carbon nanomaterials as constructive elements of gas sensors, gas storage devices, supercapacitors.

Keywords: hybrid structures, sensors, carbon nanowalls

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Tritium release from submicronic uncontrolled tungsten dust shapes with/ without microwave plasma treatments

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Abstract

Tungsten (W) is a well-known material [1], with properties which makes it suitable for large scale usage, e.g., in ITER, the future fusion facility [2]. Hereon, during ITER operation, thermonuclear plasma will interact with the inner walls, producing dust with various morphologies. In this line, the current abstract deals with Tritium (³H) release from submicronic (diameter below 600 nm) uncontrolled tungsten dust shapes, with or without microwave (2.45 GHz) plasma treatments. Briefly, ³H gas loading in W dust implies a 2-steps process: the first one reduces oxide from dust, by exposing them under Hydrogen (¹H) atmosphere, using heating conditions, and with cold trap, during four hours. In the second step, W dust are exposed under ³H gas atmosphere, under heating conditions, during two hours, before being frozen in liquid nitrogen. This protocol for ³H gas loading inside dust was used in previous paper [3].

In addition, we have studied the ³H release activity, from tritiated W dust at different ³H pressures. Herein, W dust were exposed to ¹H atmosphere for oxide reduction, followed by ³H gas loading under three pressures: 40 mbar, 100 mbar and 400 mbar. We have then studied the impact upon dust, during the oxide reduction step, by comparing ³H gas released activity from oxide-reduced dust under ¹H gas atmosphere versus ¹H plasma treatment. Herein, both oxide-reduced dust were exposed to 100 mbar ³H gas.

Afterwards, in both experiments, with or without plasma treatment, the ³H released activity from W dust was investigated via room temperature desorption, during 12 days, and final dissolutions [3]. Additionally, SEM and XPS analyses were performed upon W dust to observe the changes induced by the microwave plasma treatment.

Briefly, it can be concluded that, by exposing W dust to higher ³H pressures (400 mbar), the ³H gas release is higher and in two waves. On the other hand, by reducing the oxide via ¹H plasma, one can produce additional smaller morphologies at the surface of the treated dust, which are responsible for increasing the ³H gas loading/ releasing.

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Material- and gas-dependent changes induced by atmospheric pressure plasma treatments on metallic surfaces

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Atmospheric pressure plasma modifications include surface cleaning, etching, functionalization, grafting, and polymerization on different surfaces, including metals [1].

In this contribution we report the influence of atmospheric pressure plasma treatments on metallic surfaces (e.g., duralumin and stainless steel) with different initial roughnesses, obtained by abrasive grinding. The experiments were performed using a plasma jet/torch operated with a radiofrequency power supply (13.56 MHz). The working parameters were kept constant (gas flow: 15000 sccm, power: 250W, and the number of scans: 20), while two types of gases were tested (compressed air and N₂).

The surfaces were analyzed by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), profilometry, and contact angle measurements.

The results indicated that the surface morphology is gas-, roughness-, and material-dependent. For instance, no significant morphology changes were observed for the stainless steel samples after compressed air plasma treatments. However, different morphologies were observed on duralumin samples for the same working conditions, when compared to the initial samples. On duralumin surfaces, the N₂ plasma treatments determined a higher increase of the surface roughness (up to 0.3 μm for the roughest initial surface) than compressed air treatments. The atomic percentages of surface chemical elements obtained by EDX were also gas-, roughness-, and material-dependent. The contact angle measurements showed that, on duralumin aged surfaces, the wettability is increased by N₂ plasma treatments and decreased by compressed air plasma treatments. In this case, the most hydrophobic surface (120.35°) was obtained for the sample with intermediate roughness. However, on stainless steel surfaces, the contact angle values corresponding to the compressed air plasma-treated samples were lower after the aging process (53°- 84°) than the values obtained before plasma treatments (71°-103°).

The results have relevance for plasma cleaning, oxidation and nitridation treatments, and ability of cold welding of metallic surfaces.

Keywords: atmospheric pressure plasma, surface cleaning, surface treatment, metallic surfaces;

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In-liquid plasma synthesis of Ag Np by means of a filamentary jet discharge

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The unique properties of nanoparticles [1], such as high catalytic activity, cohesive energy, electrical, magnetic, optical, biological properties, make them desired materials for utilization in several fields as sensors or biomedical applications. Besides chemical synthesis routes, physical synthesis methods were also developed, among which some implying plasmas, like sputtering, arc discharge, and more recent cold atmospheric pressure plasma sources [2].

In the present work, we demonstrate the successful utilization of a filamentary jet operating in liquid, for the synthesis of Ag Np starting from solutions of silver nitrate at various concentrations, in the range 0.05 – 20 mM. The plasma is ignited in Ar, at 3000 scm flow, upon supplying an RF power of 100 W, and the formed plasma jet is introduced in the AgNO₃ solution, and kept for time intervals in the range from 1-20 min.

The morphology and dimension of Ag NP were evaluated by means of Scanning Electron Microscopy and Dynamic light scattering (DLS), while the composition was determined using X-ray Photoelectron spectroscopy (XPS) technique. The optical properties of the solutions containing Ag NP were determined by means of UV-Vis absorption spectroscopy.

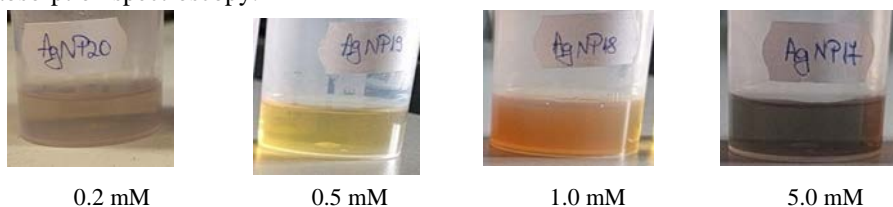


Figure 1. Solutions containing Ag NP as resulted upon plasma treatment using various concentration of silver nitrate

Keywords: silver nitrate, Ag nanoparticles, in-liquid plasma, filamentary jet.

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Topic 4

Plasma applications in environment, biology, medicine and agriculture

Silver containing transparent films on polymer substrates for antimicrobial applications

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Silver and silver nanoparticles are among the usual choices for antimicrobial application, due to their known effect of eliminating or reducing a wide variety of pathogens [1,2]. It's efficiency is conditioned by the amount of active silver that is exposed to the contaminants and the correspondent Ag ion release. For the specific case of transparent coatings, one has to find the balance between the minimal amount of silver that provides sufficient antimicrobial activity and the maximum amount that maintains a good transparency of the coating. These characteristics can be incorporated by including Ag nanoparticles into a transparent oxide matrix [3].

Magnetron discharge was used to obtain very thin transparent films, few tens of nm, deposited on flexible self-adhesive polymers. The amorphous oxide matrix consisted of either SiO₂ or TiO₂, and was obtained by RF sputtering from oxide targets. The silver target was operated in HiPIMS regime in a co-sputtering configuration, so that the Silver is distributed evenly throughout the whole thickness of the coatings. The deposition conditions were chosen so that the metal flux is significantly lower than that of the oxide, with equivalent deposition rates that are typically 10 times smaller. Using low repetition rates of HiPIMS pulses it is possible to control the metal flux to the substrates down to equivalent deposition rates of 0.1nm/min. This allows the incorporation of a finely tuned quantity of Ag into the growing film. The presence of Ag-NP into the film and its characteristics were investigated by various techniques. AFM provides information on the topography of the coatings and their preferential growth on a textured polymer foil. The presence of Ag in the films and the dependence on process conditions was investigated by SEM-EDX and XRD. The preferential absorption of the films in the 400 to 500 nm range are indicative of silver surface plasmon resonance. The position of the peak was tuned both by incorporating the silver in a different matrix, either SiO₂ or TiO₂, or by adjusting the amount of silver in the coating. The overall transmission in the visible range was kept above 80%, making the thin films suitable as transparent coatings with antimicrobial properties.

Keywords: silver nanoparticles, magnetron sputtering, transparent thin films, antimicrobial coatings

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In vitro analysis of the cytotoxic effect of ITER-like tungsten nanoparticles on dermal fibroblast cells

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Based on the current configuration of the International Thermonuclear Experimental fusion reactor, tungsten (W) has been chosen as the plasma-facing material for the divertor component. Nevertheless, during operation, the expected power and temperature of plasma can trigger the formation of W dust in the plasma chamber. According to the scenario for a LOss of Vacuum Accident (LOVA), in the case of confinement failure, the dust can be released into the environment, causing occupational or accidental exposure [1, 2]. In order to undertake the cytotoxicity studies, fusion devices relevant dust have been produced on

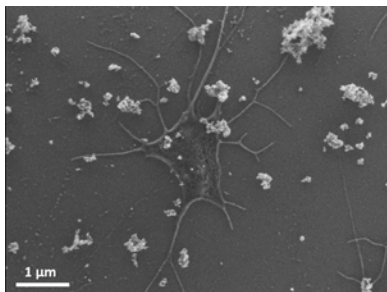


Figure 1. Scanning electron microscopy image of BJ ATCC cells following 24 h incubation with 400 µg/ml of 30 nm W nanoparticles

purpose using a gas aggregation cluster source based on a magnetron sputtering discharge [3]. We synthesized two different size nanoparticles, corresponding to 30 nm, respectively 100 nm in diameter. Possible toxic effects were studied with a normal human skin fibroblast cell line (BJ ATCC CRL 2522) dosed with different concentrations of the produced tungsten nanoparticles [4].

Two cytotoxicity assays with different sensitivities were used, depending on the mechanism which leads to cell death: MTT assay for metabolic activity and ViaLight for the assay of cellular ATP. Optical and scanning electron microscopy were used for direct observation of BJ cells and nanoparticles during their interaction (Figure 1). It was concluded that, in the interaction

with BJ human fibroblasts, smaller (30 nm) W nanoparticles showed lower toxicity compared to larger ones (100 nm).

Keywords: tungsten, nanoparticles, cytotoxicity, plasma, fusion

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Augmented biocompatibility properties of PET and PET+TiO₂ foils after He plasma jet exposure

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Polymeric materials are important nowadays in many fields, going from bio-medicine, food to textile industry. A convenient method for tailor polymer surface properties is the usage of non-thermal, atmospheric pressure plasma discharges. Present study relies on the direct usage of an atmospheric pressure, low temperature, sinusoidal He plasma source, based on a cylindrical dielectric barrier discharge, for augmenting the biocompatibility properties of poly(ethylene terephthalate) (PET) and poly(ethylene terephthalate) with titanium dioxide (PET+TiO₂) foils. The plasma jet source was diagnosed by electrical and optical methods. We used a 2 SLM He flow through the discharge tube and applied a 12 kVpp sinusoidal voltage on the discharge electrodes, at 48 kHz, keeping a gap of 5 mm between the discharge tube and the sample.

Commercial PET and PET+TiO₂ foils (100 μm thick), carefully cleaned with distilled water and ethyl alcohol prior to experiments, were plasma exposed for 60 s on both sides. Atomic force microscopy, static contact angle method and ATR-FTIR spectroscopy were used for polymer characterization prior and after plasma treatment. The biocompatibility of untreated and plasma treated polymer samples was tested using Albino rabbit primary fibroblast cells. The cells were cultured for 24 / 48 / 72 h on the PET / PET+TiO₂ foils, the cell viability being assessed using 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide assay. The cellular adhesion onto the treated / untreated polymer samples were also studied. Cell staining was done using Calcein-AM and Giemsa dyes (Sigma Aldrich), and an inverted laboratory microscope (Leica DM IL LED) was used for imaging the cells onto the polymer under investigation.

The characterization of jet plasma source revealed an up to 50 W mean power discharge, which has in the global emission spectrum along He lines also bands and lines of reactive species such as: OH radicals, N₂, N₂⁺, O. The polymer characterization revealed good surface properties after plasma exposure: increased roughness, water work of adhesion, as well as surface oxidation. No cytotoxic effect of the untreated/treated PET / PET+TiO₂ was observed. Increased cell adhesion was found on the treated samples in respect to the untreated and control ones.

These experimental findings, proven by the good correlation of the obtained results from plasma diagnosis, surface characterization, along with cell viability and adhesion tests, indicate the usage of plasma sources in polymer processing for biocompatibility purposes.

Keywords: plasma physics, biomaterials, plasma medicine, plasma-bioengineering

Antimicrobial and biocompatible nanocomposites based on chitosan and Ag NPs obtained by atmospheric pressure dielectric barrier discharge plasma treatment

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On the way to develop advanced wound healing patches, the utilization of Ag nanoparticles, known for their antimicrobial effect [1], and of natural polymers [2], with minimum environmental impact, are among the most novel approaches. This work presents a method for obtaining antimicrobial and biocompatible Ag NPs/chitosan nanocomposites by using an atmospheric pressure plasma source based on a dielectric barrier discharge. Non-woven polyester textiles were used as substrates, identified as appropriate for development of medical patches. A linear DBD plasma source working in radiofrequency (13.56 MHz) at 100 W in a continuous argon flow of 3000 sccm was used for ensuring superhydrophilic behavior of the substrate, and plasma induced graft polymerization (PIGP) of chitosan, with or without Ag nanoparticles inclusion (30 nm diameter, concentration in the range 0.05 – 5 %), was performed either in single step or in multiple steps, in order to allow different loading of the patches with active substances. The antimicrobial effect of the dressings depending on the Ag NPs concentration and degree of loading was proved against a wide range of strains, including *S. Aureus*, *E. Faecalis*, *P. Aeruginosa*, *E. Coli*, and *C. Albicans* yeast. The study of the compatibility and cell proliferation of the dressings was tested for fibroblast and keratinocyte cell lines. The results show that is a window of parameters, with moderate Ag NPs loading and low number of PIGP procedures, which allow the obtaining of both antimicrobial and biocompatibility effect of the dressings.

Keywords: AgNPs/chitosan nanocomposites, drug loading, antimicrobial effect, biocompatibility

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Organophosphorus toxic compounds degradation in aqueous solutions using single filament DBD plasma jet source

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Pesticides are used in agriculture to reduce pest infestation for increase production. However, pesticides are one of the main environmental problems, especially for water pollution. Pesticides in drinking water may cause long term chronic diseases. For instance, Dichlorvos and DMMP (Dimethyl methylphosphonate), which are organophosphorus toxic compounds used as insecticide and simulant of neurotoxic chemical war agents or flame retardant, respectively, affects nervous system of human body. They can cause ADHD (Attention deficit hyperactivity disorder) and disorder of DNA growth. In principle, the OPEs are a class of organophosphorus compounds used as insecticides and herbicides which are harmful to human nerve cells in case of inhalation, ingestion, and dermal absorption [1].

Atmospheric pressure plasma treatments are considered a promising method for water decontamination, with low environment effects, and low costs based on increased efficiency. In this contribution, we used a dielectric barrier discharge (DBD) filamentary plasma jet source which is very versatile, because they can operate in open air within a large domain of parameters (with various admixed gases types, flow rates, and radio frequency power). These DBD plasma sources were studied previously for decolorization of methylene blue solutions [2]. In the present work, we report the application of filamentary plasma jet for the cleaning of waters intentionally contaminated with Paraoxon, Dichlorvos, Parathion, Reldan, Novadim (Dimethoate) and Dimethyl methylphosphonate (DMMP). In order to prove the efficiency of this plasma source for organophosphorus toxic compounds degradation, the initial and decontaminated mediums were characterized by gas chromatography - mass spectrometry (GC-MS). After plasma treatment most of the pesticide solutions had a degradation rate over 90% and the resulting compounds are less toxic than initial solutions (eg, phosphoric acid which is also frequently found in many food and drink).

Keywords: wastewater decontamination, filamentary plasma jet, pesticides degradation

Acknowledgement: This work was supported by project number PN-III-P1-1.2 PCCDI-2017-0637/33 PCCDI-MultiMonD2 within PNCDI III.

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Determination of HMF Contents and Sugar Composition of Cold Plasma Applied Raisins

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The Maillard Reaction is the reaction between reducing sugars and amino acids during the heat treatment of foods. The reaction is affected by processing and storage conditions such as temperature and food content [1]. In general, 5-Hydroxymethylfurfural (HMF), which is formed as a result of the Maillard Reaction, is used as a quality factor in many heat-treated food products. High HMF concentration has been reported to have harmful effects on humans and is negatively related to the safety of the product. In addition, due to the dangerous effects of HMF on human health such as genotoxic, cytotoxic, mutagenic and carcinogenic, there is a legal limitation for some foods such as fruit juices, molasses. Heat treatment applied to fresh grapes used for raisin production is effective in terms of furfural formation [2]. In this study, the effect of surface temperature on HMF formation during cold plasma treatment applied to raisins for microbial decontamination or detoxification after drying was investigated. For this reason, the color values, HMF contents, sugar composition of raisins applied cold plasma at different times were determined by HPLC and evaluated in terms of food safety.

Keywords: Cold Plasma, HMF, Maillard, Sugar Composition

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Determination of Free Radical Formation in Dried Figs with Cold Plasma Treatment and Evaluation in Terms of Food Safety

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The basic principle of action of cold plasma technology, which is generally used for microbial inactivation in the food industry, is mainly related to the formation of reactive species and their effects on microorganisms. Depending on the parameters of the process and the gas used, a wide variety of reactive species (UV photons, charged particles, free radicals, and oxidants) are produced that contribute to the antimicrobial activity and its successful use in fresh and dried food products. Electron spin resonance for free radical measurement has been used to measure free radicals produced in plasma-treated liquids [1], but the use of the technology in real foods has not been reported. It is important for food safety to determine the concentration and structure of free radicals produced by cold plasma treatment in relation to undesirable changes in processed foods [2]. For this reason, in this study; the free radical structure and concentration of dried fig samples, which were plasma treated at different times, were determined and evaluated in terms of food safety.

Keywords: Cold Plasma, Free Radical, Fig, Food Safety

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Workshop

Invited lectures

Reactive oxygen and nitrogen species in plasma activated water: tuning their concentrations and functions in plant growth promotion

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Non-thermal (cold) air plasmas in interaction with water induce chemical activation and generate *plasma activated water* (PAW). PAW typically contains various reactive oxygen and nitrogen species (RONS), especially long-lived H₂O₂, NO₂⁻, NO₃⁻. Their concentrations and ratios depend on the plasma discharge and dissipated power (e.g. low power corona or DBD vs. high power spark, glow or gliding arc discharges), the gas flow system (e.g. open air vs. confined volume) and the way of interaction with water (e.g. bulk treatment vs. aerosol). Selection of these discharge/gas flow/water interaction parameters enables us to tune the concentrations and ratios of RONS in PAW for various applications [1]. Furthermore, the water solution characteristics, its pH, buffering capacity and storage temperature strongly influence RONS concentrations upon delayed application.

PAW has been successfully demonstrated to stimulate germination and plant growth [2-4]. These effects depend on the RONS concentrations and the way of application, e.g. seed imbibition in PAW, plant watering or foliar application [3-4]. We show that these effects also strongly depend on plant species. Using different types of PAW generated by glow discharges with bulk water or by transient spark with water aerosol, exhibited a positive effect on amylase activity of pea seedlings and did not inhibit seed germination, seedling length, total protein concentration or protease activity. PAW caused only moderate oxidative stress that was effectively alleviated by natural plant antioxidant enzymes (SOD, G-POX, CAT). In pea seedlings, we observed a faster turn-over from anaerobic metabolism (related to imbibition) to aerobic metabolism. RONS contained in PAW did not affect the DNA integrity. On the other hand, the high DNA damage in barley and the reduced root and shoot length and decreased amylase activity was attributed to the oxidative stress caused by PAW, which was exhibited by enhanced activity of G-POX or alcohol dehydrogenase related to grain suffocation [5].

Besides influencing the physiological plant responses upon exposure to PAW, significantly increased plant growth parameters and harvest production were detected in peas and maize. PAW of controlled properties represents a high application potential in farming.

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Plasma-based water treatment in agricultural applications

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This contribution concerns recent advancements in our research on the application of plasma-based water treatment in the agrifood field. Specific issues addressed include the decontamination of water from poly- and perfluoroalkyl substances (PFAS) and from pesticides.

Contamination of groundwater with PFAS is affecting wide areas worldwide, including Veneto region in northern Italy, where the University of Padua is located and where a major international company, now closed, has operated with PFAS for over half a century. These compounds are taken up by plants and vegetables and bioaccumulate in human blood. Due to growing indications of possible damage to human health caused by PFAS [1], the use of well water for irrigation and animal watering has been restricted in contaminated areas, promoting instead the use of tap water for these purposes or the installation of filters to purify the well water before use. Filters based on granular activated carbon are indeed the most diffused means for PFAS removal from water. They are effective but have a limited lifetime, are not easily regenerated and become thus a troublesome waste to be disposed of [2]. To overcome these limits, new technologies are urgently needed to achieve not only the removal but also the degradation of PFAS in water. Atmospheric plasma is particularly promising for this application [3]. A self-pulsing discharge reactor specifically designed for PFAS degradation was recently developed in our laboratory [4] and tested successfully in the degradation of perfluorooctanoic acid (PFOA). The investigation on the reactive species and on the intermediate products formed during the treatment and analysed by liquid chromatography coupled to mass spectrometry was then completed to achieve a comprehensive mechanistic picture of the complex chemistry occurring in this system [5]. Improvement of the reactor design and use of combined approaches of plasma with catalysts are currently investigated in our laboratory to increase the energy efficiency of the process and decrease the treatment time.

Other important targets in the application of plasma-based water treatment in agriculture are pesticides, which are often persistent organic compounds. Aqueous solutions containing pollutants belonging to different classes of pesticides, such as herbicides and insecticides, were thus subjected to treatment in different plasma reactors. The effects of major experimental variables on the degradation kinetics and on the products of the process were investigated. The results allowed us to describe the chemical reactions responsible for the degradation of the various contaminants.

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Cold atmospheric pressure plasma applications along the entire food production chain: examples and perspectives

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1. General Aspects

When the high hydrostatic pressure process was introduced for the minimal processing of food, pressure was also referred to as the "third dimension", in addition to the important process variables of temperature and time [1]. With cold atmospheric pressure plasma (CAPP), the so-called "fourth state of aggregation", a further area of application can now be opened up for food processing, in addition to the states of aggregation solid, liquid and gaseous that have been considered so far. The lecture is intended to identify the potential and limitations of various plasma technologies for the preservation and modification of food.

2. Short Abstract

The presentation will include a thematic introduction to the topic of cold atmospheric pressure plasma application and summarises the important fundamental aspects related to plasma application in food processing. The introduction of a new decontamination technology in industry requires understanding of the mechanisms of microbial inactivation and the associated interactions between process and product. Therefore, experimental studies will be presented with the following objectives: i) to investigate the underlying inactivation mechanisms in plasma treatment of microbial contaminants; ii) to clarify how the different process- and product-related parameters influence the inactivation process; and iii) to evaluate the treatment effects on relevant quality parameters of plant products (exemplary for food). The individual aspects follow a comprehensive overall approach, from the targeted inactivation of microorganisms on temperature-sensitive food systems to the customised influence of quality attributes along the entire food value chain. The multi-scale approach takes into account the influence of cold atmospheric pressure plasma on molecules, macromolecules, single cells, complex plant food systems and food processing equipments [2]. Finally, the future perspectives for the transfer of plasma applications to industrial food production will be discussed.

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From Bench to Prototype for Fresh Produce Processing with Non-thermal Plasma

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1. General

The diversity of microbiological safety and quality challenges in addition to the persistent issue of antimicrobial resistance drives the research agenda and technological development for sustainable fresh food production and processing. Consumer demand for less processed goods provides further rationale for effective decontamination methods that can maintain produce quality characteristics and that can provide shelf-life advantage in the context of increasingly complex food chains.

Atmospheric cold plasma (ACP) has found increasing attention in the food processing sector, with successful and wide-ranging decontamination applications demonstrated. The concept of non-thermal plasma processing has reached a state of knowledge where industrial prototypes have been developed in cooperation with industry stakeholders.

This presentation outlines the key issues for the fresh produce sector, and how these have been addressed at bench scale, and how this has translated to prototype development. This talk will discuss how an iterative process between bench and prototype scales can promote scale up and successful adoption of gaseous and liquid mediated cold plasma technologies. The reported antimicrobial efficacy, the nature of the risks, as well as the importance of considering the mode of delivery and stage of implementation compatible with food processing unit operations are highlighted.

Keywords: Fresh Produce, Critical Process parameters, In Package, Plasma functionalized Liquids, Biofilm, Safety

Dry atmospheric plasma priming: a sustainable approach to improve seeds germinative parameters

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Seed-stacked dielectric barrier devices (S²DBD) operating at atmospheric pressure are processes well suited to agricultural issues, especially to improve the germination parameters of seeds such as vigor and germination rate but also to release their dormancy or reduce their pathogen charge. The efficiency and the sustainability of these biological effects depend on several properties including (i) reactive species generated in the plasma phase, (ii) the plasma-seed interaction and (iii) the amorphous solid states of the seeds.

First, the question of the reactive chemistry will be discussed in the case of S²DBD supplied in helium (1slm) with/without reactive gas (O₂ or N₂). Based on MS, OES and WCA measurements, the role of the main reactive species will be highlighted as well as the mechanisms bridging gaseous chemistry with surface chemistry. Besides, we will show why the He-N₂ plasma drives to the most promising results (in comparison with those obtained using a He-O₂ gas mixture). The sustainability of the plasma-triggered biological effects will be also demonstrated through a 2-months ageing study, hence supporting the relevance of the process for technological transfer towards seed companies.

Second, the question of the plasma-seed interaction will be discussed, and its crucial role will be demonstrated through an equivalent electrical model of the S²DBD (based on Peeter's model). Complementarily, the role of the plasma-seed contact surfaces in inducing stronger biological effects will be deciphered through a solid modelling of the seeds stack, enabling the distinction between areas of the seed-seed contact surfaces and of the seed-wall contact surfaces. We will explain how the characterization of these contact surfaces can contribute to design more appropriate plasma atmospheric processes.

Third, the question of the amorphous solid state of the seeds will be discussed in the case of an air DBD treatment. We will show how seeds dormancy can be drastically alleviated by controlling the amorphous solid state of the seeds which can be either rubbery or glassy. Two distinct methodological approaches will be compared: modulation of seeds water content and modulation of seeds temperature upon plasma exposure.

The molecular mechanisms involved in plant response to stress induced by seed exposure to cold plasma

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The potential of seed irradiation with low temperature plasmas or cold plasmas (CP) for increasing agricultural production is under intensive investigation. Numerous effects on seed microbial contamination, germination and seedling growth have been reviewed recently [1-4], and the accumulated body of evidence indicates to the high complexity of seed response to CP treatment on the molecular level. The overview of the most recent findings on the molecular changes induced in dry or germinating seeds and in growing plants will be provided in the talk, including the results of our research group.

Earlier studies on effects of seed exposure to CP were focused mostly on microbial decontamination, changes in seed surface wettability and germination. Certain later studies have demonstrated enhanced plant growth for the entire vegetation period resulting in increased production yields, indicating to the persistence of CP effects on much longer time-scale due to involvement of complex molecular responses in growing plants. The most important novel findings on such response induced in dry seeds are changes in: 1) EPR signal; 2) DNA methylation; 3) protein carbonylation; 4) balance of phytohormones; 5) expression of genes; 6) expression of proteins; 7) enzyme activities; 8) seed microbiome. CP-induced changes in ROS production were reported in the germinating seeds. The events of seed response to the CP stress signal are further developed in the growing plant as multiple inter-related changes in: 1) gene expression due to DNA methylation; 2) protein expression; 3) enzyme activities (including enzymes of photosynthetic system and enzymes of secondary metabolism); 4) changes in secondary metabolism that followed by 5) a modified plant communication with microorganisms (both pathogens and plant growth promoting microorganisms, e.g. N-fixating rhizobacteria).

Thus, CP induces significant and complex changes in plant metabolism. Upregulated photosynthesis results in better plant growth, stimulated secondary metabolism leads to better plant establishment, fitness and stress resistance. CP effects “beyond plants” (plant communication with microorganisms) are important for improved agricultural performance. The persistence of CP effects implies that CP induces complex mechanisms of plant adaptation that can be exploited for Plasma in agriculture goals.

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Keywords: cold plasma; DNA methylation; phytohormones; proteome; seeds.

Plasma activated water as disease resistance inducer in plants

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1. General

Current global food sustenance by intensive agriculture is mainly based on economic crop monocultures that, however, drastically reduces the biodiversity, increasing the yield losses due to the presence of biotic and abiotic stresses. A technology based on plasma activated water (PAW), characterized by the presence in liquid of reactive oxygen and nitrogen species (RONS), was tested to try to ensure yield stability also enhancing the plant resistance responses and to promote an eco-sustainable management of plant diseases. The exposure of sterile distilled water (SDW) to a cold atmospheric pressure plasma (CAP) causes a reduction of pH and the production of RONS, that induce plant defense responses. The use of PAW for the treatment of infected plants is developed and applied with the design, production, optimization and characterization of different CAP sources. The use of PAW on micropropagated shoots and plants in orchards and in greenhouse cultivation systems to evaluate its effectiveness as pathogen resistance inducer was exploited.

2. Studied cases

The effects of PAW applications were tested on tomato plants experimentally inoculated with *Xanthomonas vesicatoria* (Xv), phytoplasma infected periwinkle micropropagated shoots and plants, and grapevine plants in greenhouse and in vineyards. Quantitative (q)RT-PCR analyses allowed to determine the transcription level of genes involved in the plant defence response (phenylalanine ammonia-lyase, pal) and in the plant phytoalexin metabolism of PAW treated plant materials. The number of leaf spots caused by Xv in tomato plants and the number of symptomatic grapevine plants in vineyards were significantly reduced by the treatments [1]. Transcriptional and post-transcriptional molecular analyses highlighted the PAW's ability to enhance the expression of genes encoding the main enzymes involved in the phytoalexin biosynthetic pathway (alkaloids and stilbenes) and to modulate some of the stress response genes through miRNAs regulation [2]. The PAW ability to enhance some of the plant defence mechanisms, also improving the health status of the treated plants, was experimentally demonstrated. The main results indicated the suitability of using PAW to reduce the disease severity, induce plant resistance both in open field and greenhouse, improving the plant healthy status and fruits yield production [3].

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Plasma Treatment of Growing Plants – Effects on Plant Growth, Development and Stress Responses

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In the last decade, effects of cold atmospheric pressure plasma (CAPP) and plasma treated water (PTW) generated by CAPP on plant germination as well as on plant growth and developmental processes have been extensively studied for various kind of plant species ranging from crops, herbs, vegetables to ornamental plants. CAPP generated with air contain ions, electrons, neutral atoms and molecules, and a set of various reactive nitrogen (RNS) and oxygen (ROS) species. Depending on the specific chemical and physical environment, RONS have different half-life times and are converted to more stable chemical states, e.g. in water to hydrogen peroxide (H₂O₂), nitrite (NO₂⁻) and/or nitrate (NO₃⁻) ions. Gaseous and aqueous plasma-generated RONS can react with biomolecules of biological systems. Chemical modification of biomolecules alter its stability, activity and physiological function and can provoke cellular responses resulting in physiological changes affecting developmental processes and stress responses of the whole plant.

In two initial approaches, three kinds of plasma treatment modes were applied to blue lupin (*Lupinus angustifolius* L.) to evaluate plasma effects on biomass production, biochemical parameters and stress response. In the first approach, lupin roots (watering) or shoots (spraying) were treated with PTW that was generated by using a pin-to-liquid or a gliding arc discharge device. Interestingly, PTW treatment of shoots by spraying for five days resulted in higher biomass parameters after 4-5 week of growth. Biochemical parameters such as total chlorophyll content, soluble protein content or catalase activity were essentially unaltered. In the second approach, the upper shoot part including the shoot apex of lupin plants were exposed for 2 min to a surface dielectric barrier discharge reactor (SDBD). Shoots were harvested five hours after treatment and extracted soluble proteins were subjected to MS-based proteome analysis. 5608 proteins were identified in soluble protein leaf extracts. Upon those, 287 were down-regulated and 423 were up-regulated after plasma exposure. Several up-regulated proteins were assigned to secondary metabolism and to abiotic stress responses, while a set of down-regulated proteins were referred to primary metabolism, growth regulation, protein expression and turnover machinery.

In addition to RONS, other factors, such as the electromagnetic field generated by the SDBD source, may have led to the observed stress response in blue lupin. Results of both approaches are discussed in terms of the current state of knowledge and possible future approaches to plasma treatment of growing plants for potential stress priming.

Keywords: cold atmospheric pressure plasma, plasma treated water, blue lupin, biomass and biochemical parameters, soluble leaf proteom

Oral presentations

Removal of mixtures of pharmaceutical pollutants in aqueous solutions using non-thermal plasma

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Water pollution is a serious problem of the modern world due to the increasing prevalence of contaminants of emerging concern, especially pharmaceuticals. Their increased usage and resistance to conventional treatment lead to widespread environmental dissemination [1], [2]. New technologies are needed to remove these pollutants. One of the most promising approaches is non-thermal plasma treatment, capable of degrading a variety of organic pollutants [3], [4]. The objective of this paper is to investigate the plasma degradation of mixtures of pharmaceutical compounds.

A pulsed corona discharge above liquid was used for the removal of a mixture of amoxicillin (AMX), diclofenac (DCF) and ibuprofen (IBU). The pulse duration (110 ns), frequency (25 Hz), voltage (18 kV) and discharge gas (O₂, flow rate 300 mL/min) were maintained constant throughout the experiments. The plasma reactor was coupled with an ozonation reactor in order to use the excess ozone generated in the discharge [5]. A volume of 330 mL of contaminant solution (0.1 mM AMX, 0.1 mM DCF and 0.1 mM IBU) was circulated between the two reactors. The conductivity of the solution was adjusted to 300 μ S/cm with different salts (Na₂SO₄ and NaHCO₃), and the initial pH was 7.7-8.

Complete removal of AMX was achieved after 5 min of treatment with NaHCO₃ and after 10 min with Na₂SO₄. DCF and IBU were almost completely degraded after 10 min and 20 min, respectively, regardless of the salt used. However, their degradation appeared to be faster when using Na₂SO₄. The highest mineralization degree achieved was 44% (NaHCO₃, after 60 min). For comparison, experiments with single contaminant solutions were performed. The concentrations of individual pharmaceuticals were adjusted to match the theoretical oxygen demand of the mixture. The complete removal of single contaminants was achieved slower as compared with their degradation in mixture. Overall, the plasma-ozonation system shows promising results for the simultaneous removal of multiple pharmaceuticals from solution.

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Energy decomposition analysis of organic pollutants in water: a way for finding plasma degradation routes

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1. Methods

Energy decomposition analysis (EDA) [1] aims at determining bond energies inside molecules. If a complex molecule $F1\dots F2$ is expected to dissociate in 2 fragment $F1$ and $F2$, the bond energy (or binding energy) is written as: $\Delta E = E_{F1\dots F2} - E_{F1} - E_{F2}$. $E_{F1\dots F2}$ is the energy of the optimized complex molecule, while E_{F1} and E_{F2} are the energies of the optimized fragments. The bond energy ΔE can be divided in 2 terms: the strain energy which is the energy necessary to optimize geometry of fragments to their optimized geometry in the complex molecules and the interaction energies. EDA is decomposing the interaction energy in three terms: Coulomb interaction, Pauli repulsion, and attractive orbital interactions. The Coulomb interaction, which is usually attractive, is the energy between the unperturbed charge distributions of the prepared fragments. The Pauli repulsion consists in the destabilizing interactions between occupied orbitals of the fragments. The orbital interaction includes charge transfer and polarization.

The bond energy determination calculates all these energies for each component. This is done using Density Functional Theory using the appropriate exchange correlation functions and basis sets. The main limitation is that we must select the possible fragments before running calculations. So, the method has been extended in Interacting Quantum Atoms (IQA) developed in the frame of the so-called Quantum Theory of Atom in Molecule (QTAIM) [2] for obtaining all bond energies in one calculation.

2. Applications to plasma degradation of persistent organic pollutants

We have applied and compared these methods to analyzing bond energies of antibiotics such as amoxicillin and sulfamethoxazole (SMX) for comparison with plasma degradation experiments. The degradation of SMX in water by a corona discharge above liquid was studied experimentally and the plasma-treated solutions were analyzed by LC-MS to identify the degradation products. For short treatment time (2 min), the presence of compounds with higher masses than the parent molecule (m/z 270, 273, 284, 288, 296) suggests the addition of oxygen-based functional groups as a first step in the degradation pathway. Smaller degradation products (m/z 174, 204) resulting from the fragmentation of the SMX molecule are also detected. Since IQA is very powerful for halogens compounds [3], it will be used for addressing pesticide, such as chlordecon and atrazine, degradation by plasma.

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Application of a surface DBD plasma source for flour treatment

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For decades non-thermal plasmas (NTPs) have been used in various industrial processes to modify surfaces of different materials. At first, mostly low-pressure NTP was used for these applications, thus imposing a limitation to the type of the sample. With the development of NTP sources at atmospheric pressure, the number of plasma material processing applications has increased, enabling applications in medicine, biology and recently agriculture and food industry [1]. Cold plasma treatment can change the functionality and improve rheological properties of wheat flour [2] due to the radicals and ozone propagated oxidation of amino acids. Additionally, plasma-created reactive species has demonstrated ability for mycotoxins detoxification. Therefore, in this study we investigated both effects of cold plasma treatments on flour – plasma influence on amino acids in flour as well as reduction of *Alternaria* toxins in contaminated flour.

The experimental setup used consists of a surface dielectric barrier discharge (SDBD) source which is placed on top of a box that contains a flour sample and openings for air. High-voltage sine signal is supplied to the powered electrode positioned at the bottom of a dielectric plate. Before treatments we characterized the source by recording electrical waveforms and by using optical emission spectroscopy to obtain information about excited species created in the plasma. For moisture content analysis NIR Spectroscopy analyzer was used, while for determination of *Alternaria* toxins content high performance liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS) measurements were performed [3]. Measurements of the different types of wheat flour samples showed linkage formation in the gluten-related proteins after plasma treatment, due to reaction with reactive oxygen species formed in the plasma. In flour samples contaminated with different *Alternaria* toxins we found reduction from 4% up to 74%, depending on the type of the toxin and treatment conditions. We used the experimental results for developing and fitting empirical models in the form of the second-order polynomials for prediction of toxin reduction and optimization of the process. Significant differences observed between treated and control samples in this study demonstrated great potential of the NTP DBD source for treatment of the wheat flour.

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The potential of cold plasma for the stimulation of natural sweeteners biosynthesis in *Stevia rebaudiana* Bertoni

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Seed treatment with non-thermal or cold plasma (CP) stimulates seed germination, grown plant morphometric parameters, biomass production, and disease resistance in different plant species by inducing changes in plant biochemical phenotype. The activities of enzymes, the amounts and ratios of different secondary metabolites are markedly changed after some treatments, however, there are still not enough knowledge in molecular mechanisms to control and predict treatment effect.

Stevia rebaudiana Bertoni is an economically valuable plant due to its secondary metabolites steviol glycosides (SGs) that are responsible for the sweetness of stevia and are widely used as natural sweeteners. Stevioside (Stev) and rebaudioside A (RebA) are the most abundant SGs in stevia. RebA is preferred over Stev for better taste (lack of bitterness). We have demonstrated for the first time, that seed treatment by CP can increase Stev and RebA concentrations several times.

The aim of this study was to overview our research group results on the effect of *Stevia rebaudiana* Bertoni seed treatment (2-7 min) with different types of CP (dielectric barrier discharge (DBD) and capacitively coupled (CC) CP) on the amount and ratio of Stev and RebA in the leaves of stevia, the kinetics of stimulated biosynthesis and the possibility to transfer the CP-induced stimulating effect to the vegetatively propagated plants.

Both types of CP had strong stimulating effect on steviol glycosides (SGs) in stevia leaves 8 weeks after germination. CC CP increased the RebA concentration 1.5-fold and the concentration of Stev 7-11-fold depending on treatment duration. The optimal 2-min pre-sowing seed treatment with DBD CP increased the RebA concentration 2-fold, Stev - 14%, RebA/Stev ratio - 1.7-fold. The treatment of longer duration (5-7 min) had lesser effect than 2 min. Stimulating effect persisted 14 weeks (time of cutting for vegetative propagation) but only in 5 min-treatment group and vanished in 20th week (onset of buttonization). Vegetative propagation induced additional stress to plants what resulted in 2-fold decrease of SGs synthesis after 8 weeks. Plants recovered after 20 weeks; however, the CP-induced stimulating effect was lost in vegetatively propagated plants. The concentrations of other bioactive compounds as phenolics and flavonoids were decreased or unchanged by both types of CP treatment resulting in lower or unchanged antioxidant activity of stevia leaf extracts rich in SGs.

It can be concluded that a short time pre-sowing treatment of seeds with CP can be a powerful tool for the enhancement of biosynthesis/accumulation of SG in stevia plants at least for 14 weeks of vegetation. More studies are required to evaluate the possibility of stimulation transfer by vegetative propagation by adjusting cutting time.

Keywords: *Stevia rebaudiana* Bertoni, seed treatment, cold plasma, vegetative propagation

Effects of Surface Dielectric Barrier Discharge Treatment of Some Sprout Species in Different Conditions

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Cold plasmas produced at atmospheric pressure are suitable and have been very much used for processing biological materials such as seeds. It was shown that such a powerful chemical reactor can stimulate the germination and growth among other positive effects. However, the outcome depends on the used source, but most important on the species of the seeds processed using plasma.

In our previous works we focused on establishing the results of direct and with intensified reactive species exposure of some species (*Raphanus sativus* L. var *longipinnatus* – Japanese radish, *Bassica oleranceae* L. var *italica* – broccoli, *Lepidium sativum* – garden cress). The sDBD device used for the experiments has a simple flexible configuration that can be either used for the direct treatment of different surfaces or can be attached to a closed package with the processed materials (in our case seeds or sprouts) placed inside.

Sprouts are becoming popular in the diet of Europeans because of the health benefits brought by bioactive molecules such as antioxidants and enzymes, which are found in sprouts. However, they have a short shelf life and might carry microorganisms harmful for the human consumption. So, there are two directions we turn our attention on: stimulating the growth while studying the mechanisms of plasma-seeds interaction, and increase shelf life and consumption safety. To achieve our goals, we perform a detailed characterization of plasma and of the outcome of plasma treatment starting from seeds (surface morphology imaged with Environmental Scanning Electron Microscopy, wettability – water contact angle measurements, imbibition), germination behavior (germination potential, germination speed), sprouts physical and biochemical properties (biometric measurements, chlorophyll and carotenoid contents, etc.), shelf life of sprouts produced after seeds or sprouts plasma treatment.

Our results show significant differences between species behavior: for some the germination and sprouting is stimulated, for other is inhibited; some species exhibit strong morphological changes of the surface of seeds, with increasing the hydrophilic character, while others show only minor modifications; biochemical parameters are changing for most species, especially when the seeds are treated inside a closed package, probably due to the intensified action of the reactive species that cannot diffuse in the surrounding air but remain in the closed proximity of the samples. In all cases we found an increase of the life shelf of the sprouts.

Keywords: plasma agriculture, cold plasma, sDBD, sprouting, germination.

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Tailoring plasma sources towards plasma agriculture: at the interface with liquids and solids

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Plasma discharges are rapidly gaining importance as versatile tools for material processing, since they are easy to use, technologically simple and environmentally friendly. Applications of these plasmas are widely spread, including: plasma technologies (involved in industry: food to textile, construction to automotive), plasma medicine (e.g.: biology, pharmaceuticals, oncology) and, recently, plasma agriculture; therefore, it is important to characterize and monitor plasma sources from electrical and optical point of view, in order to fulfill the applications requirements.

Our goal is to use atmospheric pressure plasma jet sources to mitigate on one hand the plasma-liquid medium interaction (treatment / production), and on the other hand to optimize the plasma-solid surface interaction in the plasma-seed direction. More precisely, in this report, by using the principle of dielectric barrier discharge, in a cylindrical geometry, we powered up an atmospheric pressure plasma jet (appj) in helium, and used it two scenarios:

1. in the food industry direction (for wine making), we used the plasma source to treat liquid media as follows: treating fresh must (white grape juice) in order to improve the storage / quality of wine;

2. in the plasma seed and plasma activated medium direction, we directly treat seeds with plasma and indirect, by means of plasma activated water (paw), and studied the effects of these interactions upon the plant evolution (from cultivation to harvest).

Furthermore, we used a 2 SLM He flow through the discharge tube and applied a 16 kVpp sinusoidal voltage on the discharge electrodes, at 48 kHz, keeping a gap of 5 mm between the discharge tube and the sample (liquid or solid). The plasma source was characterized via electro-optical diagnosis: electrical methods for applied voltage, discharge current, and power, charge and energy, as well as optical emission spectroscopy.

Plasma-treated medium (must, water or seed) was investigated by means of UV-Vis absorption spectroscopy, ART-FTIR, pH, conductivity, and optical microscopy. The results, as a correlation between the plasma characteristic parameters and treated medium properties, are in the favor of using plasma treatment for activating the medium (PAW) / preserving (wine) or stimulating processes (accelerated germination) in the studied medium. Preliminary results support the plasma treated must procedure in the preservation of young wine (1-2 years old) as well as the promotion of germination of seeds (both for the direct treatment and using paw on seeds).

Further studies, implying more analysis methods should be implicated for a better and perspective view on the topic of plasma agriculture.

Keywords: plasma physics, plasma agriculture, plasma&wine, plasma-bioengineering

Regular contributions

The effect of a cold atmospheric filamentary plasma on seeds of beans (*Phaseolus vulgaris*)

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Because of climate changes conditions, the soil contamination with microorganisms and chemicals, low quality of seeds, the rate of seeds germination and implicitly the crop yield continue to decrease. To overcome these economic problems, non-thermal plasma emerged nowadays as a promising technology in agriculture [1]. Recently, plasma treatment technology has been focused on practical ways to increase the germination percentage, at large quantity [2].

In this study, the effect of the cold radiofrequency (RF) plasma treatment on beans seeds (*Phaseolus vulgaris*) is investigated. Firstly, a preliminary research was made using a singular filamentary jet [3]. The effects of the reactive gas introduced in the Ar discharge and of the applied power were studied. It was observed that the germination was accelerated when a small amount of O₂ was introduced, at lower RF power.



Figure 1. Image of the scaled-up plasma system during operation

Secondly, in order to overcome the laboratory studies and to make the first steps towards field trials (to increase the treated seed quantity), a scaled-up cold filamentary plasma system was successfully implemented for seeds treatment, as presented in Figure 1. The importance of seed shapes was evaluated. Therefore, we treated different shapes, colors and dimensions of the seeds, under identical plasma parameters. Moreover, the importance of plasma treatment time on the germination efficiency was highlighted. These results showed that plasma treatments promote the germination rates and plant growth, which, therefore, can increase crop yields. As such, we can state that this newly developed plasma system can be successfully implemented in pre-harvest agriculture and this system will probably be transformed into an especially useful tool for future field trials.

Keywords: cold plasma, beans treatment, germination, scaled-up system

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The influence of air atmospheric pressure plasma treatment on the germination of *Tagetes erecta* seeds under saline stress

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The applications of atmospheric-pressure plasmas in agriculture grow rapidly. As shown in some recent studies, several methods of treatment are used in agriculture to enhance the rate of germination and growth in plant seeds. A large experimental flexibility is envisaged, as the seeds might be exposed to the plasma active agents either directly, as well indirectly by using the plasma activated water or plasma modification of soils, leading to promising solutions of technological transfer from laboratory to farms.

In this study, we discuss the influence of atmospheric pressure plasma exposure on *Tagetes erecta* 'Petite Yellow' seeds in saline and non-saline solutions. The seeds were exposed to the pulsed plasma generated using a dielectric barrier discharge (DBD), in air at atmospheric pressure. The DBD setup consists of one circular plane electrode, covered by glass and a second mesh electrode, with a discharge gap of 5 mm. The discharge was driven by an AC power supply at 50 Hz and 13 - 16 kV peak-to-peak amplitude. Treatment time ranged from 2 to 5 minutes. In order to monitor some of the reactive oxygen and nitrogen species in the gas phase, during the *Tagetes erecta* seeds plasma exposure, we used the Fourier-transform infrared spectroscopy (FTIR) technique. The following reactive species were identified using gas phase FTIR: HNO₂ (3630-3545 cm⁻¹, 1735-1670 cm⁻¹, 890 - 760 cm⁻¹), N₂O (2265-2145 cm⁻¹), N₂O overlapped with HNO₂ (1330-1210 cm⁻¹) and O₃ (2122 cm⁻¹, 1053 cm⁻¹) [1]. The seed lots (75 seeds/lot) were placed in Petri dishes (25 seeds/plate) with distilled water, 0.1 M and 0.05 M NaCl solutions respectively, at 22 °C, in a light incubator and monitored for 6 days.

We calculated the germination percentage/day (GP), the final germination percentage (FGP) the coefficient of velocity of germination (CVG), the germination index (GI), the germination rate index (GRI), the mean germination time (MGT) [2]. The highest final germination percentage (FGP) [4] in distilled water was recorded for the seeds treated at 15.5 kV for 5 minutes and the highest FGPs in both saline solutions, were recorded for the seeds treated at 13.5 kV for 5 minutes. Our observations prove the positive influence of the atmospheric pressure plasma on the seeds germination capacity and velocity in both saline and non-saline solutions.

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Effect of non-thermal plasma treatment on sunflower seeds

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The rapid growth of world population and consumption surpasses crop production and as a result hundreds of million people are undernourished [1]. Improving sustainability in agriculture requires new and efficient technologies to enhance productivity, while reducing the use of chemicals and their negative impact on environment. Pre-sowing seed treatment by non-thermal plasma is a promising approach, capable of improving seed germination, enhancing the early growth as well as further development of plants, decontamination of seeds, inducing resistance to environmental and biological stress etc. [2-4].

This work investigates the effect of seed treatment with a dielectric barrier discharge (DBD) on sunflower germination and early growth. The plasma was generated in a coaxial DBD reactor with the inner electrode (Φ 21.7 mm) connected at high voltage and the outer electrode (Φ 34 mm, L 230 mm) grounded. The sunflower (*Helianthus annuus* L.) seeds, a semi-early hybrid P64LE99 (Pioneer®), production from 2019, were packed between the electrodes, i.e. in the discharge gap of 4.5 mm, approximately 30 g in each experiment. The discharge was generated in air, with sinusoidal voltage of 16 kV amplitude and 50 Hz frequency. The average power dissipated in the discharge was 5 W, calculated by the Lissajous method [5], and the treatment time was 10 min. The seeds were incubated at 22°C, 60% UR and light/darkness program 8/16 h for seven days at the Institute for Plant Protection Bucharest. Four replications of 25 seeds each were used for the two variants. The germination of seeds on filter paper in Petri dishes, measured for three consecutive days (3rd, 4th and 5th) was 98% both for the untreated and plasma-treated samples.

The radicles, measured on the 5th and 7th day, were shorter for the treated seeds, and the difference was statistically significant (ANOVA, $p = 0.05$). On the 7th day, the seeds were transferred in pots with organic substrate under lab conditions (25°C, 60%UR, 8/16h), where the plant growth was further observed for three weeks. The stem length (from the soil up to the cotyledons) was measured at two-day intervals, and showed significantly higher values for the treated seeds as compared to the control (up to 18-19% difference). The seedling height was also considerably larger for the plasma-treated seeds. On the other hand, the cotyledons dimensions, measured on the 10th day, did not seem to be influenced by plasma exposure. After 30 days, the plants were removed from soil, measured and weighed. The root length and weight were similar for the treated and control samples. However, the total plant weight was significantly increased as a result of plasma treatment, by almost 30% with respect to the control.

Further work is planned to detect the optimum plasma parameters specific for sunflower seeds treatment, as well as to investigate long-term effects of plasma exposure.

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Plasma-induced morphological and biochemical changes in dwarf bearded iris (*Iris reichenbachii* Heuff.) calli

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Expansion of the plasma agriculture and plasma medicine and the demand for precise and localized *in vivo* treatments of living cells and tissues resulted in fast development of various plasma devices that operate at atmospheric pressure [1,2]. Irises can be regenerated *in vitro* by process of somatic embryogenesis and/or organogenesis by formation of shoot or root meristems on calli. During the induction of regeneration process, three types of calli could be distinguished, two friable regenerative calli: white embryogenic and green organogenic and the most abundant yellow, compact, nodular type of non-regenerative calli, designed as non embryonic [3]. Due to its lack of morphogenetic response and/or their low regeneration potential, the regeneration of non-embryogenic iris calli is one of the greatest challenges in this field of investigation. In the current study plant undifferentiated compact tissue (calli) of Balkan endemic dwarf bearded iris (*Iris reichenbachii* Heuff.) was treated using a RF plasma needle device operating with He as a working gas and changes at morphological and biochemical level were investigated. The plasma needle was positioned 3mm above the callus surface enabling direct contact between the active plasma volume and the surface. Direct plasma treatment triggered significant morphological alterations in structure of non-embryonic calli. Observed changes could be attributed to the enhanced cell division of the plant cells at the surface of the compact calli and differentiation of friable calli type stimulated by reactive species formed in the low temperature plasma. Indicated morphological changes were followed by the significant alteration in secondary metabolites in derived different calli types. Our results implicate that direct plasma treatment could serve as a significant elicitor of secondary metabolites production in dwarf bearded iris calli.

Keywords: low temperature plasma, RF plasma needle, calli, iris

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Treatment of *Chlorella vulgaris* by gliding arc discharge plasma

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Microalgae have been identified as a promising feedstock for energy and high-value products [1,2]. A major challenge for extracting valuable compounds (proteins, lipids, polysaccharides or carotenoids) from microalgae is their strong and rigid cell wall. Conventional extraction processes have different disadvantages and limitations which could be overcome by application of various plasma methods [1-2]. The aim of this work was to investigate the influence of plasma treatment parameters on the viability of algae cultivated in different medium.

The gliding arc discharge technology was used for the treatment of *C. vulgaris* cultivated in BG-11 medium and aquaculture wastewater. Compressed air (with total flow rate of ~22.8 l/min) was used as the plasma gas. The distance between the "knife-edge" type electrodes and surface of the algae suspension was 30 mm. The plasma treatment durations were 300 s or 600 s. The plasma treatment of the suspension was performed at various power supply output power values ranging from 35 W to 265 W at frequency of 270 kHz. Chlorophyll A content was measured as an indication of algae viability after gliding arc discharge plasma treatment. Pigment concentrations were determined on the 5th day after the plasma treatment using methanol extraction. Then, the absorbance of samples was measured at 470 nm, 665 nm and 720 nm wavelengths using a UV-visible spectrophotometer [3].

Obtained results showed that the chlorophyll A content in microalgae depends on the treatment duration and the discharge power. The increase in the discharge power and/or treatment duration resulted in a decrease in the pigment concentration. It was demonstrated that the concentration of chlorophyll A in *C. vulgaris* after plasma treatment was reduced up to 50 % comparing with untreated sample.

Keywords: Microalgae, *Chlorella vulgaris*, Air plasma, Gliding arc discharge, Cell disruption.

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Application of plasma activated water for phenol degradation using a gliding arc discharge at atmospheric pressure

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Phenol and phenolic derivatives are common by-products use as antioxidants or synthesis intermediates in significant industrial processes such as in plastics, pharmaceutical and food industries. These toxic pollutants present in water due to the discharge of these human activities are known to induce several damage both on human and animals health acting as carcinogen products and damaging the red blood cells [1]. Then, along the extended available methods, the use of Plasma Activated Water (PAW) as chemical medium for phenol degradation remains a promising technology [2]. In this context, the present work deals with the analysis of phenol degradation in PAW using a gliding arc discharge as plasma reactor at atmospheric pressure. Based on previous works on the characterization of the key species generated in PAW by our plasma source [3], the present research will focus on application for phenol degradation. Then, distilled water solutions ($V=50$ ml) with initial phenol concentrations of 1000 mg.l^{-1} have been exposed to the plasma source for different time treatment (0-10 minutes) using air as feeding gas. Analysis of the water composition after plasma treatment have been carried out by spectrophotometry for semi-quantitative evaluation (cf. figure 1) and by GC/MS after liquid/liquid extraction using dichloromethane for phenol concentration measurements/degradation by-products inventory (cf. figure 2). The results highlights the effect of time experiment on the phenol degradation and by-products formation. Analysis of the GC/MS chromatograms allows to clearly identify the phenol degradation and the by-products composition according to the experimental conditions. In addition, the fall of phenol concentration after plasma treatment have been determined after a calibration step using different phenol solutions at known concentrations.

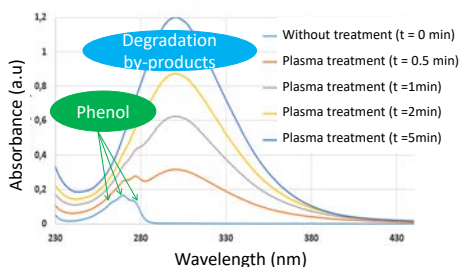


Figure 1: UV-visible spectra as a function of the time treatment.

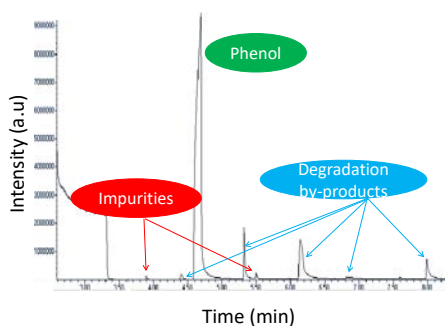


Figure 2: GC/MS chromatogram obtained after plasma exposure.

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Hydrophilization of corn seeds by non-equilibrium gaseous plasma

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Plasma agriculture is a promising niche of interdisciplinary research where the physics, of non-equilibrium gases meets surface chemistry and biological responses. Despite numerous scientific papers, the interaction of gaseous plasma with seeds is not understood enough to make the technique useful in practical agriculture. An obstacle is an improper methodology adopted by different authors. In this paper, we show that the surface wettability does not depend on discharge parameters such as power and pressure, but rather on the fluence of oxygen atoms onto the seed surface. The proper methodology is demonstrated for the case of corn seeds. The surface activation, which enables improved water uptake or good adhesion of a coating, progresses relatively linearly up to the O-atom fluence of $3 \times 10^{24} \text{ m}^{-3}$ and remains constant thereafter. The minimal water contact angle achievable using oxygen plasma treatment is a few degrees.

Keywords: Corn seeds, Fungi, Gaseous plasma, Hydrophilization, Sterilization

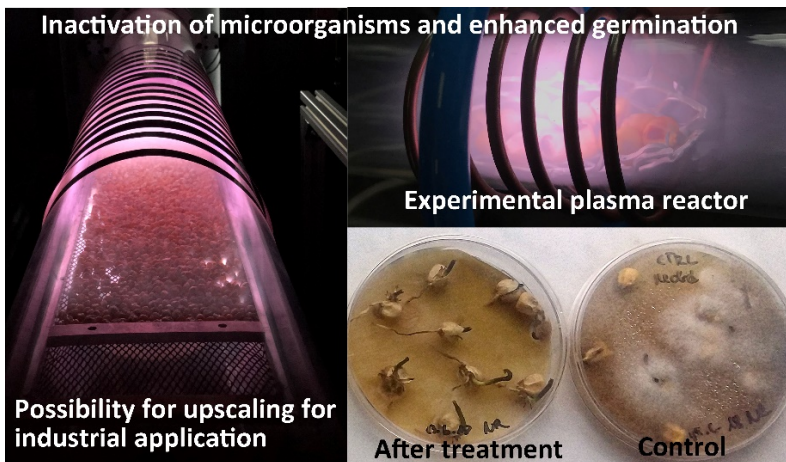


Figure 1: Graphical abstract

***Listeria monocytogenes* dynamics on multispecies biofilms formed on anti-biofilm coatings applied by Non-Equilibrium Atmospheric Plasma on stainless steel**

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Biofilms are considered an important source of microbial contamination on food industry. In a previously published work [1], we described the development and characterization of anti-biofilm coatings applied with an atmospheric-pressure plasma jet system on AISI 316 stainless steel, with the best results obtained against *L. monocytogenes* (*Lm*) biofilms formed at 12°C (90% reduction of biofilm production). The present study assesses the anti-biofilm activity of this plasma-polymerized coating against multispecies biofilms developed from food industry environmental samples, together with the efficacy of conventional sanitizers. The biofilm formation levels on stainless steel with or without the plasma-polymerized anti-biofilm coating after seven days at 12°C was evaluated for (i) a *Lm* three-strain cocktail, (ii) the indigenous microbiota of food-contact and no food-contact surfaces from processing environments of three different industries and (iii) the previous environmental samples artificially inoculated with the three *Lm* strains. In addition, the disinfection effectiveness of a 15-min treatment with sodium hypochlorite and peracetic acid at 0.5% was assessed for the three types of biofilms on both surfaces. The biofilm populations of *Lm* and the total aerobic plate count, before and after disinfection, were enumerated by selective agar plating after the recovery of the cells from the biofilms through scraping with swabs. The biofilm formation by the *Lm* cocktail was approximately 10-fold higher on the uncoated stainless steel than on the coating. However, the anti-biofilm activity of the coating against the multispecies biofilms developed from environmental samples was dependent on the industry, with the coating showing anti-biofilm activity on two of them and a slight pro-biofilm activity on the third one. The growth of *Lm* seems to be partially controlled by the microbiota present in the industrial samples as the biofilm counts are reduced from mean values of 6.0 ± 6.2 log CFU/cm² in the *Lm* mono-species biofilms to 3.4 ± 3.6 log CFU/cm² on the artificially inoculated industrial samples. In these samples, the differences between *Lm* counts on stainless steel with and without coating are not significant, which indicates that the coating anti-biofilm activity against *Lm* might be influenced by the microbiota present in the industrial sample. The disinfection treatments were insufficient to eliminate the biofilms but no *Lm* were detected in most of the cases ($<10^2$ CFU/cm²). These results continue the characterization of this anti-biofilm coating that is successfully applied by atmospheric pressure plasma-polymerization on stainless steel. They contribute to the understanding of population dynamics of mixed-species biofilms of indigenous microbiota and *Lm* under conditions that better resemble the food processing environments where the coating would be applied.

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Wheat seed surface changes after cold plasma treatment

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1. Abstract

The nonthermal or cold plasma technology is a relatively new technology in agriculture. The research focuses on two aspects of this technology: Firstly, it could be used as a new decontamination method of seed material and fresh food products, such as berries [1]. Secondly, cold plasma technology could be used as a tool for seed priming; improving seed germination, seedling growth and potentially even improve crop yield and alleviate abiotic stress from the environment [2–4].

The study's main purpose was to detect changes on seed coat and to evaluate effects on germination rate and malondialdehyde (MDA) content in wheat seeds after cold plasma treatment. Morphological changes on the seed surface after cold plasma treatment were studied by scanning electron microscopy (SEM). The results show that the longer exposure of seeds to cold plasma treatment, seed surface becomes “smoother” compared to untreated seeds, because of the etching effect of plasma components on seed surface. The X-ray photoelectron spectroscopy (XPS) showed altered surface chemistry of seed surface already after 10s of plasma treatment; an increase in oxygen and decrease in carbon content was detected. Slight traces of potassium and sulphur were also detected in seeds after plasma treatment.

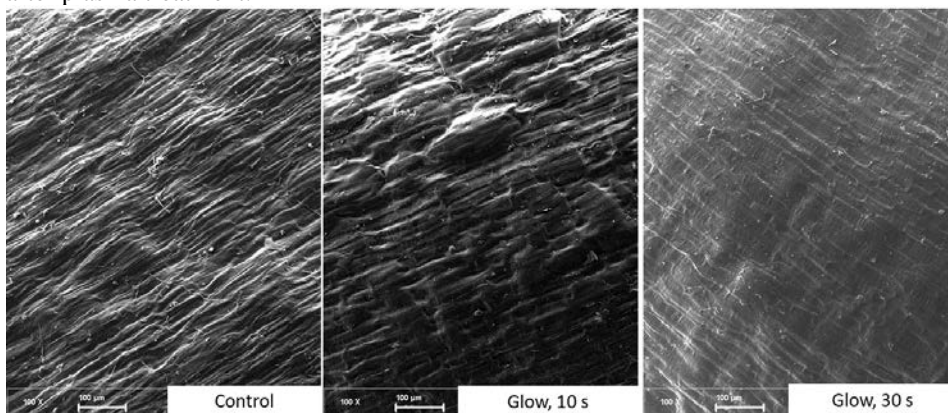


Figure 1: Seed surface of wheat seeds; from left to right: seed surface of control (untreated) seeds, seed surface after 10 s and seed surface after 30 s direct plasma treatment.

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Keywords: plasma, plant, seeds, XPS, SEM

Effect of plasma activated water (PAW) as a fertilizer improving the outdoor plant yield: maize case

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The activation of water by cold plasma induces some chemical changes such as: formation of reactive particles derived from oxygen and nitrogen (hydrogen peroxide, nitrate/nitrite ions, peroxyxynitrite, etc.) [1]. Amongst these reactive particles, Hydrogen peroxide, nitrate/nitrite ions are considered as key species which provide to the PAW an interest in the agriculture field. These key species play an important role for the rapid germination of seeds and, plant growth enhancement by providing nitrogen from the PAW to the plant. Based on the PAW's properties, it could be an interesting source of nitrogen provider for the whole growth cycle (from seed to harvest) [2].

We used two types of PAWs generated by transient spark (TS) discharge with water electropray and by glow discharge (GD) with water cathode, and Hoagland solution. After plasma treatment, each PAW was characterized by measuring long-lived RONS concentrations (H_2O_2 , NO_2^- , NO_3^-) by UV/VIS absorption spectroscopy.

After the harvest, the effect of PAWs generated by the two plasma sources and Hoagland solution on maize yield were analyzed by measuring some growth parameters (plant height, product length, product weight).

As shown in Fig.1 (right), we found that the PAW could be effective for watering the plant from seed to harvest. GD 2 (PAW from glow discharge activated for 2 min) showed a similar result to Hoagland solution (HS) which is a common synthetic fertilizer (micro and macro nutriment). The results show us that the PAW effects during the plant cycle up to harvest depend on the concentration of hydrogen peroxide nitrate/nitrite ions.

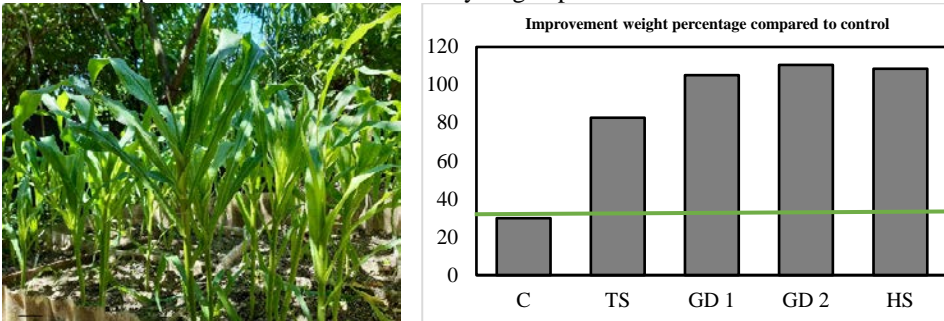


Figure 1: (left) Experiment field, and (right) improvement of the average product weight percentage compared to control (C).

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