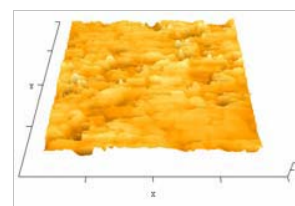
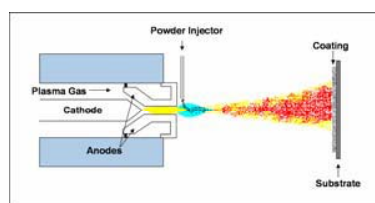
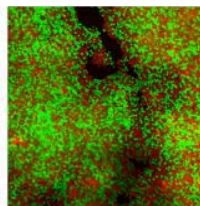
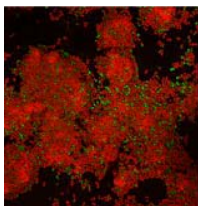


PLASMA PROCESSES FOR BIOMEDICAL APPLICATIONS

BOOK OF ABSTRACTS

ONE DAY WORKSHOP
Thursday 24th May, 2007
National Centre for Plasma Science and Technology,
Dublin City University



National Centre for
Plasma Science & Technology



PLASMA PROCESSES FOR BIOMEDICAL APPLICATIONS

ONE-DAY WORKSHOP

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Programme

Start (pm)	Event
13:00	Registration S206-209 (w/ tea & coffee)
13:30	Welcome: Miles Turner, Director NCPST Introduction: Jean-Paul Mosnier. Workshop Chair
13:40	Invited Talk: Plasma Sprayed Biocoatings: Recent Advances and a Vision Into Its Future , Joseph Stokes, DCU
14:10	Invited Talk: Application of Both Low Pressure and Atmospheric Plasmas for the Surface Modification of Biomaterials , Denis Dowling, UCD
14:40	Contributed Talk: Influence of Spray Parameters on the Phase Composition of Plasma Sprayed Hydroxyapatite Coatings , Tanya Levingstone, DCU
14:55	Contributed Talk: Advanced Analytical Platforms , Feidhlim O'Neill, CBAS
15:10	Tea Coffee
15:40	Invited Talk: Decontamination of Industrial Effluents using Pulsed-Plasma Gas Discharge , Neil Rowan, Athlone IT
16:10	Invited Talk: Laser Induced Plasmas for Enhanced Biomedical Applications , Dermot Brabazon, DCU
16:40	Contributed Talk: Atmospheric Plasma Disruption of Bacteria Vic Law, DCU
16:55	Contributed Talk: ZnO Thin Films grown by Pulsed Laser Deposition for the Photocatalytic Inactivation of Pathogenic Thin Films , Ricky O'Haire, DCU
17:10	Break
17:30	Poster Session w/ wine and food reception
19:30	Close of business

INVITED AND CONTRIBUTED TALKS

PLASMA SPRAYED BIOCOATINGS: RECENT ADVANCES AND A VISION INTO ITS FUTURE

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Advances

The selection and application of synthetic materials for surgical implants has been dependent upon the biocompatibility of specific prosthetic devices, such as femoral hip implants. Past rationale for ceramic biomaterials was based upon the chemical and biochemical inertness (minimal bioreactivity) of elemental compounds constituted into structural forms (materials). Subsequently, mildly reactive (bioactive), and partially and fully degradable ceramics have also been identified for clinical uses. Structural forms have included bulk solids or particulates with and without porosities for tissue ingrowth, and more recently, coatings (such as Hydroxyapatite thermally sprayed using atmospheric Plasma spray techniques) onto other types of biomaterial substrates (such as stainless steel or titanium alloys). While their application determined the physical shape of the bio-implants, the advantage/disadvantages of using these techniques are dependent on: (1) the basic material and design properties of the constructed device and (2) the patient-based functional considerations. Over the past fifteen years the science and technology for thick and thin bio-coatings have evolved significantly [1-3]. Applications of ceramic biomaterials range from bulk (100%) ceramic structures such as joint and bone replacements to fully or partially biodegradable substrates/scaffolds for the controlled delivery of pharmaceutical drugs, growth factors, and morphogenetically inductive substances. Other coatings such as DLC applied using Vapour Deposition Techniques have been deposited onto acetabular cups used in total hip arthroplasties (THA), to replace sinter-formed alumina or zirconia femoral head components [4]. These have also been shown to either promote or inhibit cell growth and show promising results for the future research of the human central nervous system [4]. Because of the relatively unique properties of bioceramics, expanded uses as structural composites with other biomaterials and macromolecular biologically-derived substances are anticipated in the future.

Future

Biodegradable surface coatings have been proposed to enhance bone healing, modelling, and possibly remodelling, with the bone-to-substrate interface

sequentially changing over the first months of implantation. Another aspect of these bioceramics is their basic nature that influences the osteoconduction of bone along the zones of tissue contact. These coatings are also used within load-bearing orthopaedic and dental implants applications and include inert, active, and degradable forms. In contrast to earlier times, the applications of bioceramics are increasing with emphasis on mechanical mixtures (modular or structurally integrated) or composites such as bonded coatings or chemically integrated (multi-biomaterial) device components [5]. As we move towards the regeneration of natural tissues through growth factor, morphogenetic substances or stem cell systems, the bioceramics could be an optimal carrier for many bone specific applications [6,7]. Over the next decade biocoatings will be required to provide drug delivery systems (DDS) within their structures, which will involve composite coating structures applicable to a matrix-type DDS [8]. These DDS will have to combine bio-polymeric therapeutic drugs with existing bio-ceramics such as Hydroxyapatite to aid surgical recovery and to extend the life of these biological implants. Opportunities for polymeric matrix composites with bio-ceramic structural phases and bioactive surfaces for bonding to bone is the next step. Optimisation of this technique will require multidisciplinary collaborations among the various professionals associated with the surgical implant field. This should be an interesting and exciting time for research, development, and applications of new and improved prosthetic devices.

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APPLICATION OF BOTH LOW PRESSURE AND ATMOSPHERIC PLASMAS FOR THE SURFACE MODIFICATION OF BIOMATERIALS

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This presentation will give an overview of research work at UCD on the use of both low pressure and atmospheric plasmas for the surface modification of biomaterials. This research can be broken down into three broad areas as follows

Deposition of nm thick coatings by physical vapour deposition (PVD) and plasma enhanced chemical vapour deposition (PECVD) for tribological and anti-bacterial applications. The tribological coatings include the use of diamond-like carbon (DLC) coatings on orthopaedic and catheter implants to reduce friction and to enhance wear resistant. The anti-bacterial properties of silver and silver / platinum coatings have been evaluated for use on polymeric implants.

Use of atmospheric plasmas to enhance cell adhesion by increasing the surface energy of polymers. The surface of polymers such as polystyrene and PVC have been systematically

modified using siloxane type coatings. Cell adhesion on the modified surfaces has been correlated with water contact angles ranging from hydrophilic (12°) to superhydrophobic (140°). Atmospheric plasmas can also be used for the controlled application of other functional groups e.g. $-\text{COOH}$, NH_2 etc. This will be discussed as will the influence of polymer surface roughness on cell adhesion.

Preliminary studies have been carried out to determine how cells adhesion is influenced by exposure to helium atmospheric plasmas.

In addition to the above research activity overview, this presentation will outline some of the challenges to the greater application of PVD and PECVD plasma processing for biomedical applications.

INFLUENCE OF SPRAY PARAMETERS ON THE PHASE COMPOSITION OF PLASMA SPRAYED HYDROXYAPATITE COATINGS

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Hydroxyapatite, (HA), is a calcium phosphate bioceramic material which has an almost identical chemical composition to that of bone. It has excellent biocompatibility and is osteoconductive, allowing bone cells to grow on its surface [1, 2]. For this reason it has been used successfully in the medical industry for many years. One such application is as a coating on implants, where bone cells grow onto the coating and provide fixation for the implant.

Plasma spraying (Figure 1) is the most commonly used method for the application of HA coatings. This is a thermal spray process in which powder particles are melted in a high temperature plasma flame and propelled towards the substrate material forming a coating. The advantages of this process include the high deposition rate which allows coatings to be quickly produced.

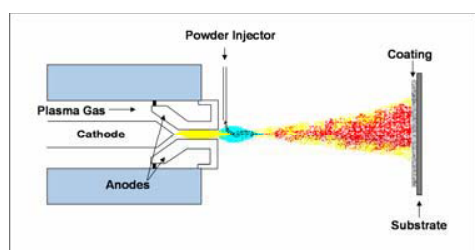


Figure 1: Schematic of the Plasma Spray Process

However, the high temperatures during spraying cause the decomposition of the HA powder particles. This leads to the formation of new phases such as α -tri-calcium phosphate (α -TCP) and β -tri-calcium phosphate (β -TCP). The rapid quenching of the particles on the substrate results in a coating with a high content of amorphous calcium phosphate (ACP) phases. These phases are known to dissolve more quickly in the body than HA. Dissolution of these phases in vivo results in a weakened coating which can no

longer secure the implant, thus causing implant failure.

The properties of plasma sprayed HA coatings are known to depend on the spray parameters used in their production. In order to produce more stable coatings the influence of these parameters needs to be more clearly understood. In this research, the influence of five parameters; Current, Gas Flow Rate, Powder Feed Rate, Spray Distance and Carrier Gas Flow Rate, on the crystallinity and purity of the coatings was investigated.

The process was investigated using the Design of Experiment technique. Mathematical models, with significance levels of 95%, were developed to describe the factors affecting both the crystallinity and purity of the HA coatings. The crystallinity of the coating was found to depend on current, gas flow rate, spray distance and carrier gas flow rate. All of the investigated parameters were found to affect the purity of the coating. Interaction effects between parameters were also observed. The models were then used to optimise the process and produce coatings with the highest possible crystallinity and purity.

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ADVANCED ANALYTICAL PLATFORMS

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The role of biosensors is becoming increasingly important across a wide range of sectors including the biopharmaceutical, food processing and defense industries. The potential of these sensors is dependent on demonstrating qualitatively verifiable sensor platforms that display high performance and reproducibility. The aim of this study is the development of high performance analytical platforms, which will be compatible with polymer micro-fabrication and mass production techniques, thus allowing the fabrication of low cost generic biosensor platforms.

A wide range of different transduction techniques have been examined for use in biosensor platforms. A significant area of research involves the use of optical based technologies. These methods are based on techniques such as optical interference, absorption and/or phase measurements and fluorescence based techniques.

The transduction method chosen for the advanced analytical platforms developed in this study was the fluorescence method. These platforms are designed to provide generic, novel fluorescence based optical sensor platforms for use in bio-pharmaceutical process stream analysis.

In this case the functionality of the sensor is provided using bio-recognition molecules. These molecules are immobilised on the surface of the biosensor and are used in a modified fluorescence based enzyme linked immunoassay (ELISA) approach. In this method the analyte is passed over the immobilised bio-recognition molecules. A secondary antibody, labeled with a fluorescence dye, is then reacted with the immobilised species to form a sandwich type assay. The optical excitation of the dye results in fluorescence output which can be measured and related back to the concentration of the analyte.

The accurate control of this immobilisation process and the resulting active area is of critical importance in developing this type of biosensor. Variations in the surface coverage, concentration or orientation of the recognition element will result in significant variations in output signal. A range of immobilisation strategies have been examined to enable the control of this active region.

These strategies require the surface modification of the initial substrate material using both chemical and plasma treatments to generate activated polymer surfaces allowing further chemical modification. This initial surface treatment is followed by a stepwise surface functionalisation chemistry approach. These techniques allow immobilisation of bio-recognition molecules to a wide range of commercially relevant substrates such as *polystyrene, gold, glass, poly-L-lysine, zeonex* and *zeonor*.

The analytical platforms developed in this study were fabricated using zeonex (Zeon Chemicals Europe Ltd.). This material was chosen as it has low water absorbency, high transparency and low auto fluorescence. The immobilisation procedure was carried out to produce the bio-functionalised surface which was examined using both AFM metrology and an in-house CCD imaging system.

The biochip platforms have been successfully utilised for a mouse IgG/anti-mouse IgG demonstration assay. Specific assays to examine bio-process product streams are currently under development within the Centre for Bioanalytical Sciences in collaboration with our main industrial partner, Bristol-Myers Squibb.

DECONTAMINATION OF INDUSTRIAL EFFLUENT USING PULSED PLASMA GAS-DISCHARGE

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This study reports on the development of a pulsed-plasma gas-discharge (PPGD) system for the novel decontamination of chilled poultry wash water (Fig. 1). Results showed that treatment of poultry wash water in the plasma generation chamber for up to 24s at 4°C reduced (≤ 8 log CFU/ml) *Escherichia coli* NCTC 9001, *Campylobacter jejuni* ATCC 33560, *Campylobacter coli* ATCC 33559, *Listeria monocytogenes* NCTC 9863, *Salmonella enterica* serovar Enteritidis ATCC 4931 and *S. enterica* serovar Typhimurium ATCC 14028 populations to nondetectable levels (Fig. 2). While similar PPGD-treatments at 4°C also produced significant reductions (>3 log CFU/ml) in recalcitrant *B. cereus* NCTC 11145 endospore numbers within 30s, the level of endospore reduction was shown to be dependent on the nature of the sparged gas used in plasma treatments.

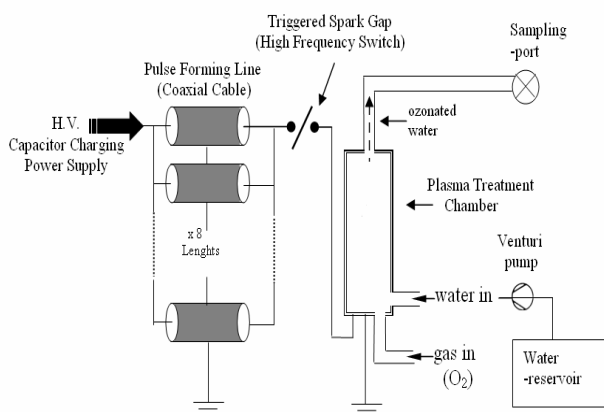


Fig. 1. Schematic of PPGD treatment system
Use of scanning electron microscopy revealed that significant damage occurred at the cellular level in PPGD-treated test organisms. Studies

also showed that while there was good agreement between use of conventional direct plate counts (PC) and novel respiratory staining for enumerating PPGD-treated *Campylobacter*, the former PC technique overestimated the level of sub-lethally injured *Salmonella* by 1.3 log CFU/ml.

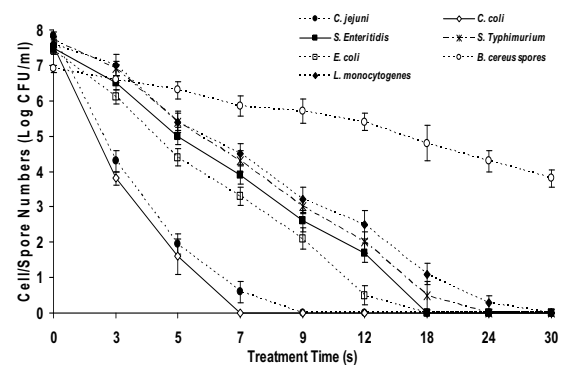


Fig. 2. PPGD-inactivation of microbial pathogens in chilled poultry wash water.

This “green-friendly” electrotechnology delivers energy in intense ultrashort bursts generating liquids of multiple biocidal properties (comprising ozone, ultraviolet light, acoustic and shock waves, and pulsed electric fields), which reverts back to water leaving no unwanted chemical residues. This technology offers an exciting complementary or alternative approach for treating raw poultry wash and for preventing cross-contamination of processing environments.

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LASER INDUCED PLASMAS FOR ENHANCED BIOMEDICAL APPLICATIONS

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Introduction

Lasers have been applied to solve many conventional manufacturing problems. Large laser machining centres are now commonly used industrially for welding and cutting of sheet metal. Other developments for manufacturing include laser shot peening to increase fatigue strength of aerospace components, laser diodes to soften metal just ahead of the tool tip prior to machining, laser cladding with injected powders for high wear resistant surfaces, and heat treatments of the metal surface, such as gas nitriding and amorphous thin film production.

Biomedical applications

Due to the unique high energy density and precision of laser systems their application to new areas has seen the development of rapid manufacturing techniques such as Selective Laser Sintering and Stereolithography. These devices are commonly used to produce parts for biomedical application, including prosthesis, surgical devices, and pre-operation visualisation models. Lasers are also commonly used as diagnostics tools. One application includes their use in electrophoresis cell health detection systems.

Lasers plasmas for biomedical applications

Lasers are used for soft tissue ablation as well as for dermatological applications, such as blemish and hair removal. An interesting new application of femtosecond lasers is for nerve regeneration and nerve tissue cutting. Nanosurgery and nanosurgery techniques to allow these applications have been developed in recent years. By using femtosecond lasers, there is not enough time for heat to diffuse and so surrounding tissue is protected. The laser parameters that lead to breakdown of water are very similar to those required for laser surgery. Tests in water can therefore indicate appropriate process parameters for size and shape of the laser-induced plasma.

Laser machining

Knowledge of the temperature and spatial profiles produced by laser ablation of solid samples is important for many applications including microfluidics chemical and biological total analysis systems and wave guides. It has been widely reported that the relation of channel micrometer-size features to processing parameters are not well known and that the walls of the ablated surfaces are not smooth. In general, ablating materials is a non-linear process that can be difficult to model and control. To assess the impact of the important physical parameters underlying the process a heat transfer model taking account of energy coupling and conduction and correlation with actual ablation within a design of experiments was undertaken recently [1]. A Nd:YVO₄ and a CO₂ laser system were used. These systems were capable of producing power densities of 273.4 MW/cm² and 23.5 MW/cm² respectively. Much higher power densities (10²⁰ W/cm²) can be achieved with ultra-short pulse laser systems. Femtosecond laser ablation can therefore offer much greater potential for highly accurate dimensional stability during the laser ablation process.

Other plasma related laser processing

X-ray beams generated using high energy plasma created by laser radiation have been used for cell diagnostics. High energy laser produced plasmas also provide a useful route to producing single walled carbon nanotubes. Pulsed Laser Deposition (PLD) presents a novel method to obtain bioactive and biocompatible thin layers PLD allows has been used for the production of functionally graded thin films, ZnO epitaxial films (with associated electro-optic properties), and Mg:Nb films for hydrogen storage devices.

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ATMOSPHERIC PLASMA DISRUPTION OF BACTERIA

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Over the past decade, the electrophysical disruption of bacterial cells has been considered as an alternative method of bacterial sterilisation. Bacterial infections including; MRSA (methicillin-resistant *Staphylococcus aureus* [1], *Pseudomonas aeruginosa* [2] VRE (vancomycin-resistant *enterococcus*) and *Clostridium difficile* [3] pose significant challenges in both nosocomial and community settings as these organisms are capable of proliferation on dry inanimate surfaces. More significantly, they remain viable following conventional sterilisation methods including autoclaving and chemical methods, due to innate resistance, which has led to the investigation of alternative methods of sterilisation. Studies have shown that the highly energised molecules act to perforate the bacterial cells, causing a shearing and breakdown of the cell wall, thus killing the cells [4]. This is an extenuated electroporation effect, causing significant irreversible damage to the cells. It is considered that this would also be the case for the removal and disruption of bacterial spores, thus resulting in a sterile surface following treatment. Atmospheric plasma devices have been examined over the past decade, to determine the most effective gas and pressure that have most significant disruption effect on bacterial cells. A novel atmospheric plasma device has been developed in the NCSPT in conjunction with Hiden Analytical Ltd. UK. This plasma device is a helium atmospheric pressure plasma dielectric barrier, which utilises a Flyback drive circuit that generates low temperature mode of operation. Chemical analysis of the plume has identified hydrated ion cluster molecules. To investigate the validity of molecular bombardment of bacterial cells and the extent of cellular disruption suspensions of sessile *P.aeruginosa* on glass slides were plasma treated. Atomic Force Microscopy scans (Fig 1 and 2), show a significant alteration of the surface of the cells.

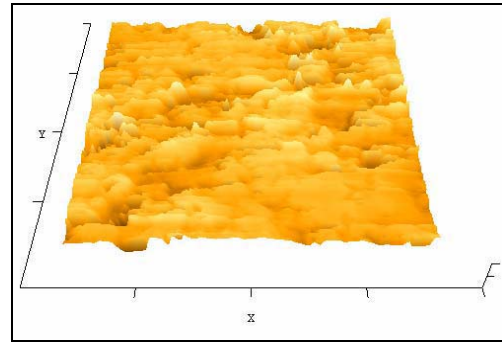


Fig 1: Surface topography of the *P.aeruginosa* cells before treatment. The undulating surface represents the multiple sessile organisms on the slide.

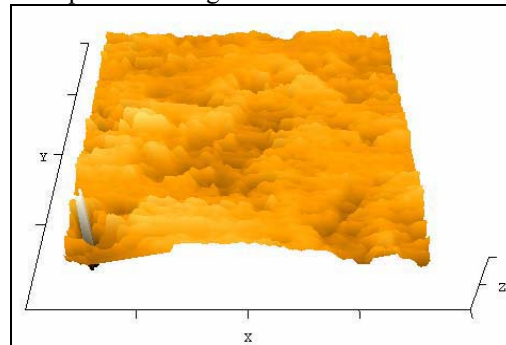


Fig 2: the AFM scan following 5min He plasma treatment. The surface appears less undulating indicating disruption of the cells on the surface.

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ZnO FILMS GROWN BY PULSED LASER DEPOSITION FOR THE PHOTOCATALYTIC INACTIVATION OF PATHOGENIC BIOFILMS

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The mechanisms by which biofilms are resistant to common antimicrobial agents are poorly understood. Therefore, new antimicrobial therapies are needed. Photocatalysis has the potential to be used in removal of biofilm formation on the surfaces of medical devices. Oxide photocatalysts such as titanium dioxide, have shown promise as effective antimicrobials [1].

In the present work [2], we carry out a first study of the antimicrobial properties of zinc oxide (ZnO) whose photocatalytic properties are known and illustrated in Fig.1.

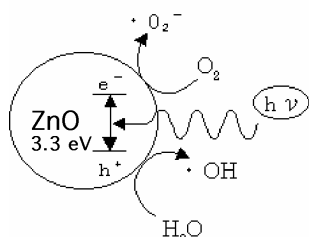


Figure 1. A band gap photon creates an electron-hole pair that is used for the decomposition of water and the reduction of oxygen species adsorbed on the surface.

The pulsed laser deposition (PLD) technique was used to deposit 3.5 μm thick ZnO films on half of the surface of borosilicate glass (Pyrex) slides at room temperature in a low-pressure (eg. 10^{-1} mbar) of oxygen gas. X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM) showed that these films were strongly c-axis oriented and had a smooth surface, respectively. X-ray photoemission spectra (Fig.2) indicated that the as-grown films were almost stoichiometric ($\text{ZnO}_{0.96}$) and contained about 25% oxygen in a less oxidised state (peak around 533.0 eV) than the bulk oxygen (O^{2+} peak at 530.4 eV). This component did not appear to be located on the surface.

The clinically relevant bacterial strain *Staphylococcus epidermidis* was then grown over the entire slide and inactivated using UV photocatalysis. The qualitative killing of the bacterial biofilm grown on the ZnO coated region of the glass substrate was demonstrated (Fig. 3).

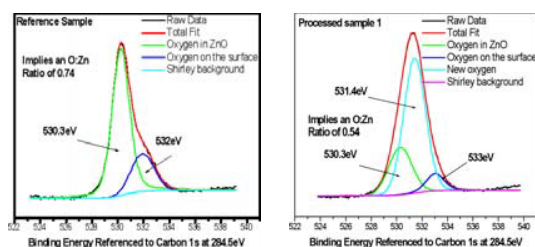


Figure 2. XPS O_{1s} : as-grown (lhs) and after UV irradiation (rhs).

The biomaterial was suitably removed from the surface of the irradiated films to allow their characterisation after the photocatalytic process took place. XPS (Fig.2 rhs) studies showed that: (1) the oxygen in the active films was reduced upon the UV/bacterial interactions and (2) the stoichiometry had changed to $\text{ZnO}_{2.3}$ compatible with the chemical formulae of zinc peroxide (ZnO_2) or zinc hydroxide ($\text{Zn}(\text{OH})_2$). SEM data showed considerable pitting of the surface (photodecomposition) compatible with a photocatalytic mechanism taking place on the active sites of the as-grown surface such as oxygen vacancy sites.

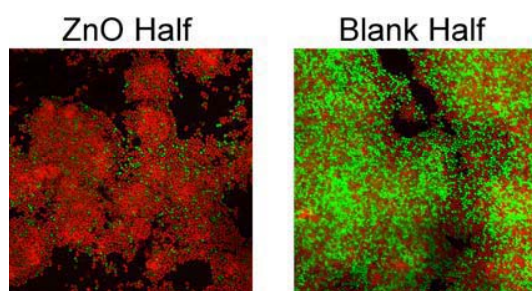


Figure 3. Microscopic evidence showing the qualitative killing of the biofilm after UV irradiation (lhs).

This study shows that photocatalytic sterilization has great potential as a new antimicrobial technology that could be feasibly utilized in the removal of potentially harmful biofilm formations.

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POSTER PRESENTATIONS

DEPOSITION OF DUPLEX SI-DOPED DLC COATING FOR ORTHOPAEDIC IMPLANT SURFACES USING PLASMA ENHANCED CHEMICAL VAPOUR DEPOSITION (PECVD)

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Introduction

Diamondlike carbon (DLC) is an amorphous, meta stable material and contains a mixture of sp^3 and sp^2 structures in a disordered network. It is well-known as good candidate for tailoring the surface performance of orthopaedic implants due to their superior mechanical, tribological and biological properties. Modification of DLC coating will increase the adhesion and also reduce the internal stress within the coating. This is a highly important task as this will increase the performance of biological implants by enhancing their wear resistance at their surfaces in applications such as the femoral ball head used in hip joint replacement therapy. Modification of DLC coatings by doping with other elements and introducing an interlayer between the coating and the substrate is the aim of this study. Si doping was reported to improve the adhesion strength and also the internal stress of DLC coatings [1-2]. Ikeyama et al. [3] found that only one to two per cent of Si was required to improve the adhesion and the internal stress of coating while maintaining their surface hardness.

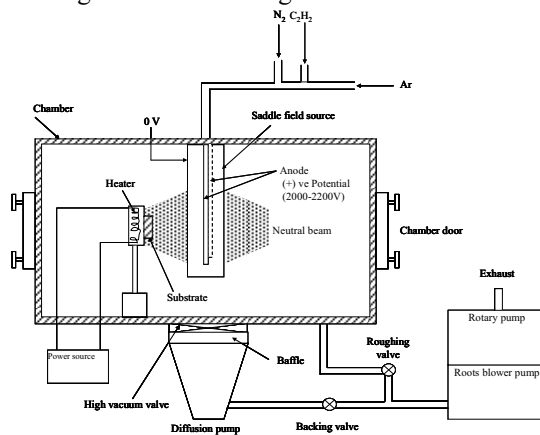


Figure 1: Schematic diagram of the PECVD; Saddle Field Fast Atom Beam Source for plasma nitriding process and Si-doped DLC coatings [6].

Research

Based on significant contribution of Si element onto DLC, a duplex Si-doped DLC coating will be deposited onto 316L stainless steel substrates using the Plasma Enhanced Chemical Vapour Deposition process (Figure 1), using the saddle field fast atomic beam source system. In this research, a mixture of methane and hydrogen (25% CH_4 and 75% H_2) will be used to deposit the DLC coating and Tetramethylsilane (TMS) gas to produce the Si precursor. This composition of gases has shown capability to provide a high level of hydrogen within the DLC surface and is expected to produce very low friction coefficients and excellent wear performance of implant coating surfaces [4-5].

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PULSED LASER DEPOSITION OF ZINC OXIDE MATERIAL

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Zinc oxide (ZnO) is a group II–VI compound semiconductor with a wide direct band gap of 3.37 eV at room temperature (RT). It has a strong exciton binding energy of 60 meV, much larger than that of GaN (25 meV) and the thermal energy at RT (26 meV), which can ensure an efficient exciton emission at RT under low excitation energy. As a consequence, ZnO is recognized as a promising material for application in optoelectronic devices in the blue–UV region. However, the lack of good and reliable p-type ZnO has been a major problem for many years. Recently, thanks to the considerable efforts of researchers, steady progress has been made in this field.

The ZnO thin films were grown on c-sapphire, (0001) orientation, substrates using the pulsed laser deposition (PLD) technique which is shown in Fig.1.

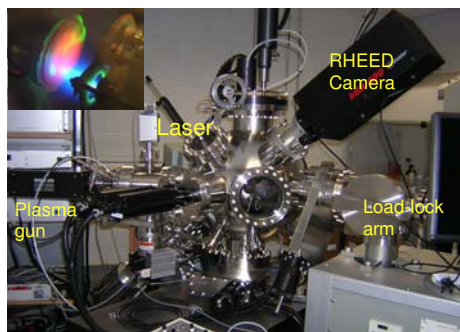


Fig.1 The PLD system consisting of an ultra high vacuum chamber, a plasma source for doping studies and RHEED apparatus for in-situ diagnostics.

The ZnO layers were ablated from a ZnO ceramic target, with a frequency-quadrupled Nd:YAG laser, (120 mJ of 266 nm laser light in a 6 ns pulse), with a fluence on target of 1.4 J cm⁻². Nitrogen was incorporated using an electron cyclotron resonance (ECR) plasma source. Ions such as N⁺ and N²⁺ were extracted with energies of the order of a few tens of eVs. No oxygen was introduced in the chamber during growth in order to create a Zn-rich environment which appears to favour the nitrogen incorporation in ZnO films [1].

To date, we have grown epitaxial, nitrogen-doped, ZnO thin films. All the samples were characterised by X-ray diffraction (XRD), photoluminescence and temperature dependent Hall measurements, in the Van der Pauw configuration. The XRD patterns show only high intensity ZnO (0001) peaks, indicating that the film is c-axis oriented. They exhibited p-type conduction within the range of 200–450 K. The hole concentration was 9.5 x 10¹⁵ cm⁻³ at room temperature in the dark.

In addition we have grown composite ZnO/CeO₂ thin films epitaxially on *r*-sapphire substrates[2]. Their crystalline properties showed the ZnO (wurtzite structure) and CeO₂ (fluorite structure) layers to be highly textured with the (20–23) and (100) orientations, respectively.

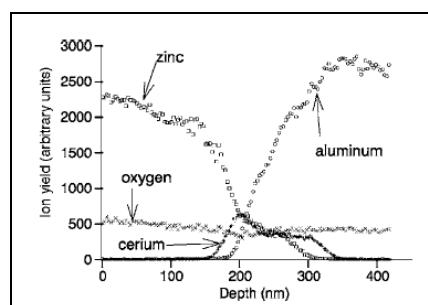


Fig.2 Relative secondary ion emission yields for oxygen, aluminum, zinc, and cerium as a function of the depth (nm) from the surface of a ZnO/CeO₂ composite layer grown on a *r*-sapphire substrate.

Depth profiling of the structure, Fig.2, showed the CeO₂ layer to be quite inhomogeneous, with evidence of interdiffusion of zinc and aluminum (from the sapphire substrate) through this layer. However, the aluminium front was found to be clearly located behind the cerium front at the ZnO/CeO₂ interface, thereby showing that the CeO₂ layer acts as an efficient barrier against the diffusion of aluminum into the ZnO layer.

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PLASMA SPRAYING OF FREE STANDING HARD TISSUE SCAFFOLDS

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Introduction:

Hydroxyapatite (HA) is osteoconductive and bioactive, but also brittle and relatively weak. This bioceramic alone can only be used in non load-bearing areas of the human body [1]. This is the reason why current research is focused on using HA as a coating applied onto metallic implants where the implant provides the strength to the component. To broaden the use of HA as a free-standing material, an additional material must be used to form a biocomposite with strength/structure similar to that of bone. Some of the solutions proposed by researchers have been to introduce additives such as bioinert ceramics (stabilized-ZrO₂, Al₂O₃, TiO₂) or recently Carbon Nano Tubes (CNT) to reinforce HA [2]. Adding synthetic or extra-pure natural minerals such as Wollastonite [3] or some polymers have been also reported.

Plasma spraying (PS) employs an ionised gas to melt the powders propelling them towards the surface of a substrate. This technique is capable of, but limited in producing thick coatings, so after a deep understanding of the influential parameters, it would be possible also to form free standing 'coatings' (scaffolds).

Research:

Initially a deep study on the possible materials to use will be carried out (polymers & bioceramics). Depending on the material selection, modification to or the new design of the powder feeder or the cooling system of the PS equipment will be required. An experimental and analytical study on the influential parameters of the PS technique on the stability, thickness and porosity of the free-standing component will be conducted to arrive at the proposed scaffold.

Project Aims:

The innovative challenge of this project is to obtain strong stable free-standing components using a technique that has never been used for this application. Resultant scaffolds will have controlled variation in porosity while attaining

required biological and mechanical characteristics.

Acknowledgments:

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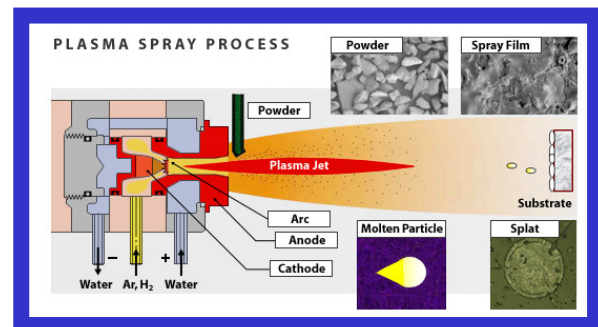


Figure 1: Plasma Spray Process [4]

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ZnO NANOSTRUCTURES ON SAPHHIRE

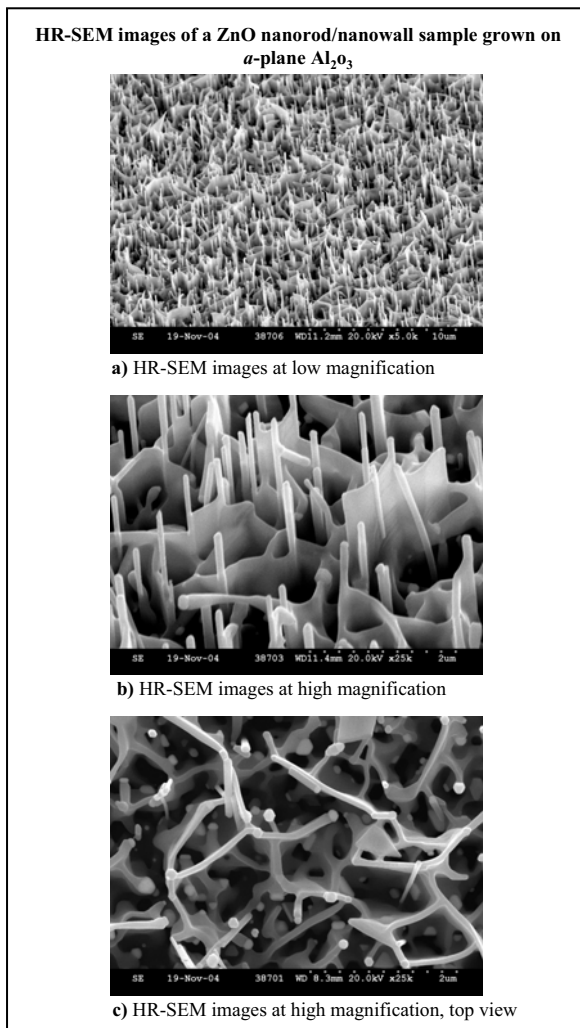
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Growth of ZnO nanowall arrays with subsequent growth of vertical nanowires using a two-step vapour phase transport method on *a*-plane sapphire is reported. X-ray diffraction and scanning electron microscopy data show that the nanostructures are aligned with *c*-axis normal to the substrate.

These structures provide an unusual 3-D nano-architecture and their interactions with bio-systems are largely unexplored.



COMPARISON OF HVOF AND PLASMA SPRAY TECHNIQUES USED TO PRODUCE BIO CERAMICS HA COATINGS

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Introduction

The Plasma Thermal Spray Process (Figure 1a) is used to spray molten or semi-molten powdered materials onto a receiving surface in the form of a coating. This sprayed material is injected into a high temperature plasma flame, where it is rapidly heated and accelerated to a high velocity [1]. These droplets rapidly cool down to form splats. Hydroxyapatite [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$], is a bioactive material. Their Ca/P ratio of 1.5-2.0 makes them an excellent choice for most dental and orthopaedic application in the form of bioceramic coatings [2]. In the case of Hydroxyapatite (HA) bio ceramic coatings, plasma spray coatings are used as a substitute for biological tissues applied onto femoral implants to aid inter-connectivity between the implant and the biological tissue bone.

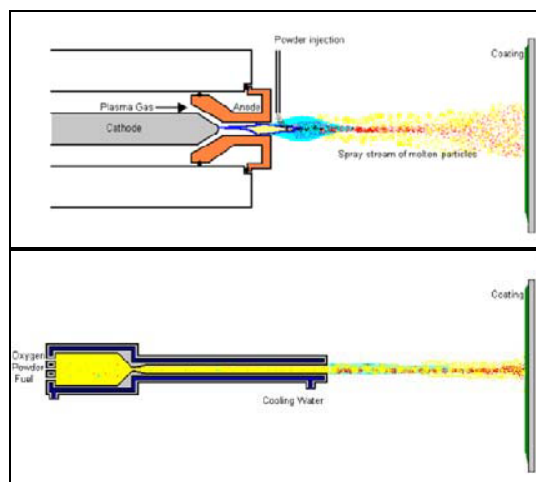


Figure 1: Schematic diagram of the Plasma and HVOF Thermal Spray Process [1]

The HVOF (High Velocity Oxygen Fuel) thermal spray process (Figure 1b) is reported to be a versatile technology and has been adopted by many industries due to its flexibility, cost effectiveness and the superior quality of coating produced [3]. To date, the only reliable method of applying HA coatings to metallic implants is

via using atmospheric plasma thermal spraying. However HVOF has the potential to also produce HA coatings which based on other sprayed materials, this technique should offer mechanical and biological results far superior to that of plasma spray.

Research

The quality of the resulting biocoating will be dependent on particle size, particle size distribution and particle morphology which also determine the flow characteristics in the powder feeding systems and the melting behaviour within the HVOF/plasma flame [4]. The smaller the granule size, the higher the specific surface area and the higher the bonding capacity. HVOF spraying process results in lower residual stresses than the conventional atmospheric plasma spraying. The coatings deposited by HVOF are significantly harder and tougher, and their abrasion resistance is two–three-fold higher [4]. Although the plasma spray process has been compared to HVOF for wear resistant ceramic materials [5], little research has focused on how the biocoatings behave as a result of each of these techniques. This research aims to identify this problem, by depositing bioceramic coatings using the HVOF process and comparing this to current HA Plasma sprayed mechanical and biological properties.

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INFLUENCE OF He_2 ($a^3\Sigma_u^+$) DESTRUCTION FREQUENCIES AND COMPLEX BEHAVIOR IN ATMOSPHERIC PRESSURE DISCHARGE MODES

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We investigate the characteristics of a dielectric barrier discharge in presence of excimers He_2 ($a^3\Sigma_u^+$) and analyze the influence of excimer destruction frequencies in different discharge regimes at atmospheric pressure. The spatial structure of APGD¹ is shifted to APTD¹ as the destruction frequency of excimers is decreased. The temporal evolution of metastable and excimer densities show their dominance in different phases of a complete cycle. The atmospheric pressure discharge modes are effected with small N_2 impurities and the discharge mode structures are described under different operating conditions in presence of species such as electrons, He^+ , He_2^+ , He^* , $\text{He}_2(a^3\Sigma_u^+)$ and N_2^+ . The distribution of ionization rates and electron mean energies are shown that associates the higher and lower rates to APG and APT discharges. The spatiotemporal profiles are displayed in presence of the parameters applied voltage, discharge gap width, operating frequencies and the electric field. The complex dynamic behavior is identified at low frequencies from 1 to 7 KHz¹ using one dimensional fluid model in pure helium gas. The discharge current density structure shows the inhomogeneous behavior that represents the discharge plasma operates between lower and higher ionization modes in several cycles. The temporal inhomogeneity of discharge plasma is emerged due to stepwise ionization processes of metastables and excimers and the discharge plasma is in homogeneous mode in the

absence of these processes. The suppression of instabilities is also viewed with small N_2 impurities and then the discharge is operated in the uniform Townsend mode. A bifurcation phenomenon is observed in an atmospheric pressure discharge and this provides more insight in the internal structure of DBD¹. The surface densities of charged particles at both dielectric barriers are illustrated in presence of the surface processes, such as charged particles absorption, surface recombination and electron desorption due to the metastables, Excimers and thermodesorption of electrons. An example is presented for the discharge current density structure in He- N_2 discharge and compared with the experimental results.

[1] APGD: Atmospheric Pressure Glow Discharge, APTD: Atmospheric Pressure Townsend Discharge, KHz: Kilo Hertz, DBD: Dielectric Barrier Discharge

MICROFABRICATION IN POLYCARBONATE WITH A Nd:YVO₄ LASER SYSTEM

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Introduction

Internal microfabrication was achieved by focusing 80 ns laser pulses of a 2.5 W, 1 MHz and 1.064 μm Spectra Physics Nd:YVO₄ laser system underneath the surface of polycarbonate (PC) samples causing the formation of microstructures in the irradiated regions. The process was studied to investigate the laser beam parameters on the resulting structures size and morphology. The process control parameters were taken as the laser power (P: 0.2-0.5 W), pulse repetition frequency (PRF: 1000-5000 Hz), and the number of pulses (N: 1000-15000).

Experimental setup

The laser beam was downsized to about 15 μm inside the samples using a 25.6 mm achromat lens. The laser emission parameters and the samples' 3D position were PC-controlled. Therefore, 3D CAD structures could be microfabricated.

Results

The breakdown threshold intensity for PC was calculated as $22.9 \times 10^3 \text{ W/cm}^2$ [1]. The laser power P and number of pulses N were observed to be directly proportional to the voxel size as seen in figure 1.

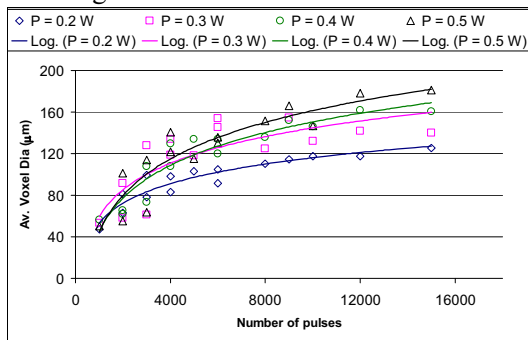


Figure 1: Effect of P and N on voxel size.

The PRF controlled the rate at which energy was deposited into the material. Voxel morphology depended strongly on PRF. At low PRF, the

voxels had star-like shapes and at high PRF they were more circularly uniform. Figure 2 shows two voxels fabricated using P = 0.2 W. Figure 2(a) shows a voxel fabricated using PRF = 1000 Hz and N = 1000. Figure 2(b) shows a voxel fabricated using PRF = 3000 Hz and N = 3000. Both settings produced the same laser fluence of 30.5 kJ/cm^2 . It can be seen that PRF has a hammering effect at low rates, while it uniformly deposits the energy at high rates.

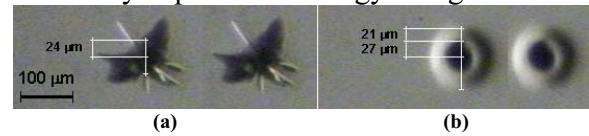


Figure 2: Voxel morphology dependence on PRF

The laser beam was also focused using an oil immersion objective lens (NA 1.25). The spot size was about 0.82 μm and the calculated depth of focus was 1.8 mm, however it was experimentally found to be about 200 μm due to the use of oil that enhanced coupling and collection of the laser beam. The calculated breakdown threshold intensity of soda lime and fused silica was 28.4 MW/cm^2 and for sapphire it was 273.4 MW/cm^2 . Figure 3 shows voxels fabricated inside a soda lime glass sample.



Figure 3: Voxels in soda lime glass.

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DIRECT WRITING OF MICROCHANNELS IN GLASS WITH A CO₂ LASER SYSTEM

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Introduction

Microchannels fabricated, on the surface or in the bulk of glass sheets, are widely used in various applications such as telecommunication, optical and bio-medical engineering. Glasses absorb laser radiation in a way that depends highly on the incident wavelength [1], most transparent materials are opaque in the visible region but absorb strongly at or near 10 μm , which makes CO₂ lasers very efficient for machining these materials [1]. Within the intensity range used, the laser-induced plasma is a laser-supported combustion scheme (LSC), which can shield up to 90% of the incident laser intensity [2].

Experimental setup

The CO₂ laser beam was focused using to a spot size of approximately 90 μm on the surface of two millimetre thick commercial soda-lime glass sheets. The laser power (P: 18-30 W), pulse repetition frequency (PRF: 160-400 Hz), and scanning speed (U: 100-500 mm/min) were chosen as the process control parameters. The glass sheet was translated in order to produce single line channels using processing parameters based on a factorial design of experiments.

Results

Microchannels were produced and the sample was later gold-coated in order to facilitate 3D scanning of the channels profiles. The responses measured from the profiles were the width (μm), the depth (μm) and the surface roughness, Ra, (μm). Figure 1(b) shows the 3D profile of a sample channel that was produced using P = 24 W, PRF = 228 Hz and U = 300 mm/min. The range of process parameters used in this work produced microchannels width range of (81-365 μm), depth range of (3-379 μm) and Ra range of (2-13 μm). The measured responses were used to statistically model of the process in terms of its control parameters. It was found that P is directly

proportional, whereas PRF was inversely proportional to the width and depth. Since the pulse energy is determined from P/PRF, these results indicate that at high pulse energies, wider and deeper channels were produced. Moreover, low speeds increased the amount of heat deposition per unit length, which lead to wider and deeper channels. The collected Ra did not enable statistical modelling due to non-linear variations with respect to process parameters.

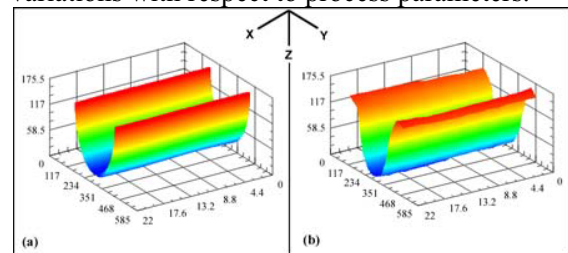


Figure 1: Sample channel 3D profile, (a) simulated, and (b) experimental. (μm scales).

The process was also simulated using a thermal mathematical model. The model showed that the laser-induced plasma shielded up to 75% of the incident laser radiation. The graphical results from simulations were used to understand the periodic nature of the microchannels' [3]. The model results also assisted in understanding Ra's relation to the control parameters.

Conclusion

A useful understanding of the process was gained from this study. Future work may include further experimental work with narrower ranges of the parameters in order to optimise the process and produce MEMS.

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EXPLORATION OF PRINCIPLES AND APPLICATIONS OF INTERNAL MICROFABRICATION IN DIELECTRICS

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Introduction

Internal microfabrication is achieved by tightly focusing a pulsed laser beam underneath the surface of a dielectric (transparent) material causing localised heating, melting and the subsequent evaporation/ablation of the material from the focal region [1]. This induces a permanent micro-scale voids (voxels) or regions with modified refractive index in the bulk material. This internal laser-induced breakdown can be defined as the generation of a practically totally ionised gas (plasma), which can be observed as a glow/flash in the focal region [2]. There are various proposed mechanisms for laser-induced breakdown in transparent materials [3]. For breakdown to occur in transparent materials, a nonlinear absorption mechanism must deposit energy by promoting electrons from the valence band to the conduction band [4]. The most commonly reported breakdown mechanisms are avalanche ionisation, multiphoton (MPI) ionisation or the mixture of both [4]. The induced structures' morphology and the responsible mechanism depend on the material properties, emission wavelength, laser focusing and the pulsed laser beam parameters.

Principles

The emission wavelength affects the material's absorption and the minimum focal spot size. Wavelengths used in this field are in the range (UV to near IR). The pulse width is a very important factor that affects the type of responsible breakdown mechanisms and therefore the morphology and size of the induced structural changes. Narrow pulse widths (pico-femtosecond) produce more confined and repeatable structures than the relatively wider pulse widths (nanosecond). For narrow pulse widths, the breakdown process becomes self-seeded and less dependant on impurity electrons in the focal region. Moreover, the relatively

higher diffusion timescale of the material allows a continuous and uniform heat deposition. The laser average power controls the laser intensity. The power and the pulse repetition frequency control the energy of each pulse and consequently the pulse fluence (J/cm^2). The number of laser pulses per voxel controls the total heat deposition amounts. Self focusing, the focusing technique, scanning speed are also parameters affecting the process.

Applications

Direct microfabrication is possible in virtually all transparent materials, which offers a cheap alternative to other optical modification processes that require specially designed materials and apparatus. The relative positioning and the pulsed focused laser beam can be controlled to facilitate various applications such as 2D and 3D optical data storage, waveguides, waveguide splitters and amplifiers, microchannels, MEMS and miniature optical sensors.

Research potentials

There is a debate on the responsible breakdown mechanisms, which is justifiable due to the wide range of laser systems and materials. Possibly there are different mechanisms that play roles in this phenomenon. Hence, comprehensive systematic experiments and studies must be conducted to understand and optimise the processes. Further research will help identifying the process and enhance controlling it.

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EXPERIMENTAL DESIGN OPTIMISATION OF THE SYNTHESIS OF BIOCERAMIC HYDROXYAPATITE AS FEEDSTOCK FOR PLASMA SPRAYING APPLICATION

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Hydroxyapatite, (HA), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, and other related calcium phosphate minerals have been utilised extensively as implant materials for many years due to their identical chemical composition and high biocompatibility with natural bone. HA, does not exhibit any cytotoxic effects and it can promote new bone growth, in what is more commonly termed an osteoconductive process.

The synthesis of an optimised HA calcium phosphate based powder for plasma spraying application is a fundamental yet crucial stage in the production of a successful Plasma Sprayed bioceramic coating. The performance, lifespan and quality of the resultant biological plasma coating *in vivo* is largely dependent on the initial HA powder particle characteristics, such as, phase composition, crystallinity, particle size and powder morphology [1]. However, these HA physico-chemical properties, synthesized by the chemical precipitation method are heavily dependent on the actual process variables implemented at this stage.

A Fractional-Factorial screening design with main and interaction effects, with the six influencing variables (Table 1), at two levels, in sixteen runs was therefore built using Design Expert 7.0, to screen a large number of the precipitation process variables in order to determine their effect

on the final HA characteristics. The several factors investigated were; T_0 : synthesis temperature ($^{\circ}\text{C}$), V_{st} : stirring speed (rpm) V_{ac} : acid addition rate (mole/min), t_r : ripening time (h), V_{ac} : reagent addition rate, Ca: initial calcium concentration and the presence of a controlled/uncontrolled environment. This experimental design highlighted the factors that have the most significant affect on HA precipitation process parameters, which was then used to build an optimisation plan based on Box-Benkhen design, in thirteen runs. Linear and quadratic polynomial equations for predicting the phase composition, crystallinity, crystallite size, lattice parameters and particle size were developed for both screening and optimisation design enabling the final determination of a set of idealised precipitation process parameters to obtain reproducible highly desirable HA feedstock specific for plasma spraying applications.

Table 1: Experimental factors and level of factors

No	Variable	Level -1	Level +1
1	Temperature	20 $^{\circ}\text{C}$	100 $^{\circ}\text{C}$
2	Stirring Speed	600rpm	1500rpm
3	Inert Atmosphere	without N_2	with N_2
4	Ripening time	0.5h	48h
5	Reagent Addition Rate	0.005 mole/min	0.01 mole/min
6	Initial Ca^{2+} Concentration	0.1 mole/L	2 mole/L

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PLASMA PLUME CHEMISTRY FOR BIOMATERIAL PROCESSING

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Dielectric Barrier Discharges (DBDs) at atmospheric pressure are a well-established technique for improvement of surfaces properties like wettability and adhesion [1]. The identification of the processes/mechanisms active in the discharge plume is now essential for optimisation of biomaterial processing.

This abstract describes preliminary Molecular Beam Mass Spectrometry (MBMS) measurements made on the Atmospheric Pressure Plasma Dielectric Barrier Discharge (APP-DBD) that is driven by a novel Flyback transformer inverter circuit [2]. The aim of these experiments is to establish the main thermal chemistry in terms of atom, molecule and hydrated clusters that are present within the expanding plasma plume and at the process surface [3].

For helium APP-DBD driven by a Flyback circuit two modes of operation have been identified using electrical and MBMS techniques with good correlations between each measurement. Two modes of discharge were observed:

- **Mode 1**, the DBD produced inside the quartz tube extended from the tube nozzle to the sampling plate of the MBMS as a diffuse glow. This discharge is typically produced from 3 to 15 mm distance between the end tube and the sampling plate of the MBMS.
- **Mode 2**, the glow developed a more intense core which was made up of a bundle of filaments terminating on the grounded sampling aperture plate. Typically produced from 0 to 3mm distance from the end tube nozzle to the sampling plate of the MBMS.

For mode 1, the MBMS reveal spectra that have cluster sequences, including positive ion hydrated clusters H_3O^+ (H_2O)_n, see figure 1. In contrast to Mode 1, the spectrum for Mode 2 is dominated by ions O^+ , NO^+ , N_3^+ as shown in

figure 2. Among the ions peaks the major intensity corresponds to the primary ions, N_2^+ and O_2^+ , formed by the reaction of the plasma with air. The presence of water clusters are only observed with $n = 1, 2, 3$ and 4.

The ability to control the relative intensity distribution of the hydrated cluster ions at the biomaterial surface is expected to have a major impact on engineering the final biomaterial functionality. Future work will investigate the surrounding environment of the plume and gas flows and their impact on processed biomaterial.

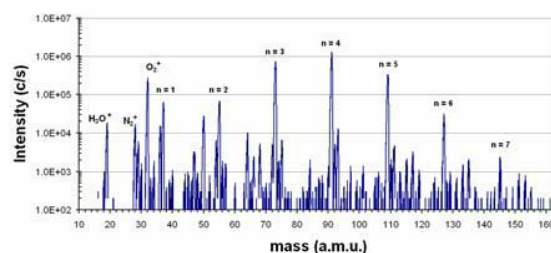


Fig.1 typical positive ion mass spectrum for Mode 1.

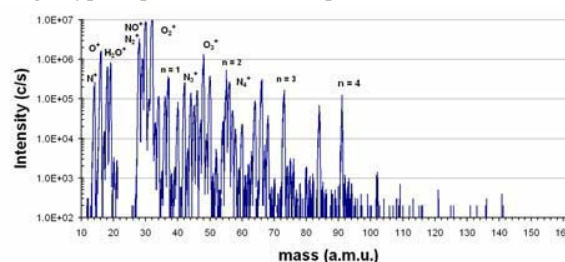


Fig.2 typical positive ion mass spectrum for Mode 2.

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NOVEL ATMOSPHERIC PLASMA APPLICATORS AND DRIVE CIRCUITS FOR BIOMEDICAL PROCESSING

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The Atmospheric Pressure Plasma Dielectric Barrier Discharges (APP-DBD) driven by low voltage transformers [1] and RF = 13.56 MHz are well-established for surfaces engineering and plasma physics studies. The renewed worldwide focus on atmospheric plasma for the treatment of biomaterial and biomedical device has brought new challenges to the existing technology. New low power drive circuits and applicator are now required for the optimisation of complex plume and volume chemistries to match solid and liquid biomaterial processing and the sterilization of biomedical devices. These markets now also include the nano-technology sector. The challenge at this new frontier generates the need for real-time non-invasive monitoring systems that can operate in small and complex 3-D geometries whilst having the measurement sensitivity to differentiate biomaterial modification. This abstract looks at Flyback drive circuits and real-time monitoring (passive and active) technique that may be applied.

The Flyback circuit is well established in C.R.T and plasma display globes sectors, yet only recently has it been used to drive a DBD plume for processing of biomaterial [1]. The feature of this drive circuit is its low voltage dc supply and simple transistor/transformer configuration that delivers a ~200 V, ~50 kHz bipolar pulse to the discharge volume. The high voltage part of the circuit can be configured to both balanced and unbalanced operation thus allowing the discharge applicator to be inserted in narrow tubes such as endoscopes and stents, or flat panel operation for plasma pumping of electrolytes and gases.

At the NCPST at DCU and Hiden Analytical Ltd. UK [2] we found that the helium APP-DBD driven by a Flyback circuit has two modes of discharge operation:

- **Mode 1**, the DBD produced inside the quartz tube extends from the tube nozzle to the work piece as a diffuse glow. This discharge

is typically produced from 3 to 15 mm in length.

- **Mode 2**, the glow develops a more intense core which was made up of a bundle of filaments terminating on a grounded surface. Typically produced from 0 to 3mm distance from the end nozzle.

Non-invasive electrical measures of the switching frequency have shown that these modes can be identified as the plasma plume reacts with different dielectric surfaces.

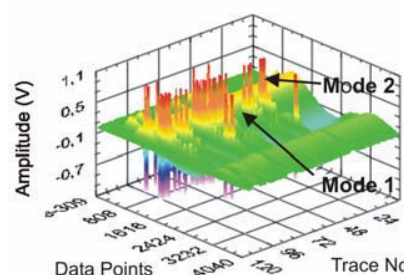


Fig.1 typical relative amplitude of voltage developed in the DBD as it is forced between Mode 1 and Mode 2.

Frequency domain analysis of a harmonic of the damped oscillations between the repetitive drive pulse chain has also provided information on the changing plume reactive component as the work surface distance is varied with a 0.1 mm resolution.

Work in progress and future work takes the advantage of the audio frequency in which the drive operates. The ability to listen to the plasma noise gives further insight to plume –surface interaction.

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THE IMPACT OF INNOVATIVE “NON-THERMAL” TECHNOLOGIES ON PRODUCT QUALITY IN MINIMALLY PROCESSED BEVERAGES

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In recent years the increase in consumer demand for higher quality, natural, preservative-free foods has increased the pressure on the food industry to find alternatives to replace traditional preservation methods for producing safe, ‘minimally processed’ food. Traditional preservation methods achieve pasteurisation by heating the product, leading to undesirable changes in the organoleptic properties (texture, colour, flavour and nutrients). Innovative “non-thermal” technologies such as pulsed electric fields (PEF), ultrasound (US), ultraviolet (UV) and high intensity pulsed light (HIPL), and their combination, have the potential to reduce or replace heat processing thereby minimising adverse changes, while ensuring product safety and improved quality. The objective of this project is to assess the potential for these novel

technologies to preserve foods while also investigating their impact on beverage quality. In particular, it will compare the effect of UV, HILP, US and PEF vs. conventional heating methods on beverage quality and shelf life (microbial and enzymatic aspects) while also assessing the impact on microbial safety using appropriate challenge studies. In addition it will generate data on economic viability of alternative preservation strategies and will build on UCD Dublin’s critical mass of expertise on innovative preservation. Finally using appropriate dissemination methods this project will help to educate industry on the potential for these innovative preservation technologies.

Key words: Innovative preservation, PEF, US, UV and HIPL

PVA/CHITOSAN BASED TISSUE ENGINEERING SCAFFOLDS FOR VASCULAR TISSUE ENGINEERING APPLICATIONS

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Introduction:

Cardiovascular diseases are one of the main causes of death. Although surgical interventions are successful, there is a substantial shortage of vascular grafts that necessitates development of artificial substitutes. Several artificial vessels have been used as large diameter vascular grafts: however these systems are not feasible for small diameter vessel replacement procedures due to their high thrombogenicity and low patency. In tissue engineering patients' own cells are combined with biocompatible cell carrier systems under *in vitro* conditions. This process can produce structures which are nearly identical to patients' tissue and induce minimal immune response.

Hydrogels are widely used tissue engineering cell carriers, due to their significant similarities to soft tissues. Polyvinyl alcohol (PVA) and chitosan are both biocompatible and biodegradable polymers that has been used in several biomedical applications. PVA hydrogels are especially suitable for vascular tissue engineering due to their mechanical characteristics. The aim of this project is the production, characterization and optimization of PVA/Chitosan hydrogels as vascular substitutes.

Materials and Methods:

Two different methods are used for production of hydrogels. In first method, PVA/Chitosan solutions are frozen at $-20\text{ }^{\circ}\text{C}$ for 12 h followed by a thawing step at room temperature for 12 h. The same cycle is repeated to obtain hydrogels with different physical properties. Hydrogels are crosslinked in a solution of KOH and Na_2SO_4 . Characterization of these hydrogels were made by tensile test, water content measurement and SEM. Second type will be manufactured by UV photocrosslinking of acrylate modified PVA in the presence of chitosan.

Results and Discussion:

The freeze-thawing method produced hydrogels with elastic properties similar to tissue. Increasing the cycle number has increased tensile

strength and stiffness of the hydrogels. Hydrogel surfaces are conducive to growth of both endothelial and smooth muscle cells. However, crosslinking of the hydrogels caused a 30% decrease in the water content (56%). SEM investigations showed that crosslinking leads to a rougher surface topography and loss of alignment of polymer crystals (Figure1). However, crosslinking is necessary for controlling the degradation rate of the scaffolds and a degradation test showed that crosslinking increased the resistance of the hydrogels to degradation.

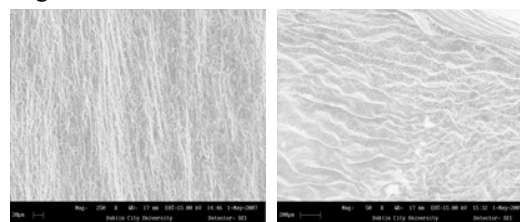


Figure 1. Effect of crosslinking on surface properties of PVA/Chitosan hydrogels.

The Main disadvantage of hydrogels produced by freeze/thawing is their low porosity which slows down cell penetration. Photocrosslinking may overcome this problem by incorporation of cells during crosslinking. Since hydrogels are highly permeable, gas and nutrient transfer is not limited to the encapsulated cells, which may decrease the necessary *in vitro* culture period.

Conclusion:

PVA/Chitosan hydrogels are very promising candidates for vascular tissue engineering scaffolds. Our studies showed that, freeze/thawing is an effective method to produce scaffolds with different physical properties in order to match the compliancy of the patient's vessels. Future studies will investigate photocrosslinking for decreasing production time and direct incorporation cells into the hydrogel network.

Acknowledgements:

This research has been supported by a Marie Curie Early Stage Research Training Fellowship of the European Community's FP6 under contract number MEST-CT-2005-020621).

EXPERIMENTAL FACILITY FOR NANOCUSTER FABRICATION USING LASER-PLASMA SOURCES

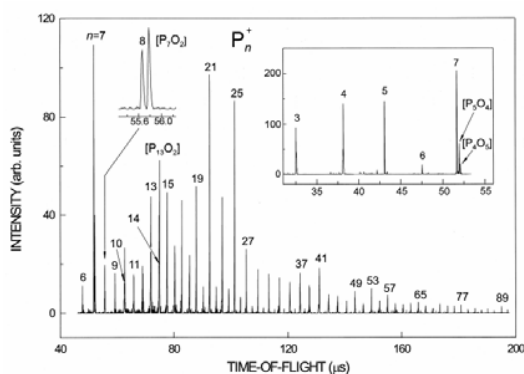
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We report the development of a new dynamic system of atomic, molecular and cluster source for materials deposition with a variation on the classical PLD method, with in-situ particle and optical-spectroscopic diagnostics. We plan to use the stagnation region of two colliding plasmas as a clusters source for deposition parallel to the expansion of the plume. This will allow the plume to flow across the top of the sample and therefore will encourage columnar growth. This growth will be analysed using a new Reflection Time of Flight (ReTOF) Mass Spectrometer, which will explore the clusters formation and their relative concentrations, tuning plasma conditions to achieve optimal growth conditions.

The mass spectrum will give detailed information about the ionized clusters in the plume being deposited on the sample and their relative abundances. An example of a mass spectrum of phosphorus is given below.

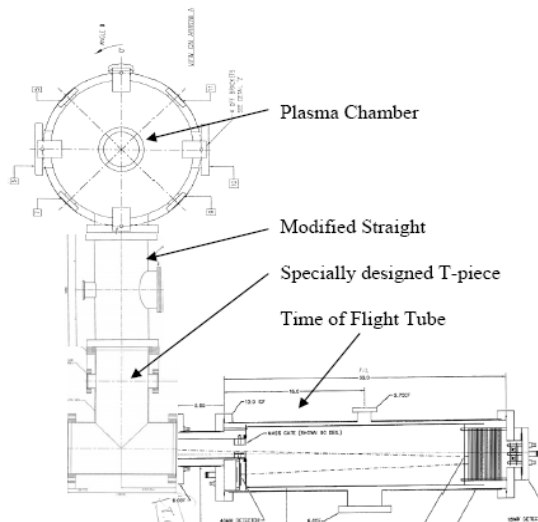


Time of Flight Mass Spectrum of Phosphorus cluster cations produced by laser ablation [1]
Figure 1

When two plasmas collide there are two scenarios that can play out - the plumes can either interpenetrate or stagnate. The ion-ion mean free path scales as T_e^{-2} , the electron temperature, so high temperature plasmas will tend to interpenetrate whereas low temperature

plasmas tend to stagnate, i.e. the two plumes decelerate rapidly to form a high density, relatively hot layer, the so-called stagnation layer. This layer is capable of producing exotic chemistries yet to be explored for materials deposition.

In addition to time of flight mass spectrometry and optical spectroscopy we will add laser interferometry and Thomson scattering to give a more detailed picture of the plasma parameters in the stagnation region.



Vacuum Schematic of the Colliding Plasma PLD chamber with Time of Flight Mass Analysis
Figure 2

The main focus of our deposition shall be the group II-VI semiconductor Zinc Oxide (ZnO). ZnO is a wide band gap semiconductor known to produce a large variety of nanomaterials and is at the cutting edge of semiconductor research with promising applications in sterilisation coatings [2].

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DESIGN OF AN INTEGRATED SEMI-AUTOMATED POWDER FEED DEVICE FOR HVOF / PLASMA SPRAY PROCESSES TO PRODUCE FUNCTIONALLY GRADED MATERIALS (FGM) OF Ti ALLOY AND HA POWDERS FOR BIOMEDICAL APPLICATIONS

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Introduction Functionally Graded Materials (FGMs) are a growing application area with significant promise for the future production of; (a) improved materials and devices for use in applications subjected to large thermal gradients, (b) lower-cost clad materials for combinations of corrosion and strength or wear resistance, and (c) improved electronic material structures for batteries, fuel cells, and thermoelectric energy conversion devices and (d) biomedical implant devices for enhanced bone-tissue attachment. The most immediate application for FGMs is as Thermal Barrier Coatings (TBCs), where large thermal stresses can be minimised. Component lifetimes are improved by tailoring the coefficients of thermal expansion, thermal conductivity, and oxidation resistance. The application of FGMs is quite difficult, but thermal spray processes like Plasma spray have demonstrated their unique potential in producing graded deposits, where researchers have used twin powder feed systems to mix different proportions of powders. FGMs vary in composition and/or microstructure from one boundary (substrate) to another (top service surface), and innovative characteristics result from the gradient from metals to ceramics or non-metallic to metals.

Research The present study investigates an innovative modification of a HVOF (High Velocity Oxy-Fuel) thermal spray process to produce functionally graded thick coatings. In order to deposit thick coatings, certain problems have to be overcome. More specifically these problems include minimising residual stress, which causes shape distortion in as-sprayed components. Graded coatings enable gradual variation of the coating composition and/or microstructure, which offers the possibility of reducing residual stress build-up with in coatings.

In order to spray such a coating, modification to a commercial powder feed hopper was required to enable it to deposit two powders

simultaneously which allows deposition of different layers of coating with changing chemical compositions, without interruption to the spraying process (Figure 1). Various concepts for this modification were identified and one design was selected, having been validated through use of a process model, developed using ANSYS Flotran Finite Element Analysis. In the current research the mixing of different proportions of powders were controlled by a computer using Lab VIEW software and hardware, which allowed the control and repeatability of the microstructure when producing functionally graded coatings. This research has been carried out on DCU HVOF Feed unit system. However this semi-automated powder feed unit system could be use with Plasma techniques to produce FGM coatings of Ti alloy and HA powders for Biomedical Applications.

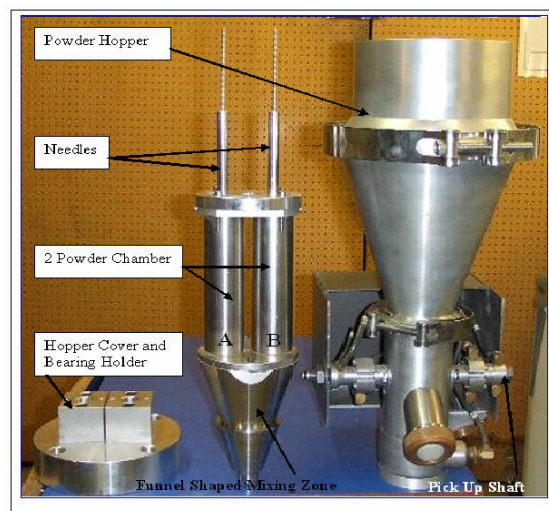


Figure 1: Digital picture of the newly designed dual powder feed device.

NANOSTRUCTURED ZINC OXIDE THIN FILMS GROWN BY THE NANOPARTICLE-ASSISTED PULSED LASER DEPOSITION TECHNIQUE

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Considerable research is presently being carried out to investigate both the growth and physical properties of ZnO epitaxial material and related devices to be used in UV optoelectronic, spintronics, gas and surface acoustic wave sensors, and transparent electronics [1,2].

Here, we report the fabrication of self-assembled ZnO nanostructures using PLD and the associated structural and optical properties. All nanostructures were grown at a substrate temperature of 650 °C by ablating a 5N-pure ZnO target with 266 nm laser light at a repetition rate of 10 Hz ($\tau = 6$ ns) onto (11 $\bar{2}$ 0) Al₂O₃ substrates (*a*-plane sapphire). They were annealed after growth for 30 min in pure oxygen at the growth temperature. The effects on the nanostructures' morphology of varying the composition of the background gas from pure oxygen to pure argon while keeping constant the total pressure (3.5×10^{-1} mbar) were studied; an evolution from compacted nanorods to terraced, hexagonal nanopyramids, respectively, was evidenced (Fig. 1). Additional work was done to identify the precursor growth mechanisms of these nanostructured films by decreasing the deposition time. The evolution of the morphology was observed using High-resolution Scanning Electron Microscopy (HR-SEM) and Atomic Force Microscopy (AFM).



Fig 1: This HR-SEM is a magnification (scale bar = 1 μm) of hexagonal nanopyramids. This was grown under an equal mix of Ar/O₂. As the particles land on the surface, there is a lower potential for the particle to nucleate a growth site at the flat regions (i.e., near the centre) or next to an edge rather than to locate itself on top of an edge. This is due to a greater attraction due to surface potential. This process continues until this terraced pyramid shape forms.

The corresponding XRD and photoluminescence data, measured at room or low temperatures, are also presented and testify to the high crystalline and optical quality of these nanostructures, respectively. It has been found that despite the large change in morphology, the crystalline properties remained rather constant [3].

Additionally, we report on the properties of ZnO nanostructured thin films grown on either bare or gold-patterned *a*-plane sapphire substrates. The pulsed laser deposition technique was used to deposit all the films at a temperature of 700 °C in a mixture of oxygen and argon under a total pressure of 3.5×10^{-1} mbar. SEM surface characterizations typically showed pyramidal nanostructures with hexagonal symmetry and a coverage density strongly dependent on the O₂ partial pressure (Fig. 2). X-ray diffraction results confirmed the high crystalline quality of the nanostructures, with the rocking curve widths of the (0002) reflection as low as 0.09°. Similarly, photoluminescence results at room temperature testifying to the high optical quality of the material is displayed [4].

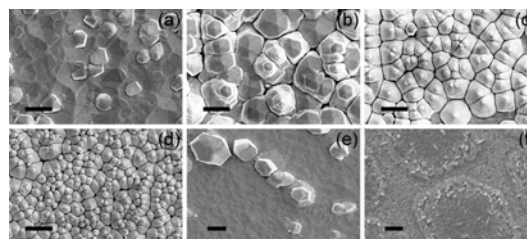


Figure 2: SEM images of ZnO nanostructured thin films grown on sapphire with partial O₂ concentrations under a total pressure of 35 Pa with O₂:Ar concentration ratios of (a) 0%:100%, (b) 25%:75%, (c) 50%:50%, (d) 75%:25%, and (e) 100%:0%, respectively. The final image (f) was grown on gold-patterned islands atop the sapphire under conditions similar to (c). The scale bar is 1 micron in length for images (a)-(e) and is 2 microns for image (f).

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INACTIVATION OF POLYPHENOLOXIDASE AND PEROXIDASE IN APPLE JUICE BY PULSED ELECTRIC FIELDS

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Pulsed electric field (PEF) treatments were applied to freshly prepared apple juice using a laboratory scale continuous PEF system to study the effect of this technology on the inactivation of polyphenoloxidase (PPO) and peroxidase (POD) compared to conventional pasteurisation (72 °C; 26 s). In this study, square wave pulses (1 micro s pulse width, 15 Hz frequency) were used with three different electric field strengths (20, 30 and 40 kV/cm) and three treatment times (25, 50 and 100 micro s) in a 3 × 3 factorial arrangement. The kinetic data for the inactivation of both enzymes could be described with a 1st-order model ($P < 0.001$). Higher electric-field strengths (40 kV/cm) and longer

total treatment times (100 micro s) led to a decrease in the enzymatic activity by 59% and 52%, for PPO and POD respectively.

This level of inactivation was significantly higher ($P < 0.05$) than that recorded in juice processed by conventional pasteurisation, where the activity of PPO and POD decreased by 46% and 48%, respectively. It can be concluded that the PEF method was more effective in inhibiting PPO and POD activity than conventional pasteurisation.

Keywords: Apple juice; PEF; Peroxidase, Polyphenoloxidase

OPPORTUNITIES FOR PLASMA PROCESSES FOR BIOMEDICAL APPLICATIONS RESEARCHERS IN EUROPEAN FRAMEWORK 7

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The European Union supports research activities covering almost all disciplines through the “Framework Programmes” (FPs). They have been implemented since 1984 and FP7 has been operational since the 1st of January 2007 and run until the end of 2013.

FP7 is designed to build on the aims of its predecessor towards the creation of a European Research Area and the development of a knowledge based society within Europe.

FP7 is organised in four programmes corresponding to four basic components of European research:

Cooperation

Support for industry-driven research activities carried out in trans-national cooperation, from collaborative projects to the coordination of national research programmes. Cooperation between the EU and third countries (US, China etc.) is an integral part of this action. The Cooperation programme is sub-divided into ten distinct research themes

Ideas

Support for excellence of European research at the frontier of knowledge in all scientific and technological disciplines. The newly formed action will be overseen by a “European Research Council”..

People

Support to improve the quality of the human resources in European R&D and promote a career path in research, encouraging European researchers to stay in Europe and attracting the best researchers from around the world

Capacities

Support research infrastructures, research for the benefit of SMEs and the research potential of European regions.

In addition, there will be a “Specific Programme” for the Joint Research Centre (non-nuclear activities) and one for Euratom (nuclear research and training activities).

The overall budget for FP7 exceeds €50 billion. However, researchers often perceive participation in FPs to be difficult to achieve and the processes involved beaucratic. This poster presentation will show how it is possible to be successful in FP7, the support mechanisms in place and the considerations that need to be made in determining the level of participation.

It will be shown that FP7 is not just about large, unmanageable networks and there are levels of participation that suit everyone, including simply becoming an evaluator to get used to the processes involved in reviewing proposals. Particular emphasis will be placed on opportunities for researchers in the area of plasma processes for biomedical applications.

A COMBINATION OF ULTRAVIOLET IRRADIATION, MILD PRE-HEATING AND PULSED ELECTRIC FIELDS FOR INACTIVATION OF *STAPHYLOCOCCUS AUREUS* IN APPLE JUICE

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The application of pulsed electric fields (PEF) as an emerging technology in combination with other novel technologies could represent a possible alternative to conventional high-temperature short-time (HTST) pasteurisation, which could achieve comparable microbial inactivation while potentially improving product quality.

A hurdle strategy including batch ultraviolet irradiation (UV), continuous pre-heating and PEF treatment (1µs square-wave pulse, 15 Hz frequency) was used on apple juice made from concentrate. The reconstituted juice was used for the analysis of physical properties, but it was autoclaved (15 min, 121°C) prior to microbial inoculation. The autoclaved juice (15 min at 121 °C) was then inoculated with *Staphylococcus aureus* in order to produce an initial microbial population of approximately 10¹⁰ CFU/ml. The juice was subsequently exposed to UV (30 min, 20 °C) and the PEF was applied with four different levels of pre-heating temperature (35, 43, 46 and 50 °C), electric field strength (28, 32, 36 and 40 kV/cm) and total treatment times (25, 50, 75 and 100 µs). A 3x3 orthogonal design was used with pre-heating temperature, field strength, and treatment time as main factors. In turn, each factor was kept constant, while the two remaining factors were varied over four levels, so that any two factors were never repeated in

different permutations. The effects of individual factors on microbial inactivation as well as on conductivity, pH, °Brix and colour (Hunter L*a*b colour space) were analysed and compared to a control treatment, consisting of HTST pasteurisation of the juice (26 s, 94 °C).

The highest level of bacterial inactivation was reached under the most severe treatment conditions applied (40kV/cm, 100µs and 46°C), significantly higher than HTST pasteurisation (9.5 vs. 8.2 log cycles, P < 0.05). Different pre-heating temperatures showed no significant effect on microbial inactivation (P > 0.05), while

there was a significant microbial reduction due to field strength and treatment time, in both cases following a linear pattern (P < 0.05). No significant changes in the physical properties were detected between the samples processed with the hurdle treatment approach and the HTST samples (P > 0.05).

It can be concluded that the combined impact of UV irradiation, mild pre-heating and PEF was more effective towards inactivation of *Staphylococcus aureus* in apple juice than conventional HTST pasteurisation. Moreover, from a product quality perspective, neither treatment caused any significant change to the physical properties of the apple juice compared to the untreated reconstituted juice.

Keywords: *Staphylococcus aureus*; Apple juice; PEF; Ultraviolet Irradiation; HTST; Hurdle strategy