

Monday, October 13th Sessions 1 & 2

Next-generation fluorescent nanodiamond for biological quantum sensing

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Abstract

Nitrogen-vacancy color centers in fluorescent diamond nanoparticles (NDNV) possess a uniquely coupled optical output related to their spin states, which, in turn, are highly sensitive to external fields and their local environment. This allows NDNV fluorescence to be selectively modulated through external electromagnetic fields, making diamond particles well-distinguishable in biological systems where high fluorescence background limits the use of other fluorophores. This capability is being advanced beyond background-free imaging to quantum sensing of local electromagnetic fields, free radicals, temperature, and pH with nanoscale resolution. The translational capability of such quantum sensors stems from improved sensitivity, resolution, and speed with respect to classical methods; however, successful implementation requires further fine-tuning of the NDNV material properties. One of the most impactful features in the design of quantum-grade NDNV is the substitutional nitrogen content, which needs to be an order of magnitude less than in commercially available starting particles typically used for production of NDNV for imaging applications. In parallel, the content of other parasitic paramagnetic impurities in the diamond lattice needs to be minimized. Finally, engineering of the particle surface states is required to stabilize the charge state of the NV centers. We discuss recent developments of fluorescent NDNV, including commercial synthesis of NDNV with low nitrogen content, annealing of particles at ultra-high temperature, including annealing at high (few GPa) pressure, and atmospheric plasma-based production of surface termination with negative electron affinity for NV charge stabilization. The outcome of these efforts is a pathway for production of next-generation nanodiamond particles adapted for quantum sensing applications as deployable biological sensors.

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Keywords: fluorescent nanodiamond, nitrogen, vacancy centers, quantum sensing

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Fabrication and opto-physical characterization of Magnesium-Vacancy color centers in nanodiamonds

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Abstract

Color centers in diamond are promising canditates for quantum technologies. The nitrogenvacancy (NV) center is the most extensively studied, owing to its optically addressable spin state, which has enabled appealing pathways in quantum sensing and information processing. However, intrinsic limitations, such as broad spectral emission and relatively long radiative lifetime, can hinder its performance in certain quantum applications, motivating the exploration of new classes of color centers.

In this context, the magnesium-vacancy (MgV) center has recently emerged as a compelling alternative, featuring intense photoluminescence with a narrow zero phonon line (ZPL) and a shorter radiative lifetime (1). Additionally, the temperature dependence of MgV emission has been experimentally demonstrated and its strain sensitivity has been theoretically predicted (1,2)

In this work we report on the opto-physical characterization of these emitters embedded in nanodiamonds (20-100 nm), previously surface-treated to reduce external graphitic phases (3). The MgV centers were fabricated upon 50 keV MgH- ion implantation and subsequent thermal annealing. A systematic photoluminescence investigation was performed at room temperature both at ensemble and at single-defect levels, comparing the results with MgV centers in bulk diamond. Single-emitter analysis revealed that MgV centers in nanodiamonds maintain radiative lifetimes comparable to their bulk counterparts, while exhibiting distinct spectral features. The fine structure observed in the ZPL (Fig.) is attributed to strain-induced effects, suggesting that internal stress in nanodiamonds could modulate the MgV centers emission.

These findings open new avenues for the exploitation of MgV centers and of their tunability at the nanoscale, highlighting the potential of strain engineering to control the optical properties and the spin state of this quantum emitter.

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 $\textbf{Keywords:} \ \ \text{color centers, ion implantation, single photon sources, strain}$

Scalable synthesis of SiV-doped nanodiamonds via bottom-up and top-down CVD approaches

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Abstract

Nanodiamonds (NDs) doped with silicon-vacancy (SiV) centers are emerging as versatile platforms for applications in quantum sensing, bioimaging, and photonics due to their stable and bright photoluminescence at room temperature. This study presents a comparative analysis of two complementary chemical vapor deposition based approaches for synthesizing SiV-doped NDs: a bottom-up (BU) employing porous diamond film growth followed by Si-doped overgrowth, and a top-down (TD) based on molten salt thermal etching of nanocrystalline diamond films. Both approaches result in brittle, porous diamond structures that are subsequently disintegrated into nanodiamonds using ultrasonic processing. Detailed morphological, structural, and optical characterizations confirm the successful incorporation of SiV centers with distinct spatial distributions and photoluminescence characteristics. The BU method yields core-shell-like NDs with surface-localized SiV centers, while the TD method yields homogeneousl core-like doped NDs. Furthermore, temperature-resolved photoluminescence measurements reveal linear redshifts and broadening of the SiV zero-phonon line, demonstrating their potential for nanothermometry applications. These scalable fabrication routes offer a high-yield alternative to traditional methods, paving the way for the development of tailored nanodiamond materials in next-generation sensing technologies.

Keywords: na	anodiamonds,	silicon, v	vacancy	centers,	${\it chemical}$	vapor	deposition,	molten	salt th	ıermal
etching, photolumi	nescence, nano	othermon	netry							

^{*}Speaker

Controlled HPHT annealing of SiV-doped nanodiamonds at SOLEIL synchrotron: structural and optical investigations

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Abstract

Diamonds and nanodiamonds (NDs) containing quantum color centers, such as nitrogen-vacancy (NV) centers, have been widely studied in recent years for their promising applications across various fields, including cryptography and telecommunications, medical sciences, and sensing. In the context of sensing, recent studies on color centers such as silicon-vacancy (SiV) and germanium-vacancy (GeV) centers have shown great potential for measuring physical quantities like magnetic fields, temperature, and strain. This is due to their intense zero-phonon line (ZPL) optical signal, about 80% of their total luminescence, which is characteristic of group-IV color centers (G4V) (1). In our recent papers, we demonstrated that

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as-grown CVD NDs containing SiV and GeV centers, featuring luminescent ZPLs and excellent photostability at room temperature, can function effectively as nanosensors of high stress and pressure conditions (up to 180 GPa) (2, 3). To advance our research, in this work we investigate the optical properties of SiV centers in as-grown NDs at cryogenic temperatures. For SiV centers, which have spin 1/2, the fine structure should become accessible through the Jahn-Teller effect, which lifts the degeneracy at cryogenic temperatures (< 20 K). However, experiments on our as-grown NDs reveal significant spectral broadening even at 9 K, caused by lattice strain, which prevents access to the optically addressable electronic spin states of the SiV center (see Fig.). To overcome this limitation, we propose an original post-treatment involving high-pressure and high-temperature (HPHT) annealing using the Paris-Edinburgh press. In fact, the gradual increase of temperature and pressure allows for control over the undesired diamond-to-graphite phase transition. HPHT annealing experiments were conducted at the PSICHE beamline of Synchrotron SOLEIL (4), where X-ray diffraction and tomography analyses enabled precise monitoring of P,T parameters and made it possible for the first time to access single fine-structure transitions of the in such SiV-NDs (see Fig.).

Figure. PL spectra of SiV-NDs before (orange line) and after (blue line) the HPHT annealing treatment registered at 9 K.

Keywords: HPHT annealing, SiV, doped nanodiamonds, SOLEIL synchrotron investigations

Advanced characterizations of nanodiamonds by synchrotron-based techniques

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Abstract

Easy generation of solvated electrons from solar illumination represents a significant advancement for green chemistry and environmental applications. Diamond materials have been proposed as promising emitters of solvated electrons1. Visible-light excitation could enable solar-driven CO or N reduction reactions in aqueous media.2,3 Recent developments in electrode design have shown increased activity even under visible light.4 However, subbandgap excitation remains challenging. Our recent studies have demonstrated that while sub-bandgap surface states can be excited in air, leading to charge separation and trapping,5 this does not necessarily translate to photocurrent generation in electrolytes.6 Therefore, a thorough investigation of the excitation pathways at the interface is required to engineer new materials.

In this work, we explore the strategy of functionalizing nanodiamonds with transition metal dyes to facilitate visible-light excitation of solvated electrons. Using soft X-ray spectroscopy techniques, we discuss the role of surface modifications on nanodiamond surface states and charge transfers at the interface between the nanodiamond and the electrolyte. These results open new perspectives for solar-driven emission of solvated electrons in aqueous phases using nanodiamonds.

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Keywords: Diamonds, Surface, Interface, X, ray absorption spectroscopy, X, ray Photoemission Spectroscopy, Photoexcitation, Charge Transfers

Extreme surface chemistry of isolated nanodiamonds probed by synchrotron X-ray photoemission

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Abstract

Nanodiamonds (ND) are currently under active investigation for their valuable and combinable properties in the fields of energy, quantum applications and nanomedicine1–3. ND inherits its semiconducting behavior from bulk diamond. The surface chemistry strongly governs the physicochemical properties of ND: band structure, interaction with solvents in colloids, photoluminescence of hosted color centers. To shine a light on these different properties of ND, especially the photoreactivity of ND suspended in water under illumination, an investigation of their extreme surface chemistry is required to appreciate the real interface formed with water molecules when ND are in colloidal suspension.

At the PLEIADE beamline (SOLEIL synchrotron), a jet of isolated (or slightly aggregated) nanoparticles is produced from an aqueous colloid through a nebulizer and an aerodynamic lens4. The jet of nanoparticles intersects the synchrotron photon beam and emitted photoelectrons are then analyzed in energy. The energy of the incident photons can be adjusted to probe the extreme surface of the nanoparticles (a few tenth of nm for diamond) by X-ray photoelectron spectroscopy (XPS). The present study compares the extreme surface chemistry for isolated hydrogenated and oxidized milled nanodiamonds (MND) suspended in jets. The combination of synchrotron XPS data with conventional XPS results allowed us to build band diagrams for both milled nanodiamonds. A negative electron affinity (NEA) is obtained for H-MND whereas the affinity turned to positive for Ox-MND. As a comparison, hydrogenated DND were analyzed in the same experimental conditions.

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Keywords: nanodiamonds, XPS, surface chemistry

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Fluorination: a smart approach to designing high-value sp³ carbon-based nanomaterials-diamane and ultrapure nanodiamonds

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Abstract

Most studies on fluorinated (nano)carbons focus on gas/solid treatment of sp2 hybridized materials (graphite, graphene, nanotubes, fullerenes). The aim is to functionalize the bulk or surface for applications in energy storage, lubrication, band gap engineering, hydrophobicity, or composites. Carbon materials with sp3 hybridization are less explored for gas/solid fluorination. The low reactivity of diamond carbons with elemental fluorine is valuable for high-value applications. Fluorinating detonation nanodiamonds (NDs, 4-5 nm) removes the sp2 carbon shell and hydrogenated impurities (COH, COOH, CH). Chlorination of metallic impurities gives ultrapure NDs, ideal as neutron reflectors. Fluorine enhances stability in air, shifting decomposition temperature by 200°C. Fluorine also delays diamond-to-graphite conversion, similar to diamane, a bilayer of diamond carbons. Diamane is metastable at ambient temperature and pressure without stabilization by heteroelements (Cl, H, F) or hydroxyl groups (-OH). Fluorine is essential for preparing diamane, including exfoliation of fluorinated graphite (C2F)n, which consists in stacked fluorinated diamane. Fluorine-stabilized diamane are promising in optical and photonic applications like lenses, coatings, and waveguides. Diamane, like diamond, is an excellent insulator with high dielectric strength. Its hardness and stability under extreme conditions offer advantages over other materials. Fluorine-stabilized sp3 hybridized materials present a promising synthesis route, as controlled defluorination may induce partial graphitization. This allows preparation of ultrapure nanodiamonds with a perfect sp2 carbon shell, functionalized for heterogeneous catalysis under harsh conditions (e.g., high-temperature HF reactions). The core-shell structure, with a stable diamond core and functionalized sp2 carbon shell, enables well-controlled catalytic sites with enhanced stability.

Keywords: Fluorination, diamane, thermal stability

^{*}Speaker

Nanodiamond band gap engineering: towards visible range photo activity

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Abstract

Nanodiamonds are gaining attention as cutting-edge materials for photocatalysis. While their wide band gap might seem like a limitation, their highly tunable band structure, strongly governed by their surface chemistry, unlocks possibilities for critical reactions, such as CO2 reduction1 or PFAS degradation2. Initially, photocatalytic activity was demonstrated under UV light1,2, but recent breakthroughs involving our team revealed their potential under visible light3,4. To push visible light photoactivity further, enhancing light absorption is key. A promising approach lies in embedding new graphitic energy states into the band gap, enabling the absorption of lower-energy photons3.

Band gap engineering, involving the introduction of intermediate energy levels, can be achieved through the formation of $\rm sp^2$ -hybridized carbon surface reconstructions. Well-controlled graphitization of highly crystalline nanodiamond offers the potential to incorporate $\rm sp^2$ carbon on the surface while maintaining the integrity of the diamond crystalline core.

We achieved controlled graphitization of nanodiamonds by annealing at low pressure within a temperature range of 800–1100°C. The resulting nanodiamonds were characterized using XRD, XPS, and Raman spectroscopy, confirming the formation of $\rm sp^2/sp^3$ hybrids and revealing that the $\rm sp^2$ content increases with rising temperature. Further analyses using HR-TEM and multi-wavelength Raman spectroscopy demonstrated that the surface reconstructions are highly dependent on particle size. Nanodiamonds were sorted prior to graphitization following a protocol previously developed5. The enhanced visible absorption of graphitized nanodiamonds was validated through Time Resolved Microwave Conductivity (TRMC) measurements that allow to investigate the dynamics of charge carriers under laser illumination. Nanodiamonds were illuminated from 290 to 500 nm, enabling the evaluation of the number and mobility of charge carriers within this wavelength range.

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Keywords: nanodiamonds, surface chemistry, TRMC

Tuesday, October 14th Sessions 3 & 4

Gold and Silver Decorated Nanodiamond are multifunctional nanoparticles for generating solvated electrons.

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Abstract

Gold nanoparticles (AuNPs) and nanodiamonds (NDs) are extraordinary multifunctional nanomaterials with unique properties and a wide range of potential applications. The teamwork between AuNPs and NDs is related to the ability to control the shape and size of gold nanoparticles on the surface of nanodiamonds, modulate the colloidal properties, and their mutual interaction. This hybrid system can exhibit distinct catalytic and sensing activities, as both components contribute to these capabilities. Recently, it has been demonstrated that this system can efficiently generate solvated electrons using visible light, paving the way for numerous catalytic and synthetic applications. Similar findings were observed with silver-decorated ND particles. This lecture will discuss the synthetic approach for formulating hybrid nanosystems AuNPs-NDs and AgNPs-ND, as well as their structure, morphology, and efficiency in generating the solvated electrons.

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Keywords: gold nanoparticles, silver nanoparticles, Nanodiamonds, solvated electrons.

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Nanodiamond Technologies for High-Temperature Concentrated Solar Cells

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Abstract

High-temperature solar cells and thermal energy converters are feasible by exploiting temperature-driven mechanisms, such as thermionic-thermoelectric generation (1), thermionic-photovoltaic conversion (2, 3), and photon-enhanced thermionic emission (PETE) concept, which represent novel and promisingly efficient (> 40%) mechanisms for the exploitation of concentrated sunlight. More advanced PETE converters rely on the concept that engineered nanodiamond photocathodes can provide an efficient electron emission, obtained by a synergistic combination of photogeneration and thermionic emission.

Two nanodiamond-based conversion technologies are pursued:

- 1) fs-laser nanotextured diamond as photoelectronically active material in black diamond PETE cathodes (4);
- 2) ultra-thin nanodiamond films grown on silicon absorbers in heterostructured PETE cathodes (5).

Results under a high-flux solar simulator are reported and discussed by demonstrating for the first time the PETE conversion at temperatures from 300 to 525 oC.

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Keywords: Solar energy, electron emission, thermionic energy conversion

Laser nanotextured diamond for sunlight conversion

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Abstract

Diamond is completely solar-blind, and it cannot natively be applied for solar applications. To overcome this limitation, it has been proposed the use of diamond in thermionic-based converters, which are solid-state heat engine enabling the absorption of concentrated sunlight and transform it by the thermal emission and the collection of electrons. The application of a defect engineering strategy, that leads to the formation of nanotextured diamond with surface features below 200 nm, allows using freestanding diamond plates to be used as both solar receiver and electrons' emitting layer, i.e., in photon-enhanced thermionic emission (PETE). Such a strategy is enabled using ultra-short laser treatments, that are the key enabling technology for achieving this novel and disruptive concept. Additionally, this kind of structure has been proposed also for the hydrogen production by solar water splitting (SPEEDHY project, www.speedhy.it) since diamond represents a potential efficient source of solvated electrons in aqueous environment thanks to its chemical inertness and stability, and the wide electrochemical window. Finally, the laser technique can be potentially applied also to dope and functionalize nanodiamonds, for quantum and biomedical applications.

Keywords: ultra, short laser treatments, nanotexturing, electron emission.

^{*}Speaker

Optimizing Nanodiamond Colloidal Suspensions: The Role of Surface Chemistry and Sonication Processes

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Abstract

In various applications, ranging from biomedicine to photocatalysis, nanodiamonds (NDs) have to be utilized in the form of aqueous colloidal suspensions to fully benefit from their extensive surface area. To achieve these colloidal suspensions, NDs must undergo ultrasound treatment, which serves to disaggregate the particles and stabilize them through their surface charge. However, the sonication process is complex and frequently overlooked in the literature, while it introduces sonochemical reactions that can significantly impact the final suspension in terms of concentration and composition. In this context, this study aims to investigate how the surface chemistry and characteristics of nanodiamonds influence the properties of the resulting colloid after a sonication process.

The first part of the study focuses on the proportion of nanoparticles that can be stabilized in colloidal suspension and how their surface chemistry influences this process. We evidence that, unlike other NDs, hydrogenated milled nanodiamonds (H-MND) exhibit an unusual behavior after successive sonication steps. Specifically, their concentration continues to increase after three sonication steps before decreasing to lower concentrations, following a more typical pattern. The mechanisms governing this particular resuspension behavior of H-MND in water are discussed, taking into account complementary characterizations of the colloids, including IR spectroscopy and Cryo-TEM (4).

Secondly, we show that sonication can generate nitrite and nitrate ions from dissolved N2 (3). Sonicating water with oxidized detonation nanodiamonds (Ox-DND) increases nitrite production with higher concentrations, up to 15 μ mol/L in a 4 wt% solution, compared to 1 μ mol/L without Ox-DND. Surprisingly, H-DND do not show such an increase, raising questions about their interaction with the radicals formed during sonication. Our results also show that the formation of these species can be avoided in suspension by sonicating under a controlled atmosphere with no significant perturbation of ND colloidal stability or surface chemistry.

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 $\textbf{Keywords:} \ \ \text{Colloids, radicals, sonochemistry}$

Spatially resolved temperature sensing in working electrochemical devices by nanodiamond quantum sensors

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Abstract

Temperature plays a crucial role in the operation of electrochemical devices, serving as both an indicator of device efficiency and stability. However, the complex material architecture within the device, coupled with local variations in electrochemical activity, leads to a time-dependent and spatially varying temperature at the nanoscale. Information on the spatially resolved temperature evolution is challenging due to the lack of non-invasive and accurate nano-thermometry methods that meet the requirements for sensitivity, spatial resolution, and temporal resolution within the reactive chemical environment of a functional device. In this presentation, I will discuss our recent progress in developing nanodiamond based thermometry to monitor the spatially resolved temperature evolution in operational electrochemical devices, including miniaturized battery cells and electrolyzers. We show that the local temperature within the working device, especially at the reaction interface, is considerably higher than the spatially averaged temperature measured from outside of the device using conventional methods. Spatial temperature variation has been found in conjunction with the respective electrochemical processes, disclosing the site sensitive chemical reactivity and its dynamics. Challenges in realizing the spatially resolved temperature sensing with adequate sensitivity and temporal resolution in these working devices will also be discussed in terms of the sensor quality and the device interface. This work is carried out in collaboration with Renbao Liu, Wenghang Leung, Ruqiang Dou, Zan Li, Hui Wang, Yao Gao, Ying Wang, and Chunyi Zhi. The authors acknowledge funding support from RGC CRF C4004-23G.

Keywords: nanodiamond, temperature sensing, electrochemical devices

^{*}Speaker

Thermal mapping of heterogeneous materials using NV centers in highly doped nanodiamonds.

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Abstract

Heterogeneous materials like carbon/carbon (C/C) composites consist of multiple microscopic constituents, whose temperature-dependent properties are often poorly characterized. This lack of precise data poses a challenge to numerical modeling, which relies on accurate micro-scale inputs to predict how individual parameters affect the overall thermal behavior of the material. Due to the small size of these constituents, conventional measurement techniques are inadequate. Until now only photoreflectance microscopy has provided the spatial resolution needed to accurately assess the thermal properties of a carbon fiber with a material volume of few cubic microns. However, photoreflectance does not determine the absolute temperature of the sample. Moreover, standard implementations rely on scanning a probe beam to record the change of the refractive index induced at the sample surface by a focused heating laser. This point-by-point analysis results in a long data acquisition time (typically a few hours).

To overcome these limitations, we have developed a thermal measurement bench using nanodiamonds highly doped with NV centers as thermal sensors. Due to the temperature dependence of its spin ground states, the NV center is a promising nanoscale thermal probe. This non-invasive method provide a direct measurement of the sample temperature while maintaining the high spatial resolution of the photoreflectance method.

We have optimized the deposition of the nanodiamonds to achieve a uniform and dense coverage of the sample surface. The temperature map on the sample surface is obtained by adapting NV widefield magnetic imaging which offers a multiplex advantage over scanning measurements. This significantly reduces the acquisition time (10 minutes for a field of view of 100 microns large). Initial measurements show a linear shift of the optically detected spin resonance frequency with increasing temperature, validating the use of NV centers as nanoscale thermal probes. Preliminary results were obtained on a test sample made of nitrocellulose fibers. These results pave the way for improved thermal imaging microscopy, especially for materials under extreme conditions.

^{*}Speaker

Sensing with NV- nanodiamonds: a spin active molecule and a metallic effect case

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Abstract

Negatively charged Nitrogen-Vacancy (NV) defects in diamond are a notable platform to study spin and magnetic related properties of any system. In this study we explore the potential of these centres in nanodiamonds (ND) for high-resolution spin sensing and temperature dependent measurement at the nanoscale. The size of nanodiamonds are an advantage to ensure that the read-out probe (NV) is always in close vicinity to any desired material on the diamond surface. Here we demonstrate the pros and cons of ND usage through the following two cases.

The spin active molecule azafullerene (C59N) stands out from other fullerenes, especially when encapsulated in cycloparaphenylene (CPP) (1). The created supramolecular complex (10)CPP@C59N shows desirable spin coherence times (T1, T2) and spin lifetime at room temperature (2), as well as having significantly higher yields than comparable fullerene-like molecules (desired for scalability) (3). We simulate the interaction between (10)CPP@C59N and NV using density functional theory (DFT). Addressing the stability and coupling between the vacancy and the separated molecules allows us to identify band bending behaviour and induced charge doping effects, leading to a surface termination discussion. Experimentally using ND photoluminescence we link our calculations with optical detected magnetic resonance (ODMR), Rabi oscillations and T1 relaxometry performed on ND mixed with azafullerene samples.

Utilising a cryostat in this NV experimental set-up we are also able to study the temperature dependence of NV properties (ODMR, Rabi, T1). Taking as reference ND on insulator we discuss the practicality of NV in ND to detect a metallic effect, namely Johnson noise, on Au surfaces. This allows us to draw important conclusions concerning sample preparation, particle agglomeration and ND size effects.

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^{*}Speaker

 ${\bf Keywords:}\ \ {\rm NV},\ {\rm Fullerene},\ {\rm Spin}\ \ {\rm Active}\ \ {\rm Molecule},\ {\rm DFT}.$

Time-Resolved Thermal Sensing using Nanodiamond Cathodoluminescence Spectroscopy

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Abstract

As electronic devices become smaller, measuring their core properties with an adequate spatial resolution becomes increasingly more challenging. Particularly, local defects can create "hot spots" due to joule heating during operation, which can negatively affect device performance(1). Measuring device temperature and gradient under operation with an adequate spatial resolution would allow for better understanding of the mechanisms that currently limit device performance.

Our study seeks to achieve this goal by studying the cathodoluminescence (CL) properties of Nitrogen-doped nanodiamonds (ND). CL provides much larger spatial resolution that the commonly used PL thermal probes, since electrons have much shorter wavelength than photons. According to our simulations we can reach a spatial resolution of 13 nm, corresponding to the width of the electron penetration volume in nanodiamond.

Nitrogen doping leads to the formation of NV0 centres, which have a CL emission peak at a wavelength of 575 nm(2). We have already demonstrated the potential of the NV0 CL peak in nanodiamond as thermal probe(3). By measuring peak width using a Voigt profile, we are able to calculate the ND temperature in the 200 to room temperature range, with a spatial resolution of potentially up to 1 K.

Recently, we have been able to measure the heating originating from a μ LED circuit using the CL emission of ND dispersed on top of the sample. We have been able to measure the relationship between dissipated power and temperature, in both forward and reversed bias.

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Tuesday, October 14th Sessions 5 & 6

Synthesis and surface functionalization of CVD nanodiamond containing SiV color centers for quantum sensor applications

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Abstract

The synthesis and functionalization of quantum nanodiamonds (NDs) is a key step toward technological innovation in biomedicine, cryptography, and sensing (1, 2). While the synthesis of diamond nanoparticles already poses a significant challenge due to the need for precise control over their size and purity, the effective incorporation of specific defects with well-defined quantum properties is no less demanding. Among these, the so-called siliconvacancy (SiV) color centers present an additional difficulty, as they are typically introduced using solid-state sources (most often via post-synthesis implantation). However, they also hold great promise due to their unique properties: high photoluminescence (PL) of the zerophonon line (ZPL), excellent photostability, reduced sensitivity to external electric fields, and an optically addressable electronic spin state (3). During the last years, we developed a consolidated protocol to synthetise high-quality NDs by microwave plasma Chemical Vapor Deposition (CVD) technique with Si impurity incorporation starting from a solid source and efficiently generating SiV color centers (4). This work presents a study of the physicochemical properties of the particle surfaces, along with a statistical analysis of particle size and species obtained via the CVD process. The NDs were characterized using various techniques, including zeta potential measurements, dynamic light scattering, and scanning electron microscopy (SEM). For the first time, experiments on the manipulation of surface terminations

^{*}Speaker

have been conducted, yielding promising results regarding their influence on the PL of the SiV ZPL. These characterizations were performed using X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and PL spectroscopy. This study paves the way for biochemical applications requiring surface functionalization, as well as for a deeper understanding and control of the neutral charge state SiV.

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Keywords: Synthesis, surface functionalization, CVD nanodiamond, SiV color centers

Characterization of diamond electrodes with H-terminated N-doped nanolayers on B-doped films for CO2 reduction by electrochemical impedance spectroscopy

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Abstract

The increasing atmospheric concentration of carbon dioxide (CO) is a primary driver of global climate change, necessitating the development of efficient CO conversion technologies (1). However, the electrochemical CO reduction reaction (CORR) requires a high applied overpotential due to the high thermodynamic stability of CO and is often accompanied by the competing hydrogen evolution reaction (HER), which reduces product selectivity. Diamond electrodes are promising for CORR as their high overpotential for HER suppresses hydrogen generation (2). Yet, selectively producing CO remains challenging, especially with diamond electrodes alone, due to the complex multi-electron/proton nature of CORR and the proximity of redox potentials for different pathways.

Hamers et al. demonstrated that electrons emitted from hydrogen-terminated diamond with negative electron affinity (NEA) under deep UV (DUV) light can selectively reduce CO to CO in aqueous media (3,4). However, diamond's wide bandgap ($\tilde{}$ 5.5 eV) necessitates DUV light, which is impractical for green chemistry applications. To address this, we developed a visible-light-responsive diamond-based photocatalyst. Boron-doped p-type diamond films were synthesized on detonation nanodiamond (DND) seed layers via chemical vapor deposition (5). Nitrogen atoms from DNDs were incorporated into the near-surface region, forming a heavily nitrogen-doped layer (> 10^{21} atoms/cm³) with optically active states enabling subbandgap absorption. These states facilitate visible-light-induced electron emission from the NEA surface, driving CO-to-CO conversion in water (6).

In the study, to discuss the potential of such electrodes, we present the results of electrochemical impedance spectroscopy and the correlation between the resulting electrical properties of the electrodes and their CO-to-CO reduction performance obtained through multiple regression analysis.

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Keywords: CO reduction reaction, negative electron affinity, nitrogen doped diamond, nanodiamond electrode, electrochemical impedance spectroscopy, multiple regression analysis

Engineering Nanodiamonds for Enhanced EPR Imaging: Influence of Size, Surface Chemistry, and Paramagnetic Properties

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Abstract

Electron paramagnetic resonance (EPR) spectroscopy is a powerful sensitive technique for the detection of unpaired electron spins, with a wide range of applications. It enables the specific detection and quantification of radical species and can produce high-contrast, background-free images. However, the use of EPR for labeling and imaging is often limited by the poor stability of organic radicals from conventional probes, which can compromise the reliability and versatility of the technique. In this context, nanodiamonds (NDs) offer a promising alternative, thanks to their intrinsic paramagnetic centers and unique physicochemical properties, which may broaden the scope of EPR-based biomedical applications. Here, we report a preliminary demonstration of a practical spectroscopy and imaging application using nanosized diamond particles (< 18 nm) for electron paramagnetic resonance imaging (EPRI). We compare nanodiamonds produced by two commonly used synthesis methods, high-pressure high-temperature (HPHT) and detonation, highlighting their distinct physicochemical characteristics. Additionally, we examine how variations in particle size and surface treatment influence their EPR performance. Finally, we present experimental evidence defining the optimal conditions for achieving high-resolution imaging (spatial resolution R < 1 mm) and enhanced EPR sensitivity, thereby underscoring the potential of nanodiamonds as robust contrast agents in EPR applications.

^{*}Speaker

 $\mathbf{Keywords:}\,$ EPR, Imaging, Raman spectroscopy, air, annealing

Surface-Modified Nanodiamonds for Enhanced Photocatalytic Hydrogen Generation

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Abstract

Among nanoscale semiconductors, nanodiamonds (NDs) have only recently begun to be seriously considered for photocatalytic reactions. This is due to the confusion with monocrystalline diamond which exhibits a wide bandgap (5.5 eV) and requires deep UV illumination (230 nm) to trigger its photoreactivity. At the nanometric scale, NDs possess native defects and chemical impurities like nitrogen, which introduce electronic states into the bandgap. These defects reduce the light energy required to initiate charge separation. This phenomenon has been confirmed by several studies, including one conducted by our team, which combined experimental results and Density Functional Theory (DFT) calculations (1, 2). Moreover, unlike other semiconductors, the electronic structure of NDs can be significantly modified (by several eV) by altering its surface chemistry (e.g., oxidized, hydrogenated, aminated). This allows for optimal adjustment of band alignments (3). In this context, our study focuses on the photocatalytic production of hydrogen from water splitting using nanodiamonds. Using our custom-built photocatalysis setup, we demonstrate that NDs can act as efficient photocatalysts even under solar illumination, with performances comparable to TiO (4). However, our results reveal that the surface chemistry and intrinsic properties of NDs play a key role in H2 production, which will be discussed. As with other semiconductor photocatalysts, it is required to enhance visible light absorption and limit charge carrier recombination to optimize photocatalytic performances. This can be achieved by combining NDs with other materials (metallic nanoparticles, other photocatalysts). The second part of our study presents strategies that can be applied specifically to nanodiamonds.

Overall, this study highlights the potential of nanodiamonds as a versatile and efficient material for solar-driven photocatalytic H2 production, paving the way for innovative applications in renewable energy technologies.

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Keywords: Photocatalysis, surface chemistry, colloids

^{*}Speaker

Optical Communication via Rabi Oscillations in NV Center Ensembles

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Abstract

Optical communication systems are crucial for the future of secure, high-speed data transmission, and recent advancements in quantum technologies show promising potential in revolutionizing this field. This project focuses on using **nitrogen-vacancy** (**NV**) **centers** in diamond as a medium for optical communication, with the ultimate goal of developing quantum communication schemes. The work explores the use of **microwave-induced Rabi oscillations** to control the quantum states of an NV center ensemble within a diamond sample, utilizing **Optically Detected Magnetic Resonance** (**ODMR**) for precise measurement and modulation.

The initial step in this research is to demonstrate basic message transmission by exploiting the **drop in photoluminescence (PL)** of NV centers induced by Rabi oscillations. In this system, **bit encoding** is achieved by **ON-OFF Keying (OOK)** modulation. This binary encoding scheme allows for the transmission of information in a quantum context, where the photoluminescence response of the NV centers serves as the key indicator for data transmission.

Through this approach, we aim to establish a proof of concept for using NV center ensembles in diamond as a foundation for optical communication systems. The precision and reliability of the ODMR technique, combined with the inherent advantages of quantum states in NV centers, provide an innovative platform for achieving robust and secure communication channels. While the current focus is on encoding and detecting basic messages, the long-term objective of this research is to extend these findings toward the development of **quantum communication systems**. By leveraging the quantum properties of NV centers, the project seeks to contribute to the broader goal of implementing secure, quantum-based optical communication networks, where the reliability and security of data transmission are enhanced by the fundamental principles of quantum mechanics.

This work not only pushes the boundaries of quantum communication but also paves the way for practical applications in quantum networks, which could become pivotal in securing the future of optical communication technologies.

Keywords:	Color	centers,	optical	commun	ication

^{*}Speaker

Boron- and phosphorus-doped diamond: modification of the electronic structure after exposure to a low-pressure deuterium plasma and its effect on the production of negative ions.

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Abstract

Boron- and phosphorus-doped diamond: modification of the electronic structure after exposure to a low-pressure deuterium plasma and its effect on the production of negative ions.

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Negative ion production is of significant interest for materials processing and high-energy (> 1MeV) neutral beam injection systems for magnetic confinement fusion reactors (1). The surface generation mechanism is necessary for high-current applications. Dielectric materials, including doped diamond, are attractive candidates for increasing surface generation as a potential alternative to low work function metals (2). To this end, it is necessary to better understand the underlying mechanisms of surface charge exchange in a deuterium plasma environment. In this study, we have developed an in-situ diagnostic photoemission yield spectroscopy (PYS). We use this spectroscopy, coupled with the Fowler model and mass spectrometry, to measure the negative ion yield and ionization threshold of HOPG, microcrystalline

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diamond (μ c-D), boron-doped microcrystalline diamond (μ c-BDD) and phosphorus-doped single-crystal diamond (PDD) under sample plasma exposures ranging from 30°C to 700°C.

We observe that exposure of the PDD to deuterium plasma at 400 C reduces its ionization threshold from around 4.0 eV to 2.1 eV, which is similar to the work function of cesium. Although the ionization threshold of diamond is sensitive to sample temperature and the dopant used, this appears to have a negligible effect on negative ion production. A better understanding of which material properties are most important for negative ion production will help in the development of improved negative ion sources.

Keywords: négative ions sources for fusion applications, Hydrogen plasma, diamond interaction, negative ions production

Conversion of textile microfibers into nanodiamonds by HF-CVD

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Abstract

In recent decades, the issue of microplastic pollution has garnered significant global attention. Microplastics and nanoplastics are ubiquitous contaminants: their small size enables their widespread dispersion across atmospheric, terrestrial, aquatic, and food systems. Beyond their physical presence, these particles function as carriers of toxic substances, presenting significant threats to ecosystems and human health. Among the various sources, synthetic microfibers released during the textile laundering are identified as primary contributors to marine microplastic pollution. This study investigates a sustainable upcycling approach wherein polypropylene (PP) microfibers are employed as precursors for the synthesis of nanodiamonds via Hot Filament Chemical Vapor Deposition (HFCVD). PP was selected due to its chemical composition, consisting exclusively of carbon and hydrogen, thereby yielding gaseous precursors like those used in conventional HFCVD processes. To adapt the method for solid precursors, the PP microfibers were placed on the substrate surface before its insertion into the HFCVD chamber. The experimental protocol, optimized to 60 minutes of synthesis, was improved through the application of pretreatment procedures, substrate scratching and etching, to enhance nucleation efficiency and deposition quality. Characterization of the synthesized materials was carried out using scanning electron microscopy (SEM), X-ray diffraction (XRD), and Raman spectroscopy. The findings revealed a heterogeneous carbonaceous deposit composed of amorphous sp²/sp³ carbon structures and nanocrystalline diamond. Furthermore, SEM and Raman images revealed persistent fibrous structures on the substrate, suggesting a nucleation mechanism where, PP fibers partially carbonize and generate methane (CH) due to thermal treatment, to subsequently react with hydrogen and leading to the formation of crystalline nanostructures both on both the carbonized regions and the surrounding surface. The preliminary findings reported in this study demonstrate a promising pathway for the valorization of microplastic waste through the synthesis of advanced carbon nanomaterial by means of CVD-based techniques.

Keywords: microplastics, nanodiamonds, waste conversion, HFCVD

^{*}Speaker

New theoretical method to design quasi-atomic systems in the band gap of semiconductors by combining density functional theory and the Hubbard effective Hamiltonian: Applications to alpha boron and to the nitrogen-vacancy center in diamond

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Abstract

Crystal point defects offer one possible pathway towards the solid-state implementation of some quantum applications. Conditions for such applications are the existence of a **quasi-atomic system** (QAS) in the forbidden band gap, defined as a set of localized in-gap energy levels with the following criteria: (i) the presence of energy levels well localized in the band gap; (ii) some of which are degenerate; and (iii) the capability of occupying the in-gap levels with a number of electrons proper to generate a high-spin state (e.g., a triplet ground state as for the NV- center). Important additional criteria are the possibility to manipulate the spin state by optical excitations and the possibility to control spin selectivity via shelving states.

In the present work, we combine the calculation of total energy in DFT-HSE06 with constrained occupations of the in-gap energy levels, with an in-house Hubbard model fit on the total energy values to describe the many-body energy states of the negatively charged nitrogen-vacancy (NV) center in diamond. We show the need to extend the Hubbard model beyong usual interactions for the NV center.

We then propose a new theoretical methodology aimed to design QASs similar to the NV center. We introduce the four concepts of primary defect; under-hybridized interstitial impurity; multiple combinations of n primary defects referred to as n-wise combination; and thermodynamic charging. The effectiveness of the methodology is demonstrated by an application to carbon-based defects in alpha (α) rhombohedral (trigonal) boron.

Calculations have been performed with the Quantum ESPRESSO software and access to

^{*}Speaker

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 $\mathbf{Keywords:}\,$ Modelling, Density functional theory, The Hubbard model, Anologues to the NV center in boron

Revealing the Factors of Broadening Potential Windows of Diamond Electrodes by Redox-active Additives

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Abstract

Potential window is a significant assessment element for an electrode. A wider potential window can promote the energy density of electronic device and also more detections in aspect of electroanalysis 1-3. The introduction of redox-active additives has been considered to be an effective approach to expand the potential windows of different electrodes2. However, it remains obscure about the influence of redox couples to two sides of water electrolysis respectively. In this presentation, a highly boron doped diamond electrode and two representative outer/inner sphere redox couples are introduced to analyze the key factors associated with water electrolysis. The cation/anion rearrangement under oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) after the addition of redox couple is found to play a pivotal role to water electrolysis, where both have a prominent influence to the charge distribution and the electric field on the interface. Additionally, our experiments together with density-functional theory (DFT) calculations show that the outer/inner sphere redox couples have various influences on the destabilization of the intermediate species during the HER/OER process. The induced surface reconstruction affects the interactions of the active sites and the electrolytes, resulting in different reaction pathways of both OER and HER. This work provides novel insights into the mechanisms to realize the wider potential windows of diamond electrodes with aid of redox-active additives.

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Studying optically-induced magnetic fields by NV scanning microscopy under optical excitation

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Abstract

The inverse Faraday effect is a magneto-optical phenomenon by which a circularly polarized light can magnetize matter. In particular, in metals, the conduction electrons are put into circular motion via the non-linear forces that light applies on them (Hertel, Journal of Magnetism and Magnetic Materials, 2006). These induced currents in metals open the possibility of generating stationary magnetic fields via optical excitation only. The magnitude and direction of these optically induced magnetic fields depend on the optical electric field amplitude in the material, the field gradients, and the polarization of the light.

This is why plasmonic nanoantennas appear as an interesting playground for enhancing and manipulating magnetic fields below the diffraction limit, potentially at ultra-fast timescales, which is interesting for potential applications in data storage technologies and fundamental research in magnetism.

In our group, we recently described the theory underlying the generation of these drift currents in metals, particularly its application to plasmonic nanostructures, using numerical simulations. We demonstrated that a gold photonic nano-antenna, optimized by FDTD calculations, allows, under high excitation power, to maximize the drift currents and generate stationary magnetic fields in the tesla range and at the nanoscale (Yang et al. ACS nano, 2022). However, very few experimental reports exist to compare to this theory, and a thorough characterization of the optically induced magnetic fields has yet to be conducted. NV sensing is a quantitative magnetometry technique based on the unique quantum properties of NV centers in diamond. Our custom-made experimental setup is built to couple this near-field sensing to an optical excitation of the sample. We hope that this high sensibility technique will allow us to quantify and characterize the spatial confinement of the magnetic fields generated via inverse Faraday effect in plasmonic nanostructures.

Keywords:	NV sensing	, magnetometry,	inverse Fa	raday effect,	plasmonics,	nanophotonics,	nanoan-
tenna, magneto,	optics, optic	cal excitation					

*Speaker		

Surface Functionalization of Nanodiamonds with NV Centers as a Platform for Bioimaging and Sensing

Bénédicte Malemo*¹, Anne Vallée¹, Alexis Loiseau¹, Jean-Charles Arnault², Hugues Girard², and Souhir Boujday¹

Abstract

Nanodiamonds (NDs) with nitrogen-vacancy (NV) centers are promising probes for bioimaging and quantum sensing (1), but their practical use relies critically on surface functionalization. Only through controlled surface chemistry can NDs be integrated into hybrid architectures and tailored to exhibit new properties (2).

We investigate two complementary surface chemistry strategies to endow these NDs with additional functionalities. In the first, NDs are modified with cysteamine via EDC/NHS coupling, introducing thiol groups that act as versatile reactive anchors for subsequent chemical modifications or nanoparticle attachment. In the second approach, NDs are encapsulated within a tunable silica spacer layer (3) using a sol-gel method (Figure 1). This shell provides a chemically versatile interface while enabling precise control over ND-environment spacing, a parameter essential for distance-sensitive applications.

These strategies establish robust routes for tailoring ND surfaces and highlight surface engineering as the key enabler for exploiting the unique properties of NV centers in advanced bioimaging and sensing applications.

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Keywords: surface chemistry, biomaging, sensing

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^{*}Speaker

Ultrathin Fluorescent Nanodiamond Films for Quantum Sensing in Semiconductor Devices

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Abstract

Nanoscale quantum sensing plays an increasingly critical role in the rapidly evolving field of semiconductor nanoelectronics. In this study, we demonstrate the use of ultrathin fluorescent nanodiamond (FND) films as quantum sensors for measuring temperature and magnetic field in semiconductor devices. FNDs containing nitrogen-vacancy (NV) centers are renowned for their exceptional photostability and special quantum properties.

We developed an electrospray deposition method to produce uniform, near-monolayer FND films on bipolar junction transistors (BJTs) without compromising their performance. Applying the Optically Detected Magnetic Resonance (ODMR) technique, we measured the temperature and magnetic field of the FND-coated semiconductor device in operando. At a power load of 1.065 W on the FND-coated BJT, we observed a significant frequency shift of the ODMR peaks from 2870.1 MHz to 2860.8 MHz, which corresponds to the temperature increase of 94 ± 2 K at the emitter region. The magnetic field strength of the nearby region was estimated to be 4.0 ± 0.1 G.

These results demonstrate that uniform FND films provide reliable temperature and magnetic field sensing in semiconductor devices.

Keywords: bipolar juncion transistros, field effect transistors, nitrogen vacancy centers, optically detected magnetic resonace, quantum defects

Speaker		

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High Spectral Resolution Resonant Photoluminescence Excitation Spectroscopy of two Native Self-Interstitial Complexes in Electron-Irradiated Diamond

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Low dose (near-threshold) electron-irradiation of ultra-pure diamond (type IIa) produces intrinsic radiation damages such as vacancies, self-interstitials and their aggregates. Subsequent low temperature photoluminescence spectra consist of many lines of defect centers whose relative intensities compete upon the wavelength of the excitation incident light. Unfortunately, in the 500-650nm interval, their spectral contributions overlap so that it is difficult to be certain that a given spectral structure (i.e ZPL, vibronic replica, or localized vibrational mode) is associated to a defect or to another.

In these conditions and for all these reasons, resonant photoluminescence excitation spectroscopy (R-PLE) offers several advantages. First, by exciting resonantly a ZPL allows isolating spectrally a system and therefore ensures that the resulting measured emission features belong to the sole system under study. This considerably reduce/suppress uncertainties and limitations met in previous studies aimed to identify and characterize interstitial complexes. In addition, by sweeping the frequency of the tunable laser line across the PL transitions while integrating the red-shifted PL emission into the Phonon Side band (PSB) gives access to a measurement of the absorption spectrum of the ZPL of the defect at very high spectral resolution, the latter being limited by the spectral resolution of the tunable laser lines.

In this work and for the first time, R-PLE spectroscopy were applied to interstitial-related centers. The experiment were performed at 2.7K using a Spectra Physics 380 dye ring laser pumped by a Verdi V10 laser from a coherent cavity double diode-pumped CW Nd:YaG laser. A resolution close to or better than 0.1pm (O.1 GHz) was achieved. Two different interstitial-related centers with ZPLs at 580 nm and 591.3 nm were investigated.

As an illustration, Fig.1a displays the high-resolution absorption spectrum of the 580nm ZPL as measured at 3K on an ensemble of defect centers in an ultra-pure and high structural quality IIa diamond sample. It reveals a strong main peak with a remarkably narrow linewidth of 7 GHz, together with two fainter components blue-shifted by 30 and 96 GHz respectively. Fig.1b displays the electron-phonon coupling spectrum of this optical center measured in luminescence in the 605-655 nm wavelength range. It shows well-defined interactions with preferential lattice vibrations as well as a large number of localized vibration modes. This points towards a rather complex interstitial containing several carbon atoms. In particular, it exhibits a very high-energy local vibration mode at 237 meV that we tentatively attribute to the stretching of a double >C=C=C< bond. Noteworthy, the observation of this 237meV is shared with the 5RL defect center, another self-interstitial.

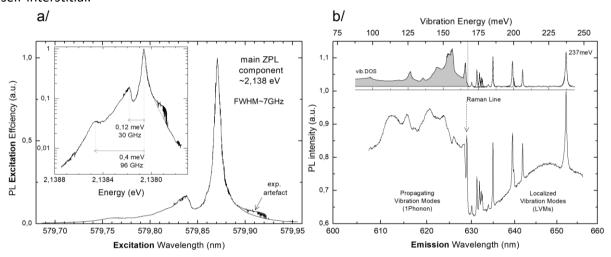


Figure 1: R-PLE spectroscopy of the 580 nm defect centre: a/ The high spectral resolution spectrum of the 580 nm ZPL in absorption measured at 2.7K in resonant excitation when integrating the PL emission in the Phonon Side Band from 605 nm to 655 nm as plotted in /b. **Inset:** same plot as function of the excitation energy and on a logarithmic Y-scale.

Localized Intracellular Thermometry Using NV Centers in Nanodiamonds with Gold Nanoparticle heaters:

From Simulation to Experimental

Department of Chemistry, Okayama University¹

Mina Tavakkoli¹, Masazumi Fujiwara¹

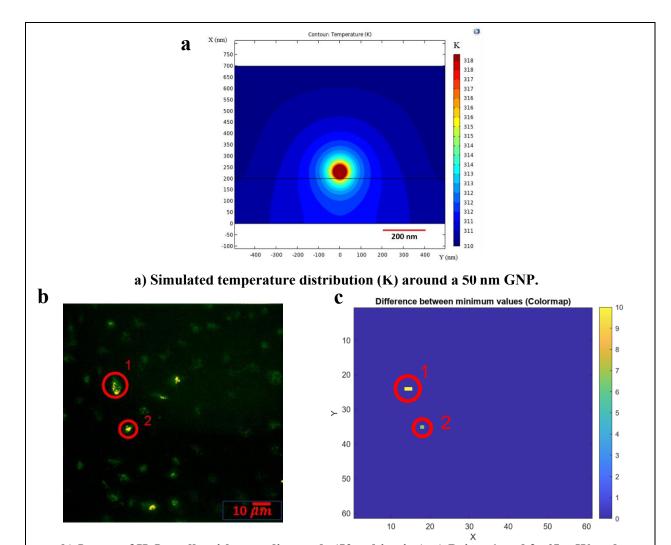
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Real-time intracellular thermometry reveals how local heat affects subcellular structures and guides targeted responses. For example, photothermal heating affects mitochondria, key players in metabolism and signaling [1]. Quantum thermometry with nanodiamonds enables submicron localization of temperature changes in vivo [2]. Intracellular heating alters organelle functions. For instance, nuclear heating promotes neurite outgrowth during neuronal differentiation, showing thermal distribution can act as a signaling mechanism [3]. In this study, to achieve localized heating and nanoscale temperature measurement, we utilized GNPs as nanoheaters and nanodiamonds (FNDs) as thermometers.

To predict nanoscale thermal behavior, we used the finite element method to assess how surrounding media influence heat propagation from GNPs under CW laser, considering intracellular thermal conductivity ($0.11 \pm 0.04~W~m^{-1}~K^{-1}$ in HeLa and MCF-7 cells) reported by polydopamine-coated nanodiamond thermometers [4]. Our simulation showed that in water, GNPs reached 318 K at the particle center and 310 K at a radial distance of 1000 nm from the GNP surface. When the boundary temperature was fixed at 313 K, the outer temperature rose to 314 K, indicating improved thermal confinement. These results highlight that thermal conductivity and boundary settings govern local heating and help define safe power limits.

Based on these insights, we initiated real-time NIR (1456 nm wavelength) heating in HeLa cells (56,000 cells, 12 mm dishes) after overnight incubation with 12 μ L nanodiamonds (1 mg/mL), followed by fixation and mounting on microwave antenna substrates. Thermometry under varying NIR laser powers (19, 26, 32, 52, 65, and 130 mW) was performed using a 50× objective lens. Preliminary results showed controllable local heating from 32–54 °C using laser powers of 19–130 mW, suggesting suitability for probing subcellular thermal responses near organelles.

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b) Image of HeLa cells with nanodiamonds (50× objective). c) Points 1 and 2: 65 mW and 52 mW NIR laser irradiation, respectively.

Etching of Nanodiamond Seeds during Early CVD Growth: Size-Dependent Modeling and Implications for Ultrathin Diamond Film Engineering

Raffaella Salerno^{*1,2}, Alessandro Bellucci¹, Eleonora Bolli¹, Matteo Mastellone¹, Veronica Valentini¹, Daniele Trucchi¹, Massimo Tomellini², and Riccardo Polini²

Abstract

Detonation nanodiamonds (DNDs) serve as indispensable seeding agents in the fabrication of ultrathin (≤ 100 nm) nanocrystalline diamond films. However, their stability under typical microwave chemical vapor deposition (MWCVD) plasma environments remains a limiting factor in achieving continuous ultrathin diamond coatings (1).

This study (2) offers a kinetic investigation and theoretical modeling of the etching of DND seeds during the initial stages of MWCVD diamond growth. Employing SEM to monitor surface seed density over time under controlled plasma conditions, we experimentally quantify the progressive disappearance of DND particles on Si (100) substrates. The phenomenon is analyzed through a size-dependent etching model, based on the Young-Laplace equation, taking into account the increase of the chemical potential of carbon atoms in DND particles and the consequent reduction of the activation energy for the reaction with atomic hydrogen. The model explains the experimental seed disappearance kinetics by proposing that nanodiamond particles smaller than a "critical radius", r*, are etched away, whereas those larger than r* can survive and grow.

Building on this understanding, the size-dependent stability of DND seeds has been harnessed to control the growth of nanocrystalline diamond films with thicknesses \geq 40 nm on Si (100) substrates, tailored for application as cathodes in photon-enhanced thermionic emission (PETE) devices (3). Thinner films offer superior electron emission performance due to the lower resistance of the diamond film compared to thicker samples. The influence of grain boundaries on cathode performance was further investigated with Raman spectroscopy and Kelvin Probe Force Microscopy measurements indicating that an 80 nm-thick diamond film yields the highest emission current density, attributed to its favorable grain boundary distribution.

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^{*}Speaker

 ${\bf Keywords:} \ {\bf detonation} \ {\bf nanodiamond} \ {\bf seed} \ {\bf etching}$

NANO-DIAMOND FILMS FORMATION FROM ENERGETIC SPECIES

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Abstract

Nano-diamond (ND) films are deposited by energetic using the direct-current glow-discharge CVD from a methane/hydrogen mixture. The growth occurs on top of a preferentially oriented graphitic precursor with basal planes perpendicular to the surface. These films consist of an agglomerate of diamond particles (size 3-5 nm) with amorphous grain boundaries. The hydrogen concentration in the graphitic precursor is only a few percent, however it increases to _~15-20 at.% in the film.

ND films were explored by complementary methods. The hydrogen content and its role in ND film formation were assessed. The experimental methods used comprise NEXAFS to prove the short range coordination of the carbon films and indirectly their phase composition. The surface and grain boundary phase composition were investigated by a combination of EELS measured as a function of incident electron energy and hydrogen etching experiments. By TEM the micro-structural evolution and their visualization were achieved. The density evolution of the films was determined by XRR. The hydrogen content and its distribution in the films was studied by SIMS and ERD. The hydrogen bonding was investigated by HREELS. Most likely hydrogen is bonded within the amorphous grain boundaries and saturates the ND particles. The surface of the films is amorphous in nature.

ND film and growth from energetic species is explained as a sub-surface process in terms of a four step cyclic process:

- (1) Formation of a dense, hydrogenated sp2 carbon coordinated oriented layer.
- (2) Precipitation of sp3 C clusters in this graphitic phase.
- (3) Growth of ND particles up to _~5 nm in size by energetic species bombardment of the diamond / hydrogenated carbon interface. It involves preferential displacement of sp2 carbon coordinated carbon atoms leaving sp3 coordinated carbon atoms intact, leading to expansion of the diamond phase.
- (4) Frustration of growth of diamond particles.
- (5) Growth of the film formed of an agglomerate of ND particles by a cyclic process which involves process 2-4.

Keywords: nanodiamond film, microstructure

^{*}Speaker

Bottom-up production of nanodiamonds using MW microplasma

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Abstract

In this study, the formation and properties of carbon nanostructures obtained from a low-pressure-low temperature microplasma operated with $\rm Ar/H2/CH4$ gas precursors were investigated. The study reveals gas phase nucleation of nanodiamonds among other phases of carbon such as graphite and amorphous carbon. The use of plasma nucleated nanodiamonds looks promising in terms of simplicity

and cost-effectiveness, and works towards a bottom-up approach of producing high-quality nanodiamonds.

Raman spectroscopy revealed that the signature of the nanostructures produced was insensitive to the nature of the substrate. The main conclusion of the study is that nucleating of nanodiamonds is sustained by high densities of H-atoms at moderate gas temperatures and optimum concentrations of hydrocarbon radicals and molecules. High densities of hydrocarbons push the equilibrium towards sp2 and amorphous nanostructures even under high H-atom densities. The conditions where maximum nanodiamonds are found are also the conditions where minimum of amorphous sp2 structures including transpolyacetylene and soot. The chemistry of hydrocarbons are complex and the resulting nucleation pathways are dominated by the abundance of dominant carbon radicals that are formed in the plasma. From OES of the C2 and CH radicals and correlating with the results from Raman analysis indicates that C2 and CH may have an important role in the nucleation of nanodiamonds. Possibility of doping the nanodiamonds with N was tested by adding small amounts of N2O. We will discuss the applications of these nanodiamonds for growing NCD films as well as larger nanodiamonds in an conventional MW reactor used for CVD growth.

Keywords: nanodiamonds, Bottom, up synthesis, Plasma

^{*}Speaker

IMPROVED GAS PHASE NUCLEATION OF NANODIAMONDS WITH ARGON ADDITION

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Abstract

Nanodiamond was synthesized using Ar/H2/CH4 microwave microplasma torch. Microwave power, pressure, H2/CH4 ratio were fixed by varying Ar concentration. Plasma was characterized using optical emission spectroscopy (OES) and pico-second two absorption laser induced fluorescence (ps-TALIF)(1) for parameters such as Tg around 1500 K and nH(1) in range of 1016 - 1017 cm-3. Nanodiamond of highest sp3 fraction were nucleated at 100 mbar pressure with 90 Watt injected MW power, and gas composition of 96 sccm H2, 4 sccm CH4 and 20 sccm Ar. A direct correlation between intensity of C2 Swann band emission to sp3 fraction (Fig 1) was observed, indicating the potential role of C2 dimer in ND nucleation. The importance of C2 in ND nucleation has been observed by other researchers as well (1). The nH is increased with increasing Ar in the plasma. It is known that, nH have strong influence in sp3 fraction because it is associated with hydrogen abstraction reaction resulting in formation of CH3. radicals(1). Even though we advocate the role of C2 dimer as a building block in ND nucleation phenomena, it is obvious that other sp3 nucleation routes through CH3. due to high nH along with other sp2 growth pathways may also co-exist.

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Kevwords:	Nucleation.	Piasma	cnemistry	. Manodiamond.	moiecular	growth.	Piasma	diagnostics

^{*}Speaker

Etching of Nanodiamond Seeds during Early CVD Growth: Size-Dependent Modeling and Implications for Ultrathin Diamond Film Engineering

Raffaella Salerno^{*1,2}, Alessandro Bellucci¹, Eleonora Bolli¹, Matteo Mastellone¹, Veronica Valentini¹, Daniele Trucchi¹, Massimo Tomellini², and Riccardo Polini²

Abstract

Detonation nanodiamonds (DNDs) serve as indispensable seeding agents in the fabrication of ultrathin (≤ 100 nm) nanocrystalline diamond films. However, their stability under typical microwave chemical vapor deposition (MWCVD) plasma environments remains a limiting factor in achieving continuous ultrathin diamond coatings (1).

This study (2) offers a kinetic investigation and theoretical modeling of the etching of DND seeds during the initial stages of MWCVD diamond growth. Employing SEM to monitor surface seed density over time under controlled plasma conditions, we experimentally quantify the progressive disappearance of DND particles on Si (100) substrates. The phenomenon is analyzed through a size-dependent etching model, based on the Young-Laplace equation, taking into account the increase of the chemical potential of carbon atoms in DND particles and the consequent reduction of the activation energy for the reaction with atomic hydrogen. The model explains the experimental seed disappearance kinetics by proposing that nanodiamond particles smaller than a "critical radius", r*, are etched away, whereas those larger than r* can survive and grow.

Building on this understanding, the size-dependent stability of DND seeds has been harnessed to control the growth of nanocrystalline diamond films with thicknesses \geq 40 nm on Si (100) substrates, tailored for application as cathodes in photon-enhanced thermionic emission (PETE) devices (3). Thinner films offer superior electron emission performance due to the lower resistance of the diamond film compared to thicker samples. The influence of grain boundaries on cathode performance was further investigated with Raman spectroscopy and Kelvin Probe Force Microscopy measurements indicating that an 80 nm-thick diamond film yields the highest emission current density, attributed to its favorable grain boundary distribution.

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- (2) R. Salerno et al., ACS Omega 2023, 8, 25496.
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 ${\bf Keywords:} \ {\bf detonation} \ {\bf nanodiamond} \ {\bf seed} \ {\bf etching}$

Wednesday, October 15th Sessions 7 & 8

12C enriched Fluorescent Nanodiamonds for biomedical quantum sensing applications

Masazumi Fujiwara*1

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Abstract

Fluorescent nanodiamonds (FNDs) containing nitrogen-vacancy (NV) centers are emerging as robust quantum sensors for biological applications, enabling nanoscale detection of temperature and magnetic fields with high biocompatibility and photostability. I will talk about our recent progress in developing quantum-grade FNDs through isotopic (12C) enrichment and reduction of substitutional nitrogen impurities. The resulting FNDs, with NV concentrations of 0.6–1.3 ppm, exhibit greatly improved spin properties-achieving T up to 1.6 ms and T up to 5.4 μ s-while maintaining bright fluorescence (1). These enhancements allow ODMR detection with significantly reduced microwave power and enable thermal echo temperature measurements with a sensitivity of 0.28 K/sqrt(Hz). I will also talk about applications of FND quantum sensors to biological thermometry and on-chip bioassay (2, 3).

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Keywords: NV, quantum sensing

^{*}Speaker

Measuring axonal transport using neurotropic fluorescent nanodiamonds

Baptiste Grimaud*¹, Fletcher Fletcher², Brigitte Potier¹, Régis Daniel³, Arfaan Rampersaud , William Buchmann³, and François Treussart*¹

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Abstract

Defects in axonal transport at the molecular level are observed in neurodegenerative diseases, but their roles in the development of the pathology are unknown. We developed a method to measure axonal transport based on the endocytosis of optically active nanocrystals into neurons, followed by recording of their movement using fast video-microscopy. We first established the approach in primary neuron culture, using fluorescent nanodiamonds (FNDs) (1) and then extended it *in vivo* to the brain of a zebrafish larvae making use in that case of second harmonic generation from nonlinear nanocrystal (2). In both cases, we demonstrated that our method is sensitive enough to detect endolysosomal transport deficits induced by a small change in concentration of transport-related molecules (1,2). However, the method suffers from a low yield of internalization of the nanoparticles.

In order to enhance their endocytosis, we covalently grafted neurotropic peptides (RVG29) onto pegylated FNDs. We characterized the formation of the FND-PEG-RVG29 conjugate by infrared spectrometry and mass spectrometry (MALDI TOF), and evaluated its affinity for its target using a surface plasmon resonance biosensor. Preliminary results confirm the formation of a covalent bond.

While continuing to analyze the FND-PEG-RVG29 conjugates, we have measured the gain in internalization efficiency compared to bare FNDs. In both neuron-like cell line (N2a) and primary hippocampal mouse neuron cultures, the functionalized FNDs exhibited a 2-to 3-fold increase in the fraction of particles having a directed motion compare to bare FNDs, suggesting a same fold increase in their uptake. We are now evaluating if the uptake enhancement remains valid in more complex models, including *in vitro* stem-cell derived human neurons and *in vivo* zebrafish larva. Altogether, this system could enable precise measurements of axonal transport in relevant models of neurodegenerative diseases.

Keywords: fluorescent nanodiamonds, neurotropic peptide, mass spectrometry, axonal transport

^{*}Speaker

Nanodiamond Conjugates for Tracking Disease-Linked RNA-Binding Proteins

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Abstract

Neurodegenerative diseases, such as ALS and FTLD, are characterized by the progressive degeneration of neurons, leading to irreversible motor and cognitive impairments. These disorders remain largely incurable, with complex molecular mechanisms still under investigation. Among the proteins implicated in these diseases, the RNA-binding protein FUS has gained attention due to its propensity to aggregate and its involvement in pathological cellular processes. To better understand FUS behavior over time in living cells, this project explores the use of fluorescent nanodiamonds (fNDs) as permanent, non-toxic, and photostable labels for bioimaging. The objective is to chemically link FUS to fNDs in a way that preserves its biological activity and allows for long-term intracellular tracking. Several strategies for constructing and characterizing the fND–FUS complex were developed, focusing on stability, specificity, and efficient cellular delivery. Preliminary experiments validated the potential of this approach, laying the groundwork for future studies on the dynamic behavior of FUS in neurodegeneration.

Keywords: Neurodegenerative diseases, RNA, binding proteins, Fluorescent nanodiamonds (fND), Chemical conjugation, bioimaging

Speaker		

Fluorescent Nanodiamond-based delivery systems for mitochondrial targeting of bioactive molecules

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 ²Aspects métaboliques et systémiques de l'oncogénèse pour de nouvelles approches thérapeutiques – Institut Gustave Roussy, Université Paris-Saclay, Centre National de la Recherche Scientifique – France
 ³Structure et activité des biomolécules normales et pathologiques – Université d'Évry-Val-d'Essonne, Institut National de la Santé et de la Recherche Médicale, Université Paris-Saclay – France
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Abstract

Metabolic plasticity and mitochondrial function play central roles in tumorigenesis and cancer cells' responses to anti-cancer therapies, making mitochondria a critical target for pharmacological intervention (1–2). If they accumulate in the mitochondria in effective doses, bioactive molecules that modulate mitochondrial activity could offer the prospect of new therapies (3-4). These molecules can be vectorized into mitochondria using nanovectors, to improve their bioavailability and reduce off-target effects (5). In this context, nanometric size delivery systems have gained significant interest due to their ability to enhance bioavailability of therapeutic agents by means of targeting specific cells or subcellular compartments (6).

Our team has focused its attention on the design and application of fluorescent nanodiamonds (FND) as carriers for bioactive molecules. The FNDs have emerged as promising nanocarriers due to their excellent biocompatibility, intrinsic fluorescence, and chemically versatile surfaces, which enable the loading of bioactive molecules and their delivery to subcellular compartments (7).

The aim of this work is to functionalize FND with a mitochondrion-targeting moiety in order to deliver to this organelle therapeutic molecules.

In this study, FNDs were coated with two different PEG-chains carrying different functional groups. The azide moiety was used to conjugate the mitochondrion-targeting peptide SS20 via copper-free click chemistry; the amines groups were used to complex the bioactive peptide N27 via electrostatic interactions (Figure 1). The N27, identified as a promising inhibitor of the AIF/CHCHD4 mitochondrial import pathway, has been chosen for the proof

^{*}Speaker

of concept (8).

This mitochondrion-targeting FNDs have been formulated. Their physicochemical characterisations as well as their cellular and subcellular localisation have been investigated and will be presented.

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Keywords: Nanovectorization, fluorescent nanodiamonds, mitochondria, targeting, bioactive molecules

Nanodiamond Scintillator: A Novel Approach to Profiling EUV and Soft X-ray Beams for Photolithography

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Abstract

Extreme ultraviolet (EUV) radiation with wavelengths of 10–121 nm has been applied in photolithography to fabricate nanoelectronic chips. This study uncovers the practical potential of fluorescent nanodiamonds (FNDs) coatings on optical image sensors for detecting extreme ultraviolet (EUV) and soft X-ray (SXR) radiation. We applied electrospray deposition to produce a uniform carbon-based scintillator thin film on indium tin oxide (ITO) coated substrates, with typical film thickness of $_{-}^{-1}$ μm . Red fluorescence was observed from neutral nitrogen-vacancy centers of the FND film, when exposed to the synchrotron radiation beam (80–1400 eV). The FND fluorescence was further collected by a fiber optic plate (FOP) to a visible light camera for imaging and beam diagnostics. Compared with a f/1 lens-coupled system, the fiber-coupled device was about eight times more sensitive. This research underscores the promising application of FND coatings by electrospray deposition as a cost-effective and versatile strategy for viewing, sensing and imaging EUV/SXR radiation, which can significantly advance the field of next generation nanoelectronics.

Keywords: nanodiamonds, color centers, imaging, sensing

^{*}Speaker

Surface tuning of nanodiamonds: investigating the impact of surface modifications for applications in radiosensitization

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Abstract

Enhancing cancer cell radiosensitivity is essential for improving radiotherapy efficacy without increasing radiation dose. Radiosensitizers, which amplify ionizing radiation effects on tumor cells, offer a promising strategy to achieve this goal. However, identifying agents that combine high effectiveness with biocompatibility remains challenging. Nanodiamonds (ND) represent a viable solution due to their excellent biocompatibility and the possibility of tailoring their surface to enhance reactive oxygen species (ROS) production under irradiation. This effect can be achieved by promoting the formation of hydrogen terminations or decorating the surface with gold nanoparticles. Moreover, functionalization with hyaluronic acid (HA) improves ND dispersibility in aqueous media and enables targeting of tumor cells overexpressing HA receptors, such as pancreatic adenocarcinoma cells.

In this study, we investigated the applicability of ND as radiosensitizers by evaluating their effect on hydroxyl radical (\bullet OH) generation in aqueous solution induced by γ -photons (15 MV) or electrons (10 MeV) delivered with the Elekta SL 18 MV linear accelerator of the University of Torino Physics Department and the National Institute of Nuclear Physics of Torino. To assess the impact of different surface treatments, we compared hydrogenated (NDH), gold-functionalized (Au-ND), and hyaluronan-coated (HA-ND) ND with unmodified ND. Terephthalic acid was employed as fluorogenic probe to detect \bullet OH species, based

^{*}Speaker

on its hydroxylation reaction yielding fluorescent 2-hydroxyterephthalic acid, allowing indirect \bullet OH quantification via fluorescence spectroscopy. Additionally, clonogenic assays on radio-resistant pancreatic cancer cells were performed following co-treatment with ND and radiation to assess possible biological effects.

Our results show that NDH, HA-NDH, and Au-ND increase •OH generation and induce greater reductions in cell survival. These findings suggest a correlation between ROS enhancement and radiosensitization, thus highlighting the potential of ND in therapeutic applications.

Keywords: Nanodiamonds, Radiosensitizers, Surface functionalization, Radicals production, Radioresistant cancer cells treatment

Precision Engineering of Immunocellular States via Intracellular Fever with Nanodiamond Composites

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Abstract

Temperature plays a vital role in cellular function, affecting numerous processes such as enzymatic activity, signal transduction, and immune responses. However, explicitly correlating intracellular temperature with cellular function is challenging, due to the lack of availability of precisely manipulating and monitoring the nanoscale local temperatures. Herein, a photostable photothermal croconium dye (CR) was designed and synthesized to work as a light-controlled intracellular heater, then the CR was conjugated with nanodiamonds containing nitrogen-vacancy (NV) centers, working as nanothermometer, to develop a novel nanodiamond composite (FNDNG-CR-PEG). It enables real-time manipulation and monitoring of intracellular temperatures within macrophages. The relationship between intracellular fever and macrophage polarization was studied using immunofluorescence microscopy and flow cytometry for the first time. Furthermore, the amount of local free radical species induced by local temperature change was monitored by integrating the T1 relaxometry of nanodiamonds. The transcriptomics was further carried out to understand the intracellular fever influence on phenotype at the genome level. This nanodiamond composite provide a powerful platform for exploring the interplay between intracellular thermal regulation, redox signaling, and immune cell behavior. This study not only advances the understanding of intracellular thermal dynamics but also suggests new approaches for enhancing the efficacy of photothermal therapies and modulating immune responses at the nanoscale.

 $\textbf{Keywords:} \ \ \text{Nanodiamond, temperature sensing, temperature manipulation, macrophage polarization}$

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Hybrid Nanozyme Systems based on Gold-Decorated Nanodiamonds for Advanced Electrochemical Sensing and SERS substrate.

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Abstract

Nanozymes, which are nanomaterials exhibiting enzyme-like catalytic properties, have garnered significant interest in the realm of electrochemical sensing due to their remarkable stability, reusability, efficiency under extreme conditions, and cost-effectiveness compared to natural enzymes. Gold nanoparticles (AuNPs) have shown exceptional peroxidase-like activity, making them particularly appealing for sensing applications. However, traditional methods for synthesizing gold nanoparticles come with several drawbacks, including the use of reducing and stabilizing agents that can negatively impact the material's catalytic performance. Therefore, developing innovative synthesis strategies is crucial for enhancing catalytic efficiency and improving sensor performance. In this study, we investigate the potential of gold-decorated nanodiamonds as a novel platform for electrochemical sensors, biosensors, and surface-enhanced Raman spectroscopy (SERS). By decorating nanodiamonds with AuNPs, we create a hybrid system that exhibits enhanced catalytic properties, as demonstrated by UV-vis spectrophotometry, and a significative effect as SERS substrate using near infrared laser source. This AuNP-ND hybrid demonstrates significant peroxidase-like activity while benefiting from increased operational stability. To adapt this system for electrochemical applications, we incorporated the hybrid materials onto the surface of graphite working electrodes within screen-printed electrodes on PET substrates. Electrochemical characterization revealed a strong synergistic effect, resulting in enhanced electron transfer, improved sensitivity, and robust performance under operational conditions. This study positions nanodiamonds as a valuable new support for nanozyme-based electrochemical sensors, paving the way for innovative, enzyme-free detection strategies in both environmental and biomedical contexts.

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Keywords: gold nanoparticles, Nanodiamonds, Nanozymes, electrochemical sensor, SERS

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