

PLASMA PROCESSING UPDATE

Issue 106



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EDITORIAL TEAM

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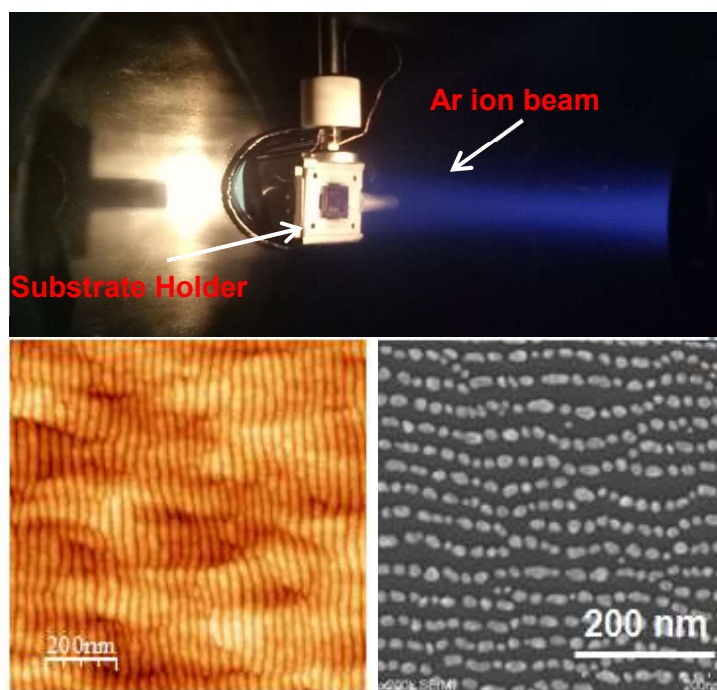
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Nanoparticles arrays grown on Plasma ion beam produced Nanopatterns



Nano material generation system at FCIPT



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Nanoscience and nanotechnology has generated tremendous interest among both researchers as well as technologists due to its potential applications in varied fields as diverse as biomedicine, optoelectronics and metallurgists. Materials when reduced to nanoscale exhibit properties completely different from its bulk counterparts. These properties have applications in many of the day-to-day devices that are used. Industrial houses have already ventured into regular use of a number of the nanomaterials. Various nanomaterials like Zinc oxide, Cadmium sulphide, Iron oxide, Titanium dioxide, aluminium oxide, metallic aluminium, metallic iron etc have great industrial demand for various applications.

Of these two nanomaterials that are in most industrial demand is zinc oxide and titanium dioxide nanoparticles. These are photocatalytic materials at nanosize range. Their applications range from self-cleaning surfaces like ceramic wall tiles, fabric surfaces etc; imparting antibacterial functionality to textile fabrics, whitening pigments in toothpaste, paints etc and as cattle feed additives; it is also used as sun screen components in cosmetic products. The industrial demand in India for these two nanomaterials is in hundreds of kilograms, if not in tonnes, per annum.

The most common method of nanoparticle production is by wet chemical process. Other methods include PECVD, Thermal CVD, hydrothermal process, ball milling, laser ablation etc. However, all these processes have the disadvantage of large time scales and involving a number of intermediate steps with impurities creeping in at various steps, low production rates or varied process for varied materials. All these factors add to the production cost and consequently limits its usage to niche market. Commonly used wet chemical process also involves use of hazardous or environment unfriendly chemicals, use of copious amount of water etc.

Here in Institute for Plasma Research, we have developed a plasma based nanopowder production system which is a quick, versatile and easy to use process without the use of chemicals or water.

Summary of the method and its potential benefits

The indigenously designed system is being used for making various metal oxide nanoparticles like Titanium dioxide, Iron oxide, Cobalt oxide, Zinc oxide etc. Since the industrial demand for Zinc oxide and titanium dioxide are huge, a system has been developed for making these two nanoparticles in large quantities.

Typical size ranges that can be obtained by thermal plasma process would be 25 - 50 nm (with an average size of 30 or 35 nm)

Salient features of plasma based process developed at IPR:

- A one step plasma route of nanomaterial production
- Reduced impurity
- A single integrated system to produce as well as collect the nanomaterials
- Fast time scales and large production rates
- Low operational cost
- PLC controlled Fully automated system

At FCIPT a new plasma based nanopowder system has been designed, fabricated and commissioned that can produce large quantities of metal oxide nanopowders especially zinc oxide and titanium dioxide. A photograph of the system is shown below along with a schematic diagram of the system.

The system consists of central chamber in which plasma is created and raw material – zinc or titanium or any other material whose oxide nanopowder is required – placed inside a crucible which acts as anode. The cathode is typically a refractory material like graphite. When arc is struck, the temperature of the plasma reaches thousands of kelvin depending on the plasma current – which could be in tens of amperes or even more than 100 A. The high heat of plasma instantaneously evaporates the material in the crucible. The vapour as it moves out of the plasma zone, starts quenching (the temperature gradient in a thermal plasma could be 1000s of kelvin per cm). Consequently, nucleation of atomic clusters followed by growth results in powder particles which are, due to sharp quenching, are in the range of few tens of nanometers. These nanoparticles, looking like vapour, are sucked towards four collection towers, by way of a pump. At the collection towers are placed 0.5 micron sintered filter tubes, which effectively blocks the escape of the fine powders into the atmosphere. These deposited powders are dislodged from the filters by a blow back mechanism within the tube filters. The dislodged powder, being agglomerated falls into the collector cup. This a continuous process – evaporation of the raw material, transporting the fine powder vapour into the collection towers followed by filtration and collection. Initial trials have shown encouraging results with respect to the generation of nanopowder as well as its collection. Optimisation of the parameters is currently ongoing for better powder collection.

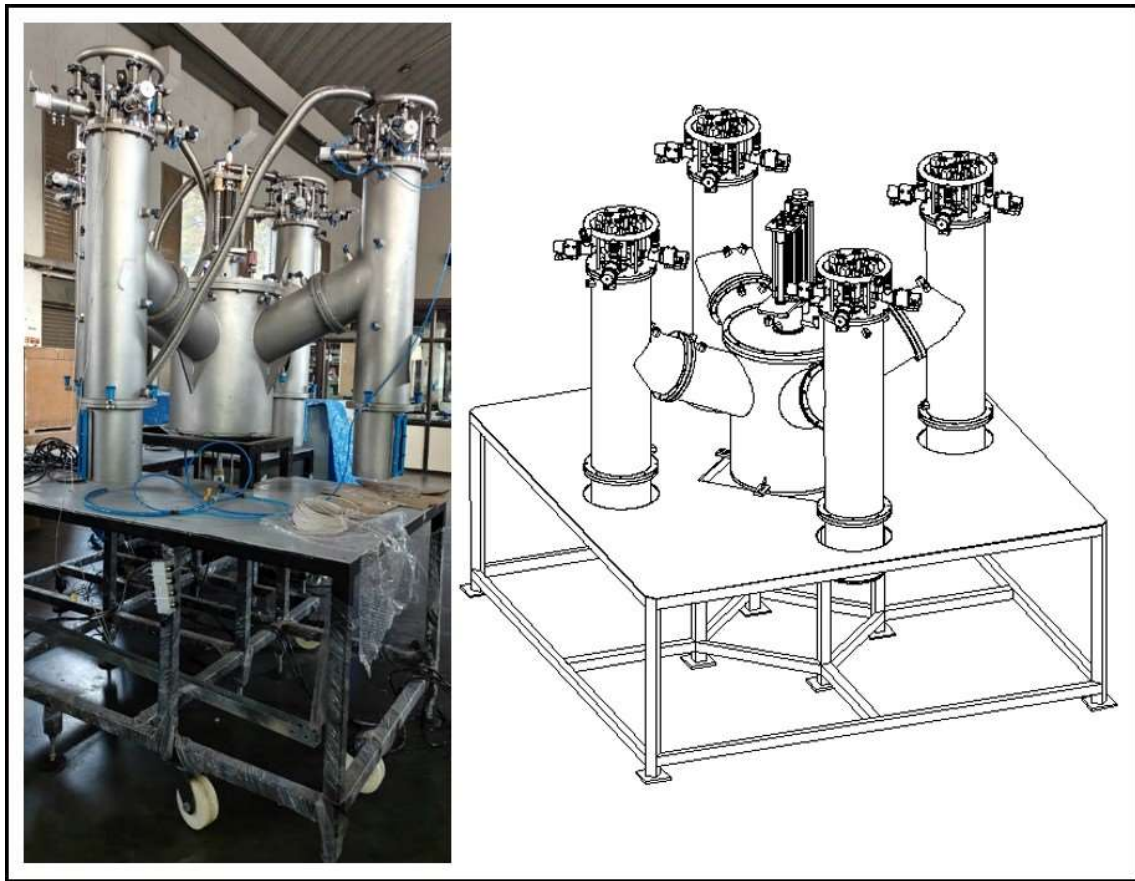


Figure 1: Nano material generation system at FCIPT

Plasma-Activated Oxygen Vacancies in ZnFe_2O_4 Nanoparticles: A Defect-Centric Strategy for Alcohol Detection



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Research Background and Objective

Semiconducting metal-oxide (SMO) gas sensors are attractive for volatile organic compound (VOC) detection because their electrical conductivity changes upon interaction with target vapors. However, their sensing response is often limited by the density and activity of surface adsorption sites. Oxygen vacancies are particularly important because they provide chemically active sites for oxygen chemisorption and influence charge transfer during gas-surface reactions. This work therefore investigates a controlled radio-frequency (RF) plasma approach (Fig.1) to engineer oxygen vacancies on zinc ferrite (ZnFe_2O_4) and establish a direct relationship between plasma-induced surface defects and alcohol-sensing performance. Also the objective is to retain the bulk spinel crystal structure, and to determine how oxygen-vacancy concentration, surface morphology and charge-transduction behavior govern alcohol-vapor sensing.

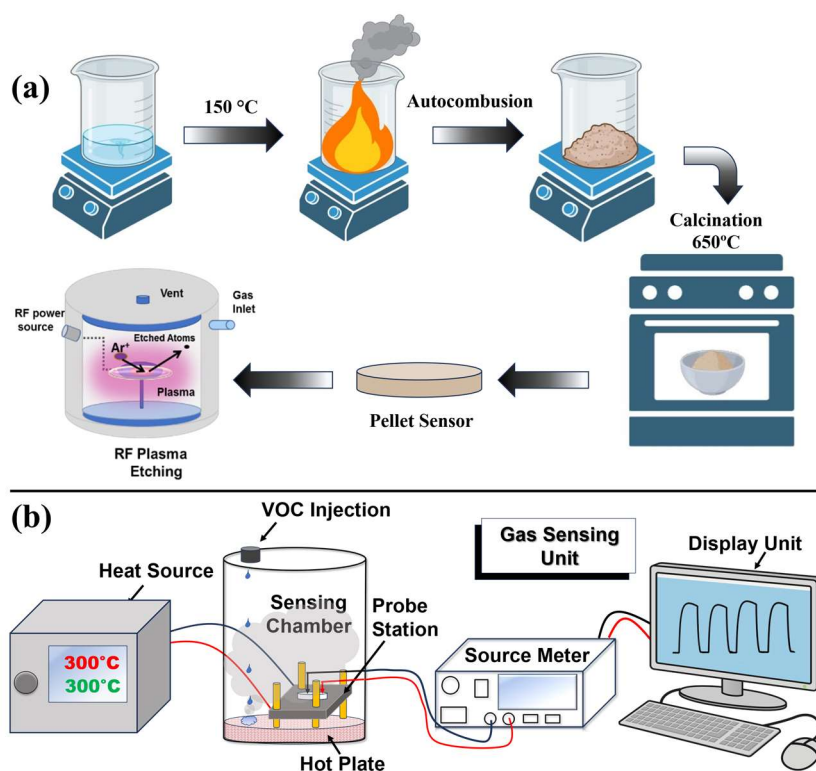


Figure 1: (a) Schematic diagram of (a) ZnFe_2O_4 preparation process and sensor modification, (b) gas sensing unit.

Experimental Approach

ZnFe₂O₄ nanoparticles were synthesized by a wet-chemical auto-combustion route using zinc nitrate, ferric nitrate and citric acid. The precursor gel was heated to induce self-combustion, followed by calcination at 650°C for 4 h to obtain crystalline ZnFe₂O₄. The powder was pressed into approximately 1-mm-thick, 6-mm-diameter pellets and equipped with Ag electrodes for chemiresistive measurements. For surface modification (shown in fig. 1a), Ar plasma was generated at 13.56 MHz and 5×10^{-2} mbar. The RF power was varied from 50 to 150 W and the exposure time from 5 to 20 min. Structural and surface changes were examined by XRD, Raman spectroscopy, SEM/EDS and XPS, while electrical sensing measurements were performed for methanol, ethanol, n-propanol and n-butanol vapors.

Structural, Morphological and Surface-Chemical Modifications

XRD confirmed that pristine and plasma-treated samples retain the face-centered-cubic spinel structure of ZnFe₂O₄ (JCPDS 89-4926) (shown in fig. 2a). At 50 W, the diffraction peaks shift toward higher angles, consistent with lattice contraction/compressive strain associated with plasma-induced defects and possible cation redistribution. At 150 W, the shift toward lower angles is attributed to stronger ion bombardment, defect clustering, structural relaxation and increased disorder. Importantly, the bulk crystal identity is preserved. The calculated crystallite size increased from 40.9 nm for P-0 to 43.0, 43.9 and 47.7 nm for P-50, P-100 and P-150, respectively. Raman spectroscopy shows the characteristic spinel modes (2F_{2g}+ E_g+ 2A_{1g}). Plasma treatment decreases peak intensity and produces broadening and shifts, indicating increased near-surface disorder, oxygen-vacancy formation and cation redistribution (shown in fig. 2b). SEM (shown in fig. 2c) further demonstrates increased surface roughness and porosity after plasma treatment. The average grain size changes from about 202 nm for P-0 to 230, 254 and 264 nm for P-50, P-100 and P-150. The P-50 surface provides a favorable combination of roughness, porosity and grain size for gas adsorption and charge modulation.

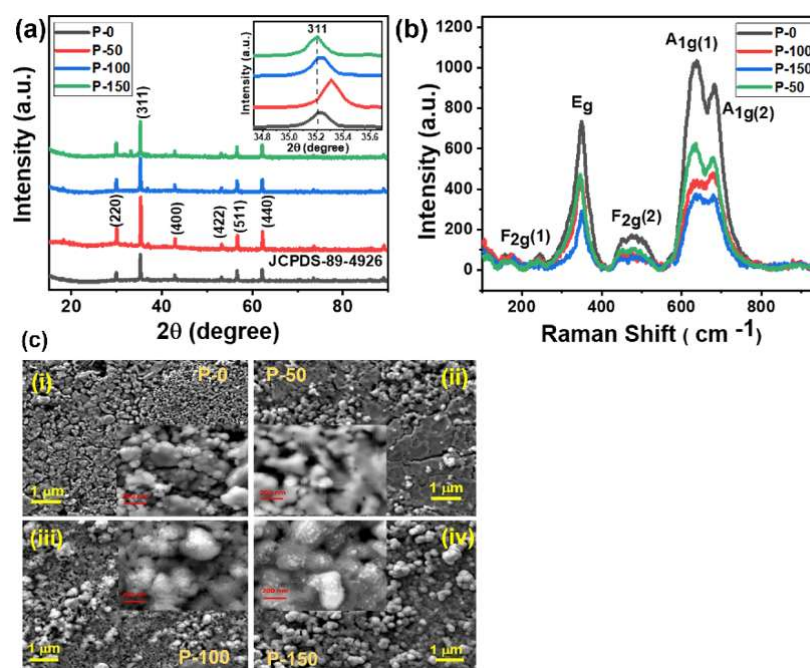


Figure 2: (a) XRD pattern, (b) Rama spectra, and (c)(i-iv) SEM images of pristine and plasma-treated ZnFe₂O₄ sensors.

Oxygen-Vacancy Engineering Revealed by XPS

High-resolution O 1s XPS spectra resolve lattice oxygen, oxygen-vacancy-related oxygen and surface-adsorbed oxygen components (shown in fig. 3). The relative oxygen-vacancy fractions are approximately 18% (P-0), 40% (P-50), 20% (P-100) and 56% (P-150). Thus, the vacancy concentration varies non-monotonically with plasma power. Moderate 50 W treatment substantially increases the population of active defect sites, whereas higher power produces a more complex defect environment involving re-adsorption/reoccupation effects at 100 W and aggressive bombardment with pronounced lattice-oxygen depletion at 150 W. Important scientific finding: the sample with the highest oxygen-vacancy fraction (P-150) is not the best sensor. This demonstrates that sensing performance depends on the balance between oxygen vacancies and lattice oxygen, surface adsorption/reaction, carrier concentration and depletion-layer modulation rather than on vacancy concentration alone.

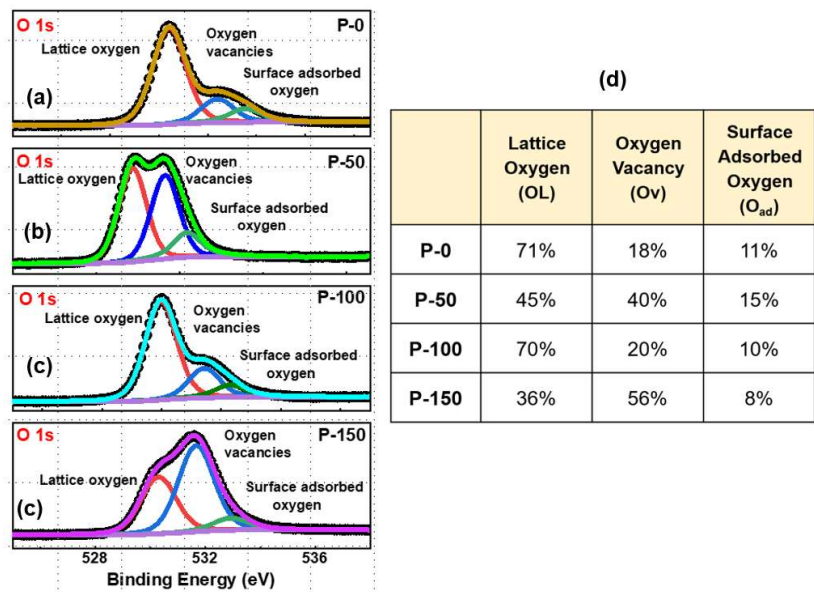


Figure 3: XPS analysis showing plasma-dependent oxygen species and oxygen-vacancy fractions.

Alcohol-Sensing Performance and Mechanism

At 300 °C and 1000 ppm, the sensors show the response sequence n-butanol > n-propanol > ethanol > methanol. The optimized P-50 sensor exhibits the highest response for all four primary alcohols and approximately a five-fold enhancement toward butanol compared with pristine ZnFe₂O₄ (shown in fig. 4a). The stronger butanol response is associated with its vapor density, diffusivity and longer surface residence, which promote stronger interaction with chemisorbed oxygen species. Repeated measurements at different temperatures and measurements after 15 and 30 days demonstrate repeatability, reproducibility and long-term stability (shown in fig. 4b). Cross-sensitivity tests show a substantially stronger response to butanol than to the other tested reducing vapors (shown in fig. 4c).

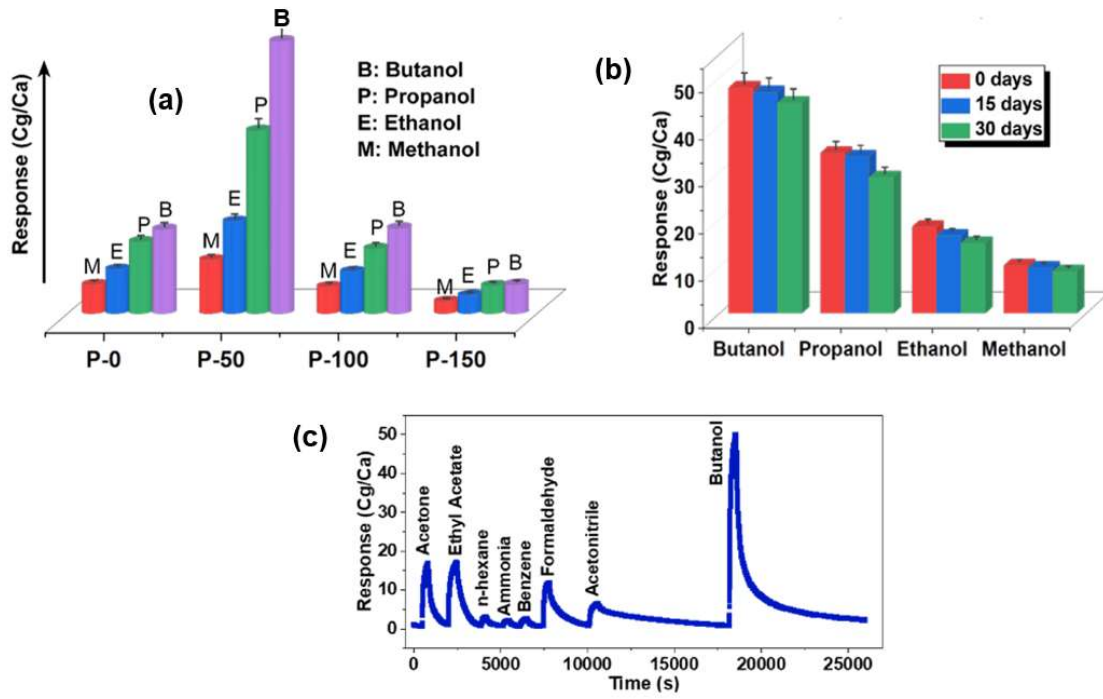


Figure 4: Selectivity, stability/reproducibility, cross-sensitivity towards alcohol vapours for P-50 sensor.

Defect-Centric Sensing Mechanism

During plasma treatment, energetic Ar ions remove lattice oxygen and create oxygen-vacancy-related active sites. When the sensor is heated in air, oxygen molecules are chemisorbed at these sites and extract electrons from the semiconductor surface, increasing the electron-depletion layer and resistance. When a reducing alcohol vapor reaches the surface, it reacts with chemisorbed oxygen species and releases electrons back to the sensing material. This changes the depletion width and associated Schottky-barrier modulation, producing the measurable conductance response. The highest response of P-50 is explained by an optimum receptor–transducer balance: sufficient oxygen vacancies provide active adsorption sites, while adequate lattice oxygen and an appropriate carrier concentration preserve strong modulation of the depletion region and barrier. In P-150, excessive vacancies and defect clustering increase carrier density and can make the depletion region too thin, reducing the relative modulation produced during gas exposure (shown in fig. 5).

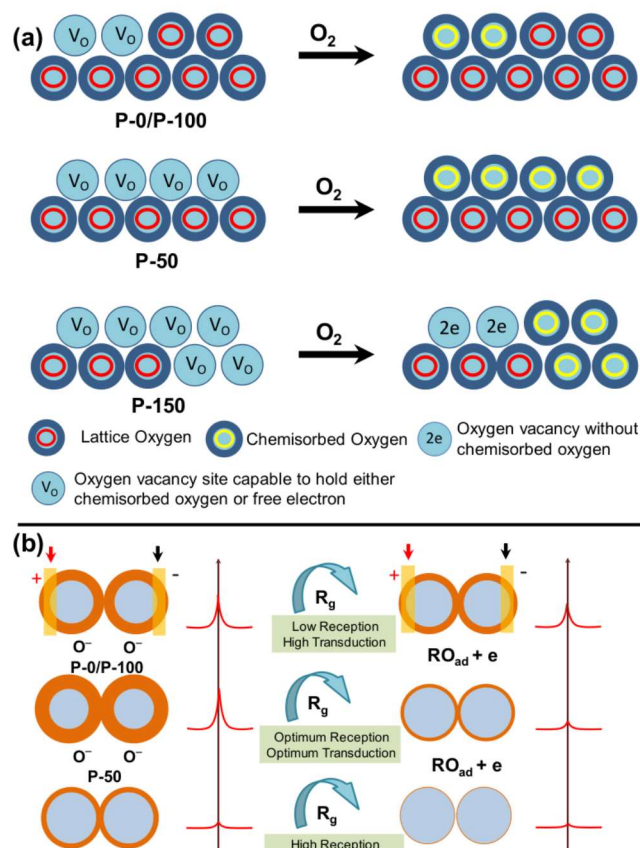


Figure 5: Proposed relationship between oxygen-vacancy concentration, oxygen chemisorption and electronic transduction.

Conclusion

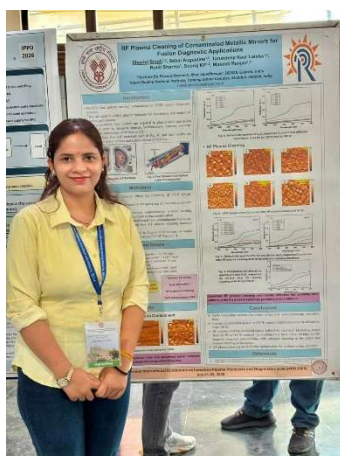
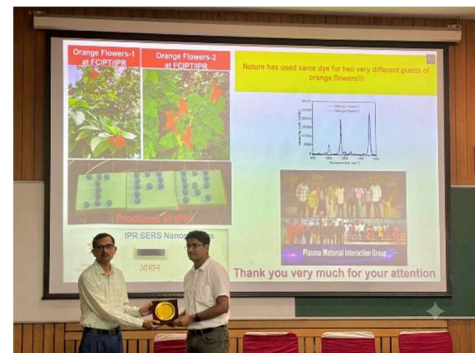
Controlled RF Ar-plasma treatment provides a simple post-synthesis route for engineering surface oxygen vacancies in ZnFe₂O₄. 50 W for 15 min gives the optimum balance of defect chemistry, surface morphology and electronic transduction. The optimized sensor gives approximately five-fold higher butanol response than pristine ZnFe₂O₄. The work establishes that maximum oxygen-vacancy concentration does not necessarily produce maximum sensing response; an optimum lattice-oxygen/oxygen-vacancy balance is essential. The approach provides a useful platform for future plasma-enabled surface engineering of semiconducting oxides for gas/vapor sensing applications.

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Sanju Rani, Snehangshu Paine, Radhe Shyam, Sheetal Singh, Rohit Sharma, Sooraj KP, Kalisadhan mukherjee, **Mukesh Ranjan**
ACS Applied Nano Materials (2026).

Conference Participations

Dr. Mukesh Ranjan gave the Keynote lecture title “Plasma Surface Engineering for Sensing and Wettability application” in The 3rd Global Forum and International Conference on Industrial Plasma Processes and Diagnostics 2025 (IPPD 2025), from July 01-03, 2026 held at IIT Delhi, Delhi, India. In this conference Ms. Sheetal Singh (FCIPT/IPR PhD student) also got the **best poster presentation award** on her poster title “Plasma Cleaning of Contaminated Metallic Mirrors for Fusion Diagnostic Applications”



Ms. Sheetal Singh (IPR PhD Student) attended the Joint ICTP-IAEA Fusion Energy School 2026, Trieste, Italy from 11-22 May 2026. The school covered wide range of topics in plasma and fusion physics from fundamentals like Introduction to Fusion and Confinement, Magneto hydrodynamics, Introduction to Gyrokinetics, fusion and climate change, innovations in materials, highlighting the role of different materials and at various location of fusion devices, How to operate a tokamak with Tritium Breeding, Plasma heating systems through Neutral Beam Heating, understanding and vision of international fusion collaboration, introduced to Stellarators and recent innovations in Superconducting coils. She also presented a poster about her work.

Visit of Students to FCIPT

Visit of GEC Gandhinagar Students

Around 60 students of Gandhinagar Engineering College (GEC), Gandhinagar from Mechanical and Metallurgy Discipline visited the FCIPT/IPR. Their subjected specific discussion and linking with plasma based technologies were discussed by Mr. Akshay Vaid and Mr. Satyaprasad. Dr. Amreen, Mr. Sooraj K P and Ms. Nisha Chandwani Coordinated for their lab visit.



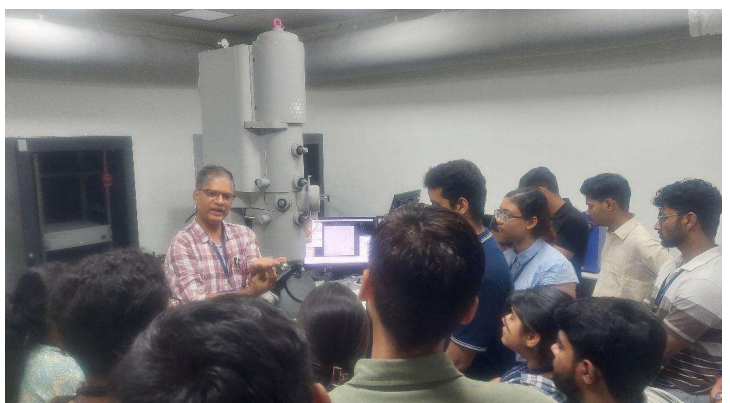
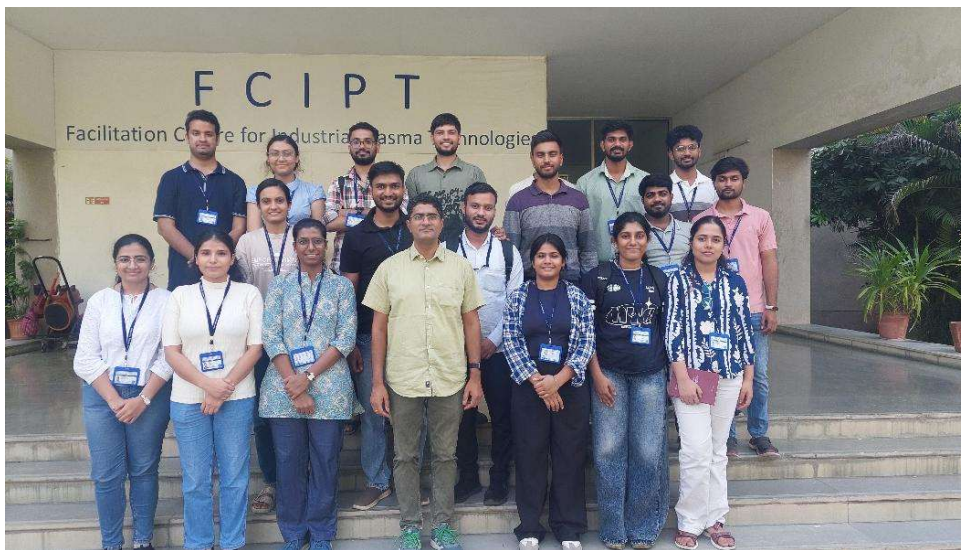
Visit of INYAS Scholars

Young students selected for the INYAS fellowship visited FCIPT. Lectures about basics of plasma and its applications along with their lab visit was arranged.



FCIPT visit of New PhD student of IPR

Newly joined PhD students of IPR visited FCIPT/IPR . A talk about industrial plasma applications was given by Dr. Mukesh Ranjan followed by labs visits of various available material characterisations facilities and Plasma systems. Dr. G.Ravi, Dr. Balasubramanian, Dr. Ramakrishna Rane, Dr. Purvi Dave, Dr. Amreen Hussain, Mr. Satyaprasad, Mr. Sooraj K P, Ms. Nisha Chandwani demonstrated the lab facilities.



Journal Publications & IPR Research /Technical Reports

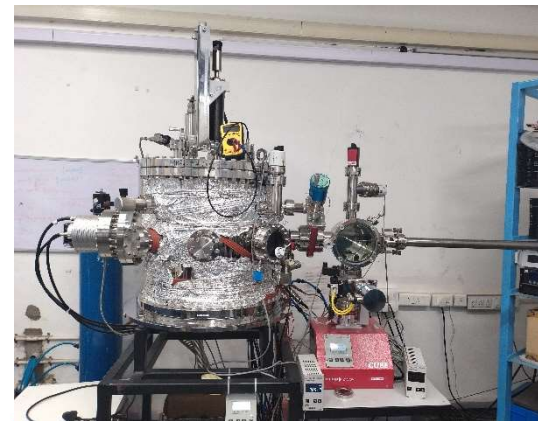
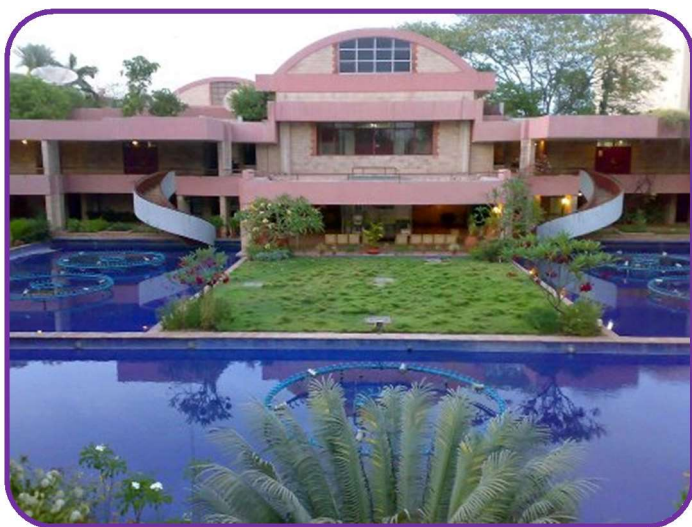
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AIC-PLASMATECH

The Department of Atomic Energy (DAE), through its units and aided institutes, has been transferring and deploying several advanced technologies for the betterment of society. In support of the Government of India's initiatives on Atma Nirbhar Bharat and Start-up India, the DAE proactively launched incubation centres at BARC Mumbai, IGCAR Kalpakkam, RRCAT Indore, IPR Gandhinagar, and VECC Kolkata. This step aims to make the expertise, sophisticated infrastructure, and technologies of DAE available to industries and society at large. Additionally, the DAE initiated the recognition of such incubation centres with the Atal Innovation Mission, NITI Aayog, New Delhi, as 'Atal Incubation Centres'. Based on this initiative, the Institute for Plasma Research established an Atal Incubation Centre as a special purpose vehicle under Section 8 of the Companies Act 2013 in December 2023, with 100% ownership by DAE. The Atal Incubation Centre, popularly known as 'AIC-Plasmatech' and 'Plasmatech Innovation Foundation.

AIC-PLASMATECH OFFER

The Atal Incubation Centre (AIC Plasmatech) offers not only office space to startups but also essential mentoring by IPR's scientists, along with access to cutting-edge technologies and infrastructure. Additionally, it provides business mentoring from experts and external mentors, as well as support in areas such as intellectual property/patenting, marketing, and branding.



**Plasma Ion Beam System
For Nanopatterns Formation**

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