Facilitation Centre for Industrial Plasma Technologies
Institute for Plasma Research

Plasma Processing Update

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Inside this Issue

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Highlights

- **Continuous high power plasma arc generation**
- **Fully automated control of plasma torch operation**
- **Very high electro thermal efficiency (>90%)**
- **No electrode cooling required**
- **Very large plasma plume ensures less erosion of electrodes**

Team members

- **Atik Mistry**
- **Ramesh Bhatia**
- **Chirayu Patil**
- **A. S. Reddy**
- **B. K. Patel**
- **A. Sanghariyat**

Technology Focus :

Successful testing of 100 kW Graphite electrode based plasma torch system for 120 hours

IPR is developing a 5 Tonnes/day plasma-pyrolysis based biomedical waste disposal plant for deployment at the Homi Bhabha Cancer Hospital, Varanasi. This fully-indigenous, environment-friendly plant, making use of three 100 kilowatt graphite-electrode based plasma arcs, is a contribution to Atmanirbhar Bharat. This is the first time that such high-power arc system has been developed in the country for 24x7 operation. In a major milestone, these arcs and associated power supplies have been tested continuously for 120 hours. In this testing, the refractory lining was also tested which could maintain the inner wall refractory temperature at 1100° C, while the outer wall temperature of the chamber remains at less than 80° C. The plasma arc system was operated using voltage-controlled feedback and current-controlled feedback. This plasma arc system does not require water cooling for electrodes and hence it has very high electro-thermal efficiency. Demonstration of long duration operation of the plasma arc system with high electro-thermal efficiency (> 90% observed during this testing) is very important for its application in high capacity plasma pyrolysis/gasification for environment friendly disposal of organic waste.

Figure 1. Plasma arc through viewport

Figure 2. Plasma arc using graphite electrodes

Team members with Dr. S. Chaturvedi (Director, IPR) during his visit to FCIPT

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Highlights

- Plasma fireball for low-cost oxygen ion implanter
- One-step fabrication of high-density non-volatile memory devices
- Dual-use of the single metallic film: Resistive switching layer and electrode
- Rapid fabrication of periodic arrays of squared memory cells

Team members

- Sudheer
- Mukesh Ranjan

Research Focus :

Plasma fireball-mediated ion implantation for nonvolatile memory application

Due to the simple structure (metal/oxide/metal) and extraordinary performance, resistive switching (RS) based-nonvolatile memories are expected to fulfil the demand for next-generation computing technologies [1, 2]. However, to compete with conventional flash memories, quick and low-cost fabrication techniques are essential for the commercialization of RS-based devices [3]. Recently, an Argon plasma-fireball-based low-cost technique has been used to modify nanoscale surface topography to prepare super-hydrophobic surfaces at room temperature [4]. Implementing the plasma-based process using reactive gases (i.e., oxygen) for resistive switching applications is crucial and challenging. Depending upon the polarity, a single device is used to grow Ti thin film followed by plasma fireball-mediated oxygen ion implantation to transform the top surface of Ti film into a functional TiO_x layer to fabricate the device. The current-voltage characteristics of the device were analyzed using conductive atomic force microscopy (cAFM) with a nanosized metallic tip and showed the forming-free bipolar resistive switching and current flipping in a two-fold erase write process [5].

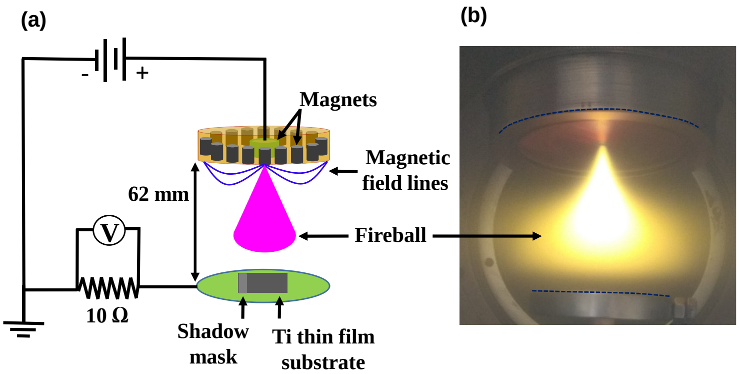

Figure 1. (a) Illustration of plasma-based ion implanter, and (b) a photo of the plasma fireball produced using oxygen as working gas.

In our recent work, published in Applied Surface Science [6], we have shown the fabrication of resistive memories at the nanoscale using plasma fireball technique. Further, the fireball technique is extended to the patterning of periodic arrays of oxide-based squared memory cells of different sizes. Figure 1 (a) demonstrates the schematic illustration of plasma fireball setup. A photo of a balloon-shaped plasma fireball developed using oxygen ions (O^+) at a discharge voltage of 550 V is presented in figure 1 (b).

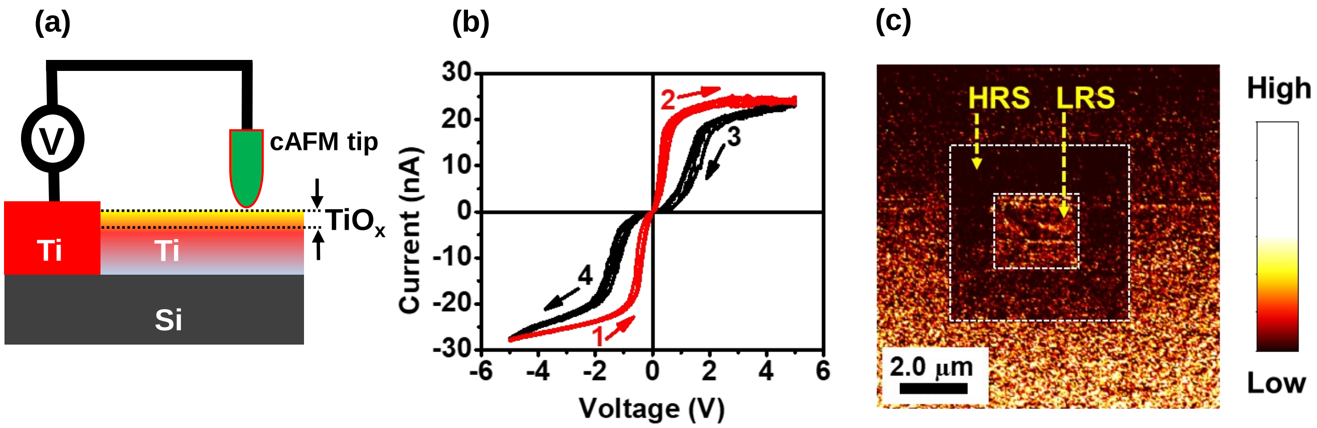

Figure 2. (a) Schematic of the local probe-based testing of the device, (b) current-voltage response curve and (c) demonstration of two-fold erase-write process.

Initially, a titanium (Ti) film is deposited on a silicon (Si) substrate. Subsequently, oxygen ion implantation is performed (fluence: 5×10^{17} ions cm^{-2}). Before implantation, masking of partial surface area on the substrate is done. At the end of the implantation process, the exposed area of the metal film converts into a functional oxide layer, and the masked area remains as it is (which works as a bottom Ti electrode). It is imperative to mention that only the near-surface layer of the Ti film in the exposed area is transformed into the TiO_x layer. The oxygen implanted part consists of three layers (TiO_x/Ti/Si). The underside part of the film in the implanted region, i.e., the Ti layer, is connected to the masked Ti film. Figure 2 (a) shows the testing set up for the prepared device (sample and cAFM connections). In this scheme a bias is applied to the substrate and current response of device is recorded using a nanosized tip, simultaneously. Figure 2b shows a highly symmetric and reproducible hysteresis loop, obtained for cyclic sweeping curve at ± 5 V scanning range which indicates the resistive switching (RS) characteristic in the sample at nanoscale. The intrinsic defects (oxygen vacancies) are mainly responsible for such electrical response of functional oxide layers [1,6]. Figure 2 (c) presents the two-fold flipping of current levels (LRS: low resistive state and HRS: high resistive state) at the nanoscale to confirm the non-volatile memory characteristics of the device. In the process, a nanosized cAFM tip works as a dynamic top electrode and scans the surface, resulting in the flipping of the current state of a particular area depending upon the polarity.

Further, the applicability of the plasma-fireball-based ion implantation technique is demonstrated for the rapid fabrication of periodic arrays of TiO_x-based squared memory cells of different sizes. The Ti-coated Cu -TEM grids of different mesh sizes are used as a mask for patterning periodic memory cells during ion implantation. Figure 3 shows the SEM images of fabricated periodic arrays of different sizes. It is observed that regularly spaced square TiO_x memory cells are formed for all the arrays, and the edges of each memory cell are sharper and well defined. The black portion represents the TiO_x and the lighter area corresponds to Ti film, exhibiting better contrast between the two adjacent memory cells. The size of the memory cells is measured in the range of 20 μm to 40 μm depending upon the number of meshes in the TEM grid. The minimum spacing between two adjacent TiO_x memory cells is noted at about 5 μm (Inset of Figure 3 (b)).

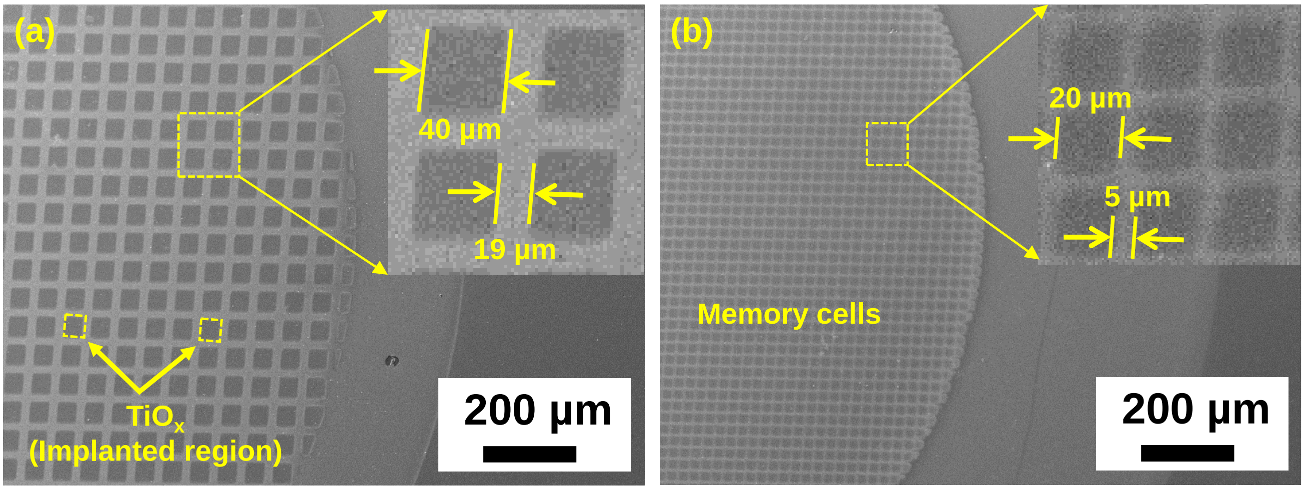

Figure 3. Field emission scanning electron microscopy image of a periodic array of TiO_x-based squared memory cells of different sizes fabricated by plasma fireball using masking method. Memory Cell size: (a) 40 µm and (b) 20 µm.

In the present work, we have shown a one-step fabrication of restive switching surfaces using plasma-mediated ion implantation for nonvolatile memory applications. A simple method is reported to conduct oxygen ion implantation. A balloon-shaped plasma fireball is developed using oxygen gas that is further used as an ion source tool. A sputtered grown single titanium (Ti) layer is used for an electrode as well as to prepare a TiO_x functional layer by oxygen implantation. The fabricated device exhibits forming-free bipolar resistive switching at the nanoscale, which is further confirmed by two-fold erase-write areal measurements using conductive atomic force microscopy. The technique is found to be highly useful for the rapid fabrication of periodic arrays of TiO_x-based squared memory cells of different sizes. This study opens up a new avenue for the quick preparation of nanoscale nonvolatile memory devices using a simple and cheap method.

References

[1] M. Lanza et al., "Recommended Methods to Study Resistive Switching Devices," Advanced Electronic Materials, vol. 5, no. 1, p. 1800143, Sep. 2018, doi: 10.1002/aelm.201800143. Shi T, Wang R, Wu Z, Sun Y, An J, Liu Q. Small Struct. (**2021**);2(4):2000109.

[2] T. Shi, *et al.,* "A Review of Resistive Switching Devices: Performance Improvement, Characterization, and Applications," *Small Structures*, vol. 2, no. 4, p. 2000109, Feb. 2021, doi: 10.1002/sstr.202000109.

[3] D. S. Jeong *et al.*, "Emerging memories: resistive switching mechanisms and current status," *Reports on Progress in Physics*, vol. 75, no. 7, p. 076502, Jun. 2012, doi: 10.1088/0034-4885/75/7/076502.

[4] S. Chauhan, *et al.,* "Plasma fireball: A unique tool to fabricate patterned nanodots," *Review of Scientific Instruments*, vol. 88, no. 6, p. 063507, Jun. 2017, doi: 10.1063/1.4989701.

[5] M. Trapatseli *et al.,* "Conductive atomic force microscopy investigation of switching thresholds in titanium dioxide thin films," *The Journal of Physical Chemistry C*, vol. 119, no. 21, pp. 11958–11964, 2015.

[6] Sudheer et al., "Plasma fireball-mediated ion implantation for nonvolatile memory application," Applied Surface Science, vol. 607, p. 154999, Jan. 2023, doi: 10.1016/j.apsusc.2022.154999.

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Highlights

- Long term antimicrobial activity of PAW
- Seed treatment with PAW enhances germination and plant growth
- PAW treatment enhances shelf life of lemon

Team members

- Divyesh Patel
- Chirayu Patil
- Adam Sanghariyat
- Nimish Sanchaniya
- Shital Bhutani
- Budhi Sagar Tiwari
- Sudhir Kumar Nema

Research Focus :

Antimicrobial, agriculture, and food preservation activities of Plasma Activated Water

The plasma-water interaction changes the physicochemical properties of water due to the formation of various reactive species occurring in water. This reactive water is known as plasma activated water. The formation of various reactive species due to plasma water interaction is shown in figure 1 [1].

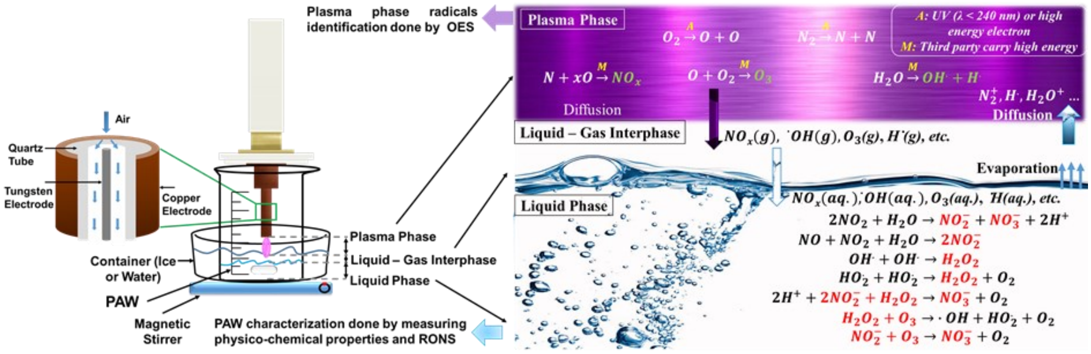


Figure 1. Mechanism of formation of plasma activated water (PAW) [1]

The presence of reactive species enhances the oxidizing tendency of PAW (450 mV to 800 mV) as well as reduces its pH (4.5 to 2.0) [1-6]. PAW's high oxidation potential is due to the presence of oxidizing species H_2O_2, ·OH, ONOO, dissolved O_3, etc. Furthermore, low pH is due to the presence of HNO_2, HNO_3, etc. PAW has the potential to be used in applications like microbial (bacteria, fungi, pest, and virus) inactivation, selective killing of cancer cells, and food (fruits, vegetables, meat products and sea-food, etc.) preservation etc. Moreover, PAW is also a rich source of nitrogen (NO_3^- and NO_2^- ions, etc.) species. Therefore, it has enormous potential to be used as nitrogen fertilizer for agriculture and aquaculture applications.

Antimicrobial applications of Plasma Activated Water

The plasma activated water technology developed at FCIPT, IPR has already shown promising results in the field of microbial inactivation, food preservation, seed germination and plant growth [3-6]. Some of these results are shown in figure 2 in which morphological analysis of bacteria, fungi, and algae cells are illustrated after PAW treatment. The morphological study is carried out using scanning electron microscopy. The treated bacteria, fungi, and algae cells appear damaged and ruptured. This is because PAW has highly oxidizing nature and therefore its treatment oxidizes the outer membrane of cells when it comes in contact with it. This leads to the inactivation of bacteria, fungi, and algae cells after PAW treatment [3-5].

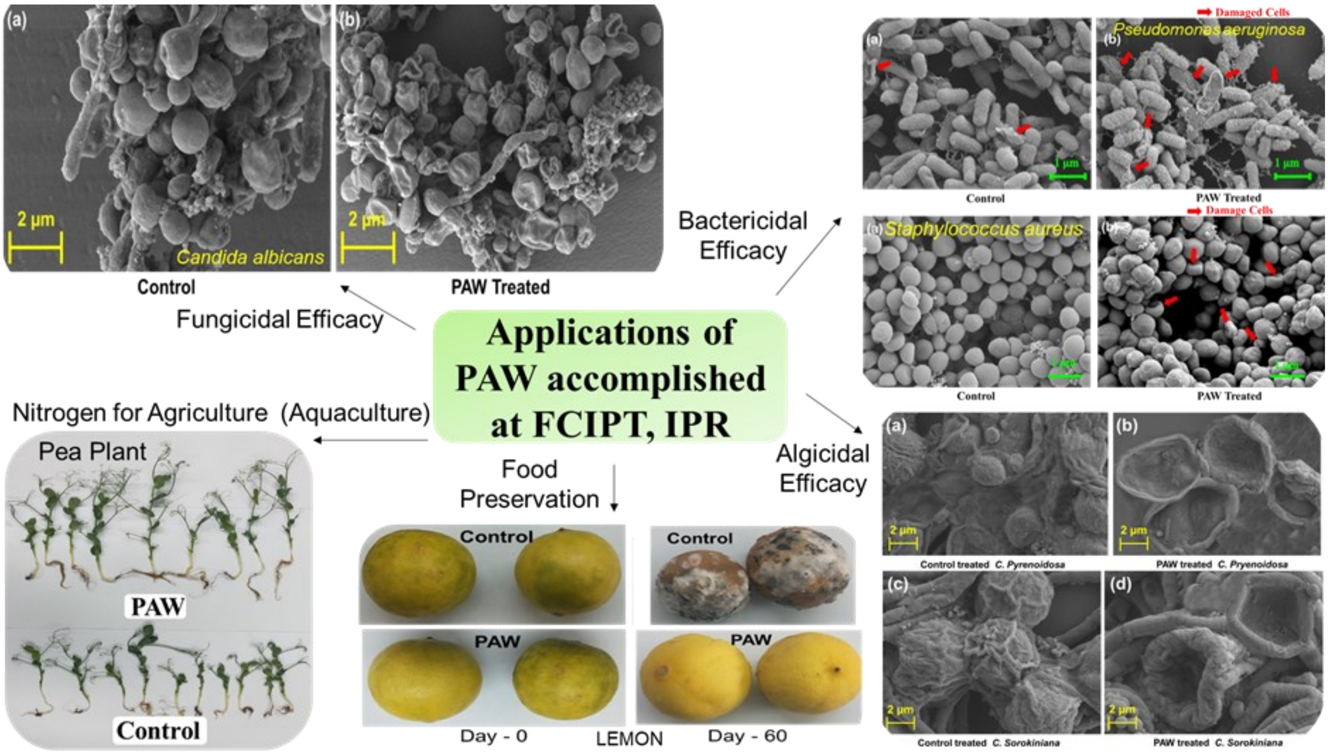


Figure 2. Applications of plasma activated water successfully achieved at Facilitation Centre for Industrial Plasma Technologies (Institute for Plasma Research, IPR) [3-6]

Food preservation using PAW

As shown above, PAW has good antimicrobial activity, and therefore, it can be used for the preservation of food as well as the enhancement of its shelf life. The post-harvest spoilage of food occurs mainly due to bacteria and fungi infections. The study conducted at FCIPT, IPR on citrus limon L. (Lemon) also showed enhancement in the shelf life of lemon after washing with PAW. The reason for enhancement of lemon's shelf-life is due to reduction in microbial load (that causes lemon spoilage over time) after PAW treatment. The colour, texture, weight, and sensory (odour, appearance, taste, and overall acceptability) characteristics of PAW-washed lemon remain intact over time (60 days study). However, control (Ultrapure Milli-Q water) washed lemon showed significant (statistical $p < 0.05$) degradation in mentioned characteristics (figure 2).

Agricultural application of PAW

To study the significance of PAW in the agricultural field, a study was conducted at FCIPT, IPR, in which we explored the effect of PAW on the germination of pea seeds (Pisum sativum L.) and plant growth. The PAW treatment of seeds significantly enhances germination as well as plant growth (picture of plant growth shown in figures 2 and 3) compared to the control sample. This is due to the fact that PAW exposure removes the naturally occurring wax from the seeds' surface that in turn results in enhancement of their wettability properties and rapid absorption of water (figure 3). Hence, higher agronomy traits (height, fresh and dry weight, germination index, etc.), chlorophyll 'a', sugar, and protein, enhanced antioxidant enzyme activity are observed in PAW-grown pea plants compared to the control [6].

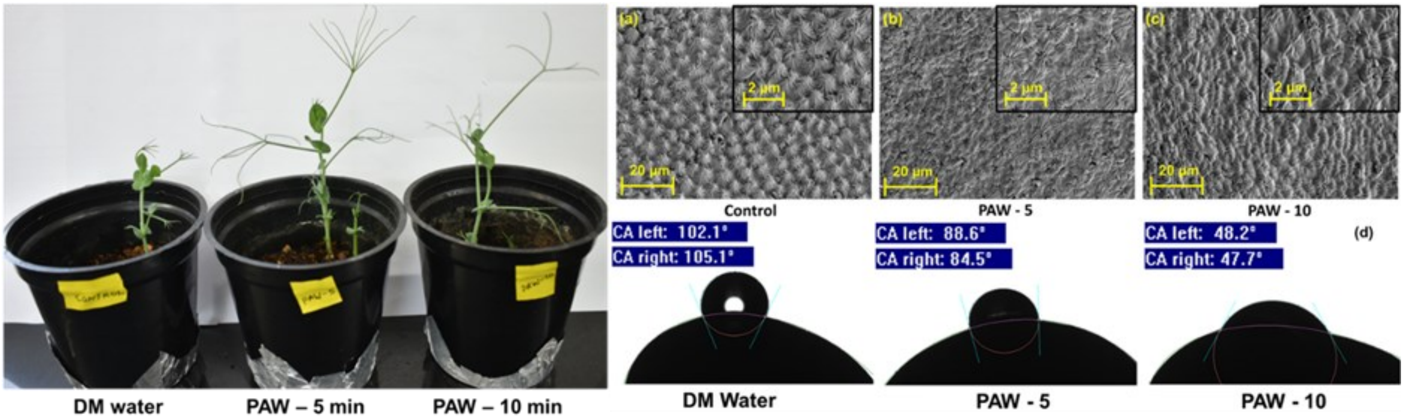


Figure 3. Picture of PAW (PAW – 5 min and PAW - 10) and control (Ultrapure Milli-Q water, DM water) grown pea plant. Change in morphology and wettability of pea seeds surface after PAW treatment [6]

References

[1] V. Rathore and S. K. Nema, "Optimization of process parameters to generate plasma activated water and study of physicochemical properties of plasma activated solutions at optimum condition," Journal of Applied Physics, vol. 129, no. 8, p. 084901, Feb. 2021, doi: 10.1063/5.0033848.

[2] V. Rathore and S. K. Nema, "A comparative study of dielectric barrier discharge plasma device and plasma jet to generate plasma activated water and post-discharge trapping of reactive species," *Physics of Plasmas*, vol. 29, no. 3, p. 033510, Mar. 2022, doi: 10.1063/5.0078823.

[3] V. Rathore, D. Patel, S. Butani, and S. K. Nema, "Investigation of Physicochemical Properties of Plasma Activated Water and its Bactericidal Efficacy," *Plasma Chemistry and Plasma Processing*, vol. 41, no. 3, pp. 871–902, Mar. 2021, doi: 10.1007/s11090-021-10161-y.

[4] V. Rathore *et al.,* "Inactivation of Candida albicans and Lemon (Citrus limon) Spoilage Fungi Using Plasma Activated Water," *Plasma Chemistry and Plasma Processing*, vol. 41, no. 5, pp. 1397–1414, Jun. 2021, doi: 10.1007/s11090-021-10186-3.

[5] V. Rathore V and S.K. Nema, "The role of plasma-activated water on the growth of freshwater algae Chlorella Pyrenoidosa and Chlorella Sorokiniana", Institute for Plasma Research, Research Reports RR-1438, Aug 2022

[6] V. Rathore, *et al.*, "Treatment of Pea Seeds with Plasma Activated Water to Enhance Germination, Plant Growth, and Plant Composition," *Plasma Chemistry and Plasma Processing*, vol. 42, no. 1, pp. 109–129, Oct. 2021, doi: 10.1007/s11090-021-10211-5.

Past Events

Participation of research personnel in conferences

The National Conference on Recent Developments and Evolving Trends in Plasma Science and Technology and Pre-conference Workshop on Modelling and Simulation of Industrial Plasmas was organized by Department of Physics Bharathiar University, Coimbatore and Beam Technology Development Group, Bhabha Atomic Research Centre (BARC), Mumbai in association with the Power Beam Society of India (PSI), Navi Mumbai during September 22 -24, 2022 at Coimbatore. Dr. C. Balasubramanian and Dr. G. Ravi were members of the National Organizing Committee and also chaired technical sessions at the conference.

There was an active participation from scientific staff, research scholars and post doctoral fellows from FCIPT.

The details of oral and poster presentations given by FCIPT staff are as follows:

Oral Presentations:

1. ***Antimicrobial, Agriculture, and Aquaculture Applications of Plasma Activated Water***
   Presenting Author: Vikas Rathore
2. ***Self-Organized Ordered Nanoparticles Arrays for the SERS Applications***
   Presenting Author: Sebin Augustine

Poster Presentations:

1. ***Plasma Assisted Ignition and Combustion of Pulverized Coal***
   Presenting Author: A. Sanghariyat
2. ***Study of the Characteristics of Plasma Discharge Current in Atmospheric Pressure Plasma and Low Pressure Plasma Using the High-Voltage Medium-Frequency Power Source.***
   Presenting Author: C. Patil
3. ***Effect of Oxygen Partial Pressure on Copper Thin Film Deposited by Planar Magnetron Sputtering***
   Presenting Author: Infant Solomon

4. ***Deposition of Titanium Interface on Stainless Steel using Magnetron Plasma Sputtering for Adhesion Improvement of Back Contact Layer***
   Presenting Author: Vishal Dhamecha

5. ***Surface Engineering and Characterization of S-Phase formed in AISI 304 Austenitic Stainless Steel by Plasma Nitrocarburizing***
   Presenting Author: Jeet Vijay Sah

6. ***Investigation of Microwave Produced Plasma at Low Pressure***
   Presenting Author: K. Nigam

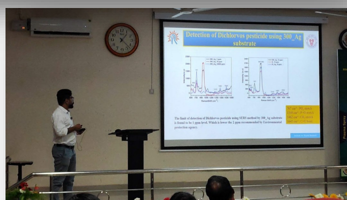
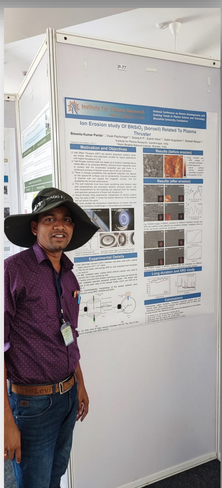
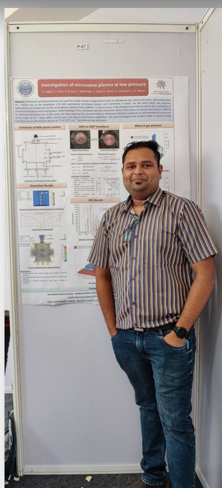
7. ***Ion Erosion Study of BNSiO2 (Borosil) Related to Plasma Thruster***
   Presenting Author: Basanta Kumar Parida

8. ***Effect of External Magnetic Field and Gas Ambient on Iron oxide Nanoparticles Prepared by Arc Plasma Process***
   Presenting Author: Savita

Pictures from the conference






Upcoming Events

2 Day Seminar
on

Plasma for Space and Aerospace Applications

PSAA – 2022

Date: 24-25 November, 2022

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