Cold Plasma Applications

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Indigenous Development of Cold Atmospheric Pressure Plasma Device for Multipurpose Applications

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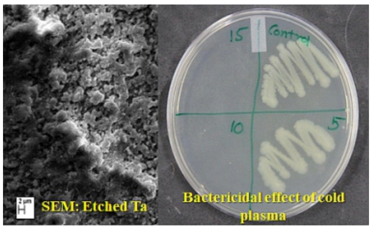

SEM image of plasma etched Ta substrate and some of the results of bactericidal study from developed cold plasma device

ABSTRACT

Cold atmospheric pressure plasma (CAPP) technology is gaining attention for/due to its low cost, simplicity, and ability to create active species. It shows promise for coating, etching, surface treatment and medical applications. In the present study, we have used an indigenously designed 10 MHz atmospheric pressure plasma jet based on Tesla coil principle. The designed device was then applied for the chemical etching as well as bacterial inactivation to show its multipurpose efficacy. Our observations revealed that the device can manage complex molecular plasma gases such as CF_4 and O_2. Effective bactericidal effects can be achieved with the device operating at power levels as low as approximately 20 watts, while efficient etching can be obtained when the device operates at around 80 watts of power.

KEYWORDS: *Cold plasma, Tesla coil, Etching, Bactericidal effects*

Introduction

Cold atmospheric pressure plasma (CAPP) has become a versatile tool with applications ranging from material processing to plasma medicine [1]. In recent years, there has been a significant surge in the research focused on atmospheric pressure cold plasma device [2, 3]. These devices offer the advantage of eliminating the need for expensive and bulky vacuum equipments [4]. Moreover, due to its low gas temperatures and generated reactive species, such type of plasma source finds versatile applications, ranging from industry to biology [5,6]. Atmospheric pressure cold plasma etching has found diverse applications in various industries. In the microelectronics sector, it is employed for the precise and high-resolution etching of semiconductor materials, enabling the production of smaller and more efficient electronic devices. In the automotive industry, it plays a role in improving adhesive bonding and surface treatments, enhancing the durability and performance of the components [7,8]. The medical field benefits from its ability to sterilize medical instruments, ensuring patient safety [9]. In packaging, it aids in surface activation for the improved adhesion of inks and coatings. Furthermore, its eco-friendly attributes align with sustainability goals, making atmospheric pressure cold plasma etching an increasingly valuable tool in modern industrial processes.

Materials and Methods

We have developed an innovative Tesla coil-based device entirely through indigenous efforts. Fig.1(a) illustrates the device's design concept, which comprises three key blocks. The first block involves an input DC source responsible for powering the second block. This second block primarily consists of a MOSFET, a tank circuit, and protective elements. It is linked to a third block, featuring an air-cored coil serving as a Tesla coil. At the output of the second coil, we have a stainless steel electrode where the plasma discharge occurs. Our primary objectives are to optimize power transfer from the second to the third block and establish a resonance between them. To generate cold plasma, the output voltage V_2 must reach a sufficiently high level (several kilovolts). In our specific setup, the circuit operates at approximately 10 MHz, and we have introduced additional resistances strategically in the circuit. This well-engineered device is highly capable of producing uniform and exceptionally stable plasma.

The actual photograph of the operational device is shown in Fig.1(b), and Fig.1(c) shows the close-up view of the developed device during etching experiment. Fig.1(d) shows the dimensions of the different components of the complete CAPP set-up.

The effectiveness of this device was investigated for chemical etching of Ta substrate. A complex plasma with combination of Ar, CF_4 and O_2 was generated for this purpose. CF_4 is the primary etchant gas as it is expected to generate F after dissociation inside plasma to facilitate etching i.e. loss of material from bulk and O_2 is known to enhance the process of etching [8]. During experiment, a flow rate of Ar (8 LPM) and O_2 (100 SCCM) was kept fixed and CF_4 gas flow rate was varied from 0 to 750 SCCM. The etching rate completely depends on the efficient dissociation of Cf_4 into volatile F/F_2. During experiment, operating power was kept constant around ~ 80 watts. Ar gas flow was kept 8 LPM during the experiments for 30 minutes. The etching rate has been measured for each experiment by weighing Ta before ($w1$) and after ($w2$) in a micro-balance for the given time period (t).

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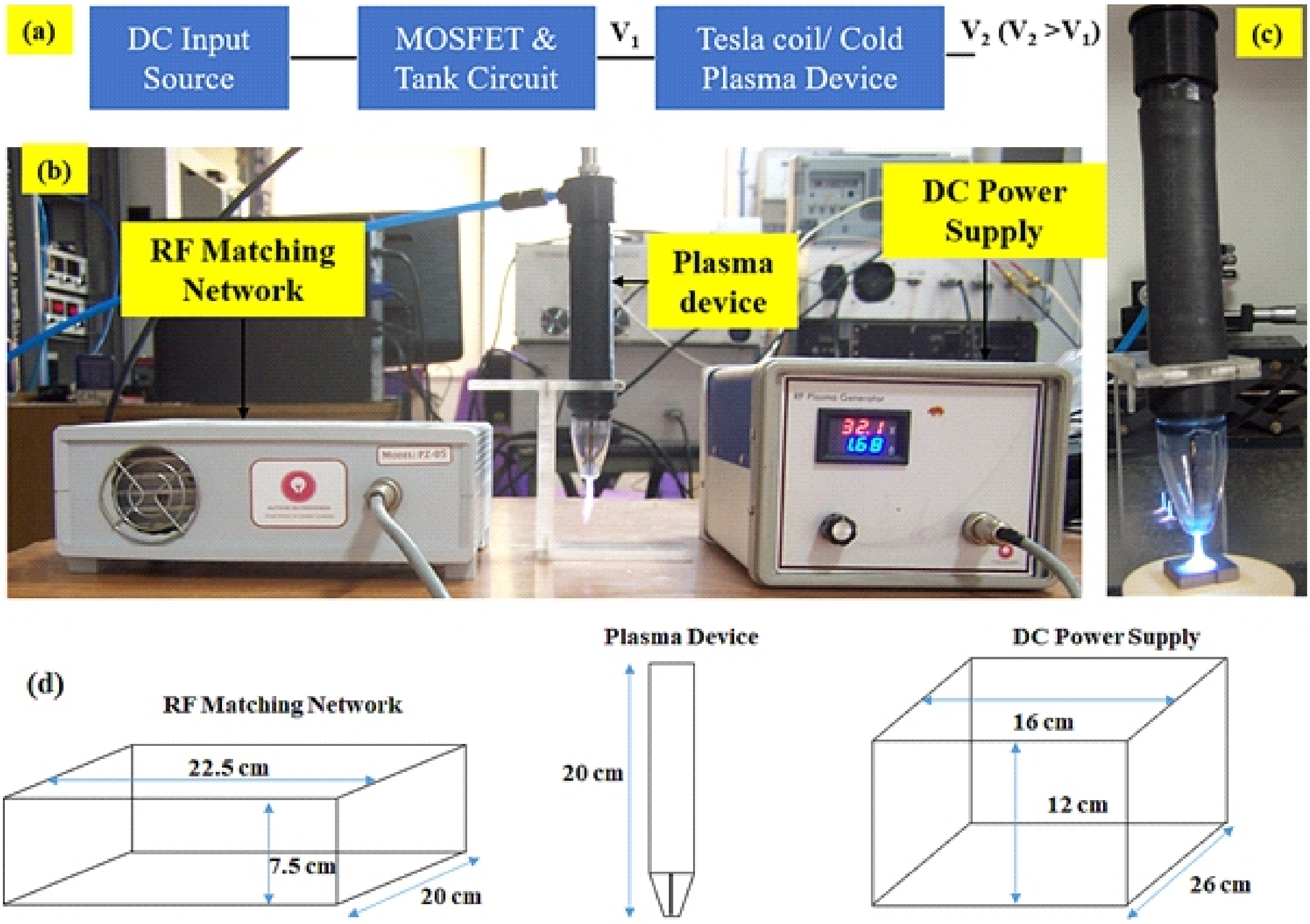


Fig.1: (a) A detailed block diagram of the CAPP device (b) The actual photograph of CAPP device, (c) close-up view of the device during etching experiment, (d) Dimensions of different components of the set-up.

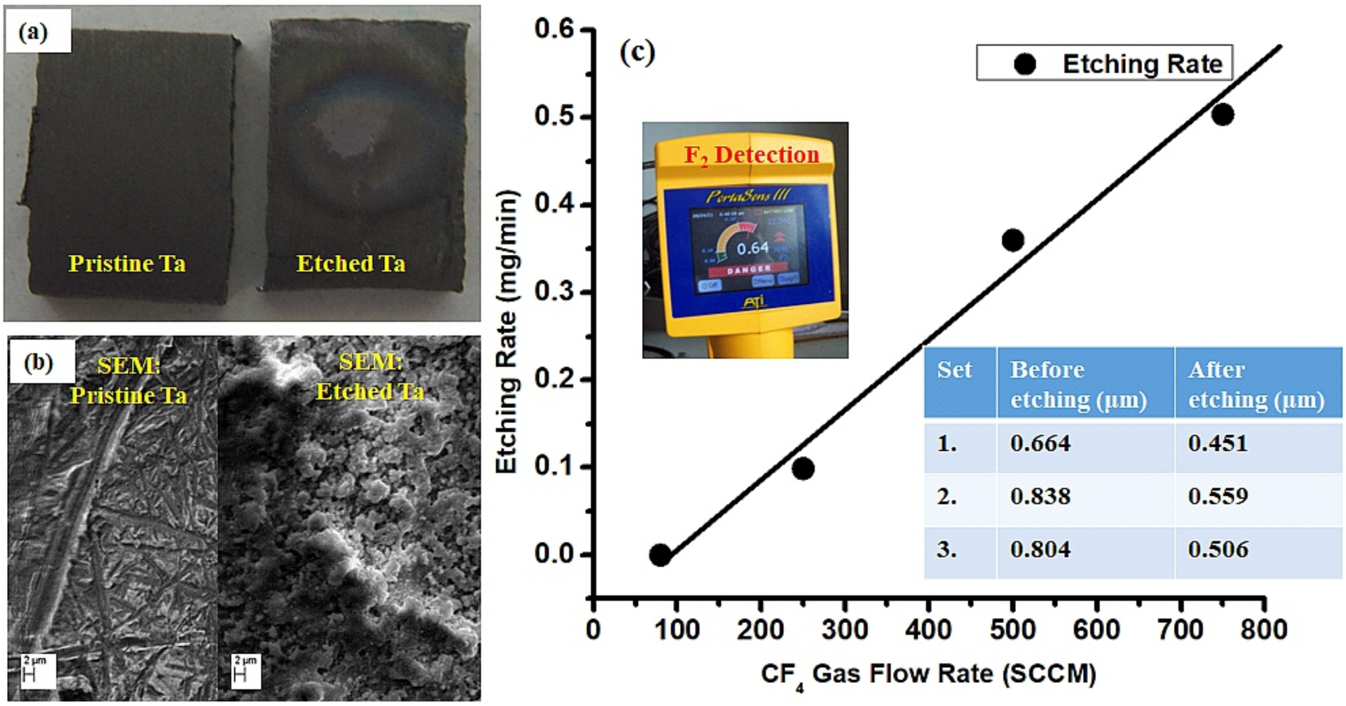
$$\text{Etching rate} = \frac{w1\text{-}w2}{t}\ \text{(mg/min)}$$

Apart from etching, the efficacy of the device was also tested for bacterial inactivation. Two representative bacteria: *Staphylococcus aureus* (*S. aureus*ATCC 9144, Gram positive) and *Escherichia coli* (*E. coli*ATCC 700926, Gram negative) were used for this purpose. During experiment, 8 LPM Ar flow was kept fixed at an operating power ~ 20 watts. Experiments were conducted for 5 to 30 seconds with 5 sec interval. Separate control was kept in both cases. Both bacterial cultures were separately grown in 25 ml tryptic soya broth (TSB) at 37°C, 150 rpm overnight. Plasma treatment was carried out directly on the grown culture. A control sample containing untreated bacteria was also streaked on the agar plate. Following the experiment, agar plates were incubated at 37°C for 24 h and then checked visually for colony formation.

Result

Fig.2(a) shows photograph of Ta substrates before and after etching while Fig.2(b) shows the SEM image of these substrates. Post plasma etching, significant changes have been seen in the micrograph as machining marks have been changed into pits and voids. Fig.2(c) shows graphical representation of the study where it is seen that etching rate increases almost linearly with increasing CF_4 value which means with the increase of CF_4, enhanced generation of nascent fluorine helps to increase the etching rate. With the increase of CF_4 in the plasma, plasma plume length start reducing slightly with each increasing step. As other parameters including the applied power is kept constant, now more energy of plasma was being used in dissociation of molecular CF_4 and eventually beyond 750 SCCM of CF_4 flow rate, the plasma could not be stabilized and it ceased to exist. There was not any significant change in the heat flux from plasma during this experiment. Inset photograph in Fig.2(c) depicts successful detection of F_2. Roughness of the etched and unetched area was measured using a while light source Solarius 3D optical profilometer. The result is presented in tabular form within Fig.2(c). It is seen that controlled etching has improved the roughness of the sample. As received Ta sample showed variation in roughness values as expected due to presence of machining marks.

Fig.3(a) shows the result of the bacterial inactivation experiment. It is seen that exposure of mere 10 seconds is enough to destroy both the bacteria when plasma is in contact with the bacteria. Fig.3(b) shows optical emission spectrum (OES) from the plasma. The spectrum shows presence of bactericidal OH and N_2 (SPS) emission bands in UV range. Presence of these two bands helps in effective destruction of bacteria along with energetic charged species and radicals present in cold plasma. It is important to note that, *S. aureus* and *E. coli* have thermal death point above ~ 60°C and hence cold plasma must have temperature lesser than this value to ensure plasma dominated effects. Fig.3(c) shows the set-up used for temperature measurement of a floating substrate. Here, a stainless-steel disc was kept in contact with plasma in floating condition allowing only radiation loss. Since, maximum



Set	Before etching (µm)	After etching (µm)
1.	0.664	0.451
2.	0.838	0.559
3.	0.804	0.506

Fig.2: (a) Photograph & (b) SEM image of pristine &plasma etched Ta substrate, (c) Graphical representation of etching rate vs. CF_4 gas flow rate, inset photograph of F_2 detection. Table in the inset shows improvement in roughness post plasma etching.

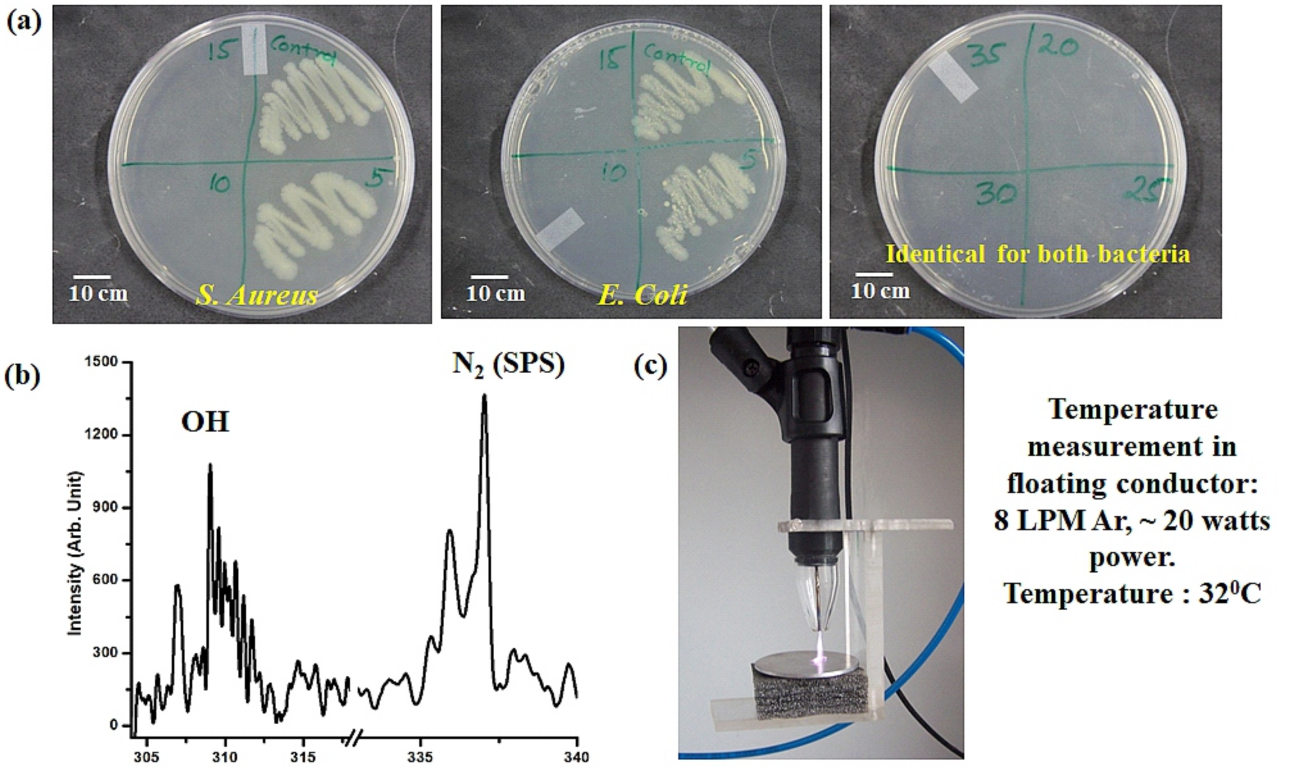


Fig.3: (a) Result of bactericidal study from developed cold plasma device, (b) OES spectrum of the plasma showing presence of OH and N_2 (SPS) bands, (c) set-up for temperature measurement of a substrate.

bacteria treatment time was 30 secs. The disc was kept in this condition for 1 minute and temperature was measured with IR thermometer which turned out to be 31°C.

Conclusion

This study shows development of an indigenous Tesla coil based 10 MHz plasma source which can be used in multipurpose applications. Two examples have been tested in the form of plasma etching and bacterial inactivation in the present case. It is seen that the device is capable of handling complex molecular plasma gas in the form of CF_4 and O_2. The device can be operated as low as ~ 20 watts for effective bactericidal effect and efficient etching can be obtained from the device at ~ 80 watts power.

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