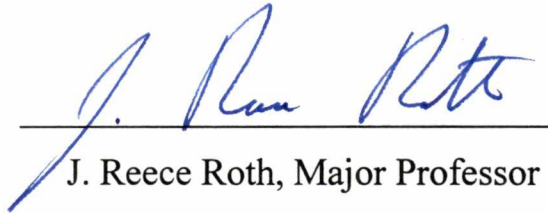


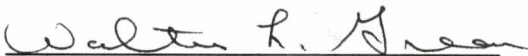
To the Graduate Council:

I am submitting herewith a thesis written by Chaoyu Liu entitled "Exploratory Applications of A One Atmosphere Uniform Glow Discharge Plasma." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Electrical Engineering.


J. Reece Roth, Major Professor

We have read this thesis
and recommend its acceptance:





Accepted for the council:


Associate Vice Chancellor and
Dean of The Graduate School

**EXPLORATORY APPLICATIONS OF A ONE
ATMOSPHERE UNIFORM GLOW
DISCHARGE PLASMA**

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Chaoyu Liu
December 1995

DEDICATION

This thesis is dedicated to my parents
Mr. Shenggang Liu; and Mrs. Chenqi Jiang.

and

My wife and daughter
Mrs. Lan Yang; and Ms Nola Joy Liu.

这篇文献给我亲爱的父母亲：
刘盛纲 (教授, 院士); 蒋臣琪 (教授)
和我亲爱的妻子和女儿：
杨岚 女士; 刘岚华 小姐.

是他们养育了我;
并给予了我人间最美好东西: 爱, 教育, 和支持!

who have given me invaluable educational opportunities and support.

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ABSTRACT

In 1990, Prof. Roth and his graduate students and associates at the University of Tennessee's Plasma Science Laboratory started the experimental and theoretical study of one atmosphere pressure plasma generation and its industrial applications. Significant results have been achieved in both experiment and theory. This work includes research on atmospheric plasma generation; atmospheric plasma applications in the textile industry; surface treatment by atmospheric plasma; and atmospheric plasma applications to fluid mechanical control.

We have developed a new type of one atmosphere uniform glow discharge plasma in parallel plate and modified geometries. This plasma is neither a corona discharge nor a filamentary discharge. It is an RF electrical discharge, and it has been shown experimentally that it can operate at one atmosphere pressure in the steady state. Good agreement between experiment and theoretical predictions have been obtained. It has been shown that the physical processes which make this discharge possible are based on an RF ion trapping mechanism. Our experimental study showed that there exists a certain range of RF frequency, electric field intensity and electrode separation which is required to maintain this discharge. The operating parameters characterizing the plasma in air at one atmosphere obtained experimentally are: RF frequency, under about 10 kHz; RMS electric field about 9kV/cm; plasma number density about 10^{10} electrons/cm³, and the input power density is about 10~200 milliwatts/cm³ (Ref. 12). These parameters imply a

great potential for industrial application of this new type of plasma. In industrial applications, the one atmosphere uniform glow discharge plasma not only would allow many plasma-related surface modification processes to be carried out under production line conditions, rather than in expensive vacuum systems, that increase the budget and make the processes complicated, but also could perform tasks that can be achieved in no other way. The quality of the wettable and wickable polymer fabrics which have been treated by our one atmosphere glow discharge, for example, has been greatly improved.

Another important line of exploratory research has been initiated. We have been able to demonstrate the creation of a one atmosphere uniform glow discharge plasma on a planar surface several millimeters thick. The electric field in this surface plasma layer can provide an electrostatic body force which might find application to boundary layer control, turbulence suppression, drag reduction, and noise reduction in aircraft and aerodynamic flow.

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1. INTRODUCTION

Plasma, since this term was first introduced by Irving Langmuir in 1928, has been extensively studied both experimentally and theoretically. The high density and high temperature regime of fusion energy has been an attractive area for more than forty years. Although a lot of effort has been expended in this area, there is still a long way to go to reach the goal of a practicable fusion power plant. Some successful highlights in plasma research activities already have been obtained, one of the most significant of which is the industrial application of plasma. Today, plasma science and technology have been extensively used in a variety of fields in industry. These include material surface treatment, production of new materials, manufacture of very large scale integrated circuits, metal refining , plasma chemistry, toxic waste treatment, lasers, and many other industrial applications.

In the early 19th century, the steady-state d.c. arc discharge was experimentally developed by Sir Humphry Davy. Plasma was identified as a fourth state of matter by Sir William Crookes in 1879. In this view, as one heats matter from low temperatures, it starts out as a solid, melts to form a liquid, then vaporizes to become a gas. Finally, if one continues heating matter and increasing its temperature, the individual atoms break apart into electrons and positively charged ions. As a result, plasma is produced, forming the fourth state of matter. The terminology plasma means an approximately electrically neutral collection of ions and electrons, which may or may not contain background neutral gas molecules and/or atoms. A plasma

is capable of responding to electric and magnetic fields much more strongly than a neutral gas.

Because of its special behavior, plasma has widespread applications in science and technology, especially in industry. Based on those applications, a new area, low temperature plasma science, has been established. Industrial applications of plasma include: plasma lighting sources, for which one of the earliest applications used electrical arcs for illumination; plasma material surface treatment, an important industrial application, which uses the active species produced by a plasma to modify the surface properties of materials; Plasma etching, which is indispensable for manufacturing very large scale integrated(VLSI) circuits, the core of modern microelectronics; and plasma ion implantation, in which energetic ions are injected into the surface of solid materials, which changes both the atomic composition and the near-surface structure. This can bring much improved surface properties, such as resistance to wear, corrosion, fatigue, and surface hardening. Still more plasma applications are being introduced to industry as the field develops.

If we take a look at most industrial plasma applications, it is very common for plasmas to be generated and operated at low pressures which require a vacuum system. However, for many plasma industrial applications, such as plasma surface treatment processes, plasma cleaning, and some others, if the plasma can be generated and operated at one atmosphere pressure, it will bring significant advantages, including saving the cost of the vacuum system, and surface modification processes carried out without batch processing. The difficulties in generating and operating plasma at one atmosphere pressure for material surface modification are that the plasma should be uniform, should contain enough energetic active species, and

should be controllable. Corona discharges, for example, can be operated at one atmosphere and have been widely used in many industrial applications. Another example is the uniform glow discharge plasma operated at one atmosphere, which can be useful in some applications where corona discharges can't. More new applications of the one atmosphere uniform glow discharge plasma are expected.

Research relating to one atmosphere uniform glow discharge plasma generation and applications has been underway since 1990 in the UTK Plasma Science Laboratory under Prof. J. R. Roth's direction. A sequence of atmospheric uniform glow discharge reactors have been developed, and a uniform glow discharge plasma has been created in several different electrode geometries. All of those plasmas were generated and operated at one atmosphere in air and in other gases. With the two parallel plate electrode geometry, a uniform glow discharge plasma can be created with a large volume(up to 2.8 liters). This uniform glow discharge plasma may have industrial applications for the surface treatment of plastics, such as films, textiles, metals, and other materials.

Figure 1.1 shows the complete one atmosphere uniform glow discharge plasma research reactor(MOD-II) system in the UTK Plasma Science Laboratory in the Department of Electrical and Computer Engineering. This is a two parallel plate capacitive RF glow discharge plasma reactor. On the far left is an RF power supply with an output power from 0 to 5 KVA, frequencies ranging from 1 kHz to 100 kHz, and voltage output from 0 to 10 kilovolts(RMS). Between the power supply and reactor are a resistive matching network and gas flow and pressure monitoring panel. The Plexiglas enclosure on the right contains the plasma reactor. We have conducted



Figure 1.1 The over-all view of the one atmosphere uniform glow discharge plasma research reactor(MOD-II) in the UTK Plasma Science Laboratory of the Department of Electrical and Computer Engineering.

exploratory studies of the parameters and characteristics of this plasma. The measurements and theoretical research results are in qualitative agreement. Two major applications, textile surface treatment and the fluid mechanical boundary layer control using a plasma surface layer, have been studied in our laboratory.

Exploratory experimental research and theoretical analysis in our laboratory of the one atmosphere uniform glow discharge plasma will be described in this thesis. Most of the work was the author's personal or joint work. The one atmosphere uniform glow discharge plasma industrial application research in our laboratory will also be introduced in this thesis.

2. THE ONE ATMOSPHERE UNIFORM GLOW DISCHARGE PLASMA AND ITS APPLICATIONS

2.1 Introduction

Natural lightning and natural ball lightning were the first and are the most familiar one atmosphere plasmas humans have experienced. Ball lightning has been a subject of scientific curiosity for many years, but unfortunately it is not accessible to scientific analysis because it cannot be reproduced in the laboratory under controlled conditions. It may have very high application potential for fusion research and other industrial plasma engineering applications once more is understood about it(Ref. 4). Corona discharges and arc discharges also can occur at one atmosphere, and both have been researched extensively and have been widely used in industry.

Corona discharges normally occur in regions of high electric field near sharp points. Their currents are very low, and typically they are barely visible. When the corona currents are relatively high, corona can be luminous, which can be referred to as a "glow discharge", but corona can't be uniform in two dimensions. In most industrial applications, the currents are low and the entire corona is dark. The power density of the corona discharges and the active species flux which they produce is relatively low. They have been used in the surface treatment of materials, xerography, and other industrial applications. Arc discharges usually operate at moderate voltages, high current, high power density and can release a very high energy and active species flux in a relatively small area. It produces a very high luminance. It has a long history

of industrial applications as a welding source and as a highly luminous source for lighting. Fig. 2.1.1 shows the voltage-current characteristic of the classical DC electrical discharge tube which operates at low pressure(Ref. 1).

We have noticed, in applications of industrial plasmas at one atmosphere, such as corona discharges and arc discharges, there exists a gap in that there are no industrial applications using one atmosphere glow discharges. There are many industrial plasma applications which could benefit from the use of a one atmosphere glow discharge plasma. The reason for this relative under utilization was that the proper way to create a uniform glow discharge couldn't be found; it is difficult to control and maintain the uniform glow discharge in an industrial context; and available power supply equipment can't fit the requirements. In addition, environmental pollution has never been as serious as today, and people have to find some alternative to chemical treatment of materials.

In this chapter we will first discuss corona discharges and their applications. Then we will discuss the RF electrical discharge, particularly the one atmosphere uniform RF glow discharge plasma which we first created in the UTK Plasma Science Laboratory in early 1992.

2.2 Corona Electrical Discharges in Gases and Its Application

Corona, sometimes referred to as a unipolar discharge, is a phenomenon characteristic of Townsend dark discharges which occurs in regions of high electric field near sharp points, edges, or wires in electrically stressed gases prior to the point of electrical breakdown(Ref. 5). It is the easiest electrical discharge that can be made to occur under a wide range of pressure conditions. If the corona currents are relatively high, a corona discharge can be technically a " glow discharge ", visible to the eye. For low currents, the

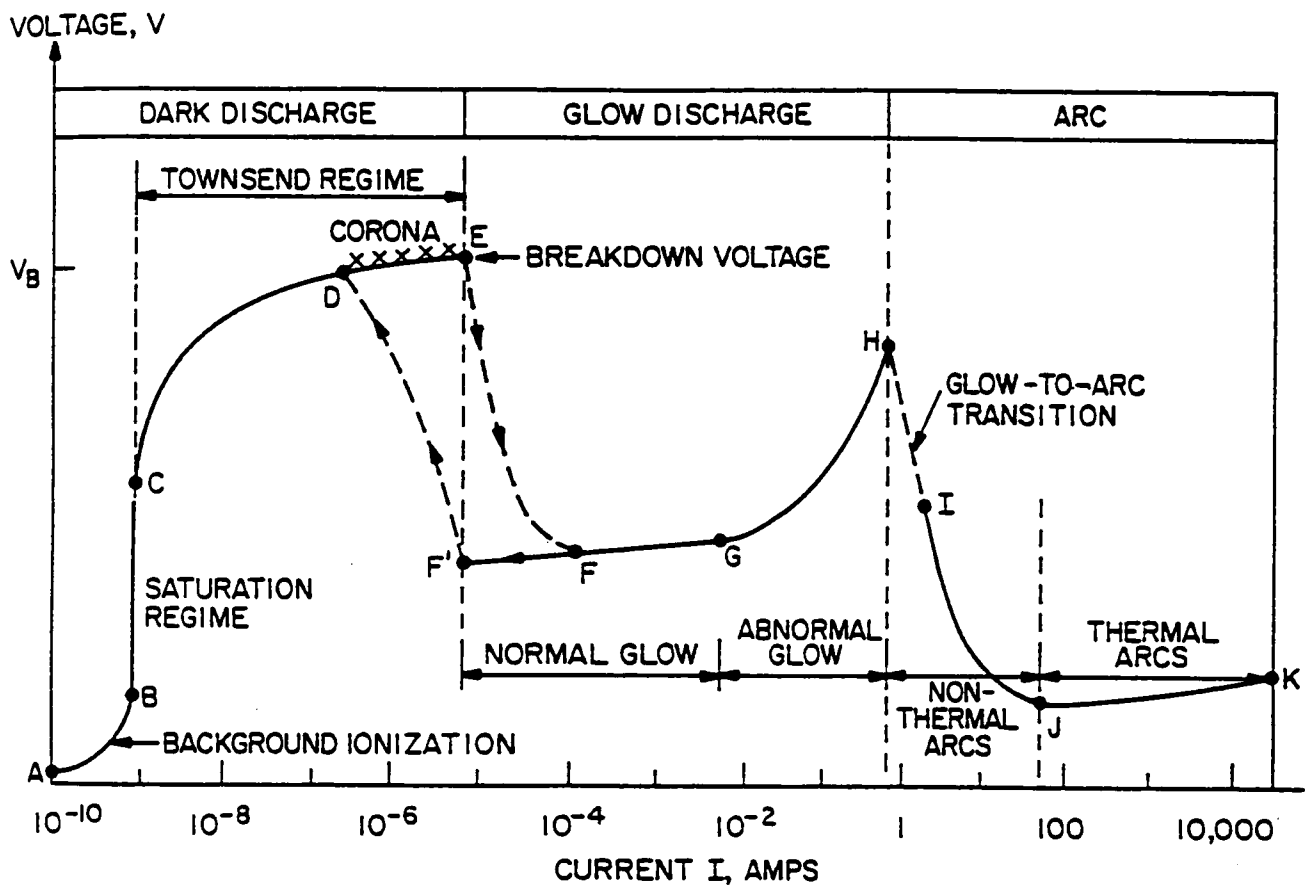


Figure 2.1.1 Universal Voltage-Current characteristic of the classical, low pressure DC electrical discharge tube(Ref. 1.).

entire corona is dark, appropriate to the dark discharges. Related phenomena include the silent electrical discharge, an inaudible form of the filamentary discharge, and the brush discharge, a luminous discharge in a non-uniform electric field, in which many corona discharges are active simultaneously and form streamers which penetrate the highly electrically stressed gas surrounding the point of initiation (Ref. 1).

Corona can initiate from sharp points, fine wires, sharp edges, asperities, scratches, or anything which creates a localized electric field greater than the breakdown electric field of the medium surrounding it. We would like to take a fine wire corona generator as an example to explain the phenomenology of corona generation. Fig. 2.2.1 shows a fine wire with radius a , which is located on the axis of a large grounded cylinder of radius b .

The breakdown voltage for plane parallel electrodes separated by the distance d (meter) of dry air at room temperature and atmospheric pressure is given by(Ref. 6):

$$V_B = 3000d + 1.35kV \quad (2.2.1)$$

The breakdown electric field can be found by dividing equation (2.2.1) by the distance d , to obtain:

$$E_B = \frac{V_B}{d} = 3000 + \frac{1.35}{d}(KV / m) \quad (2.2.2)$$

Thus, we can see that the electric field required to break down dry air at standard atmospheric conditions is about 30KV/cm. In our example of a fine wire with radius a , when the local electric field around the wire exceeds about 30KV/cm, corona is generated. The electric field will be maximum at the surface of the inner conductor, will decrease with radius, and reach a

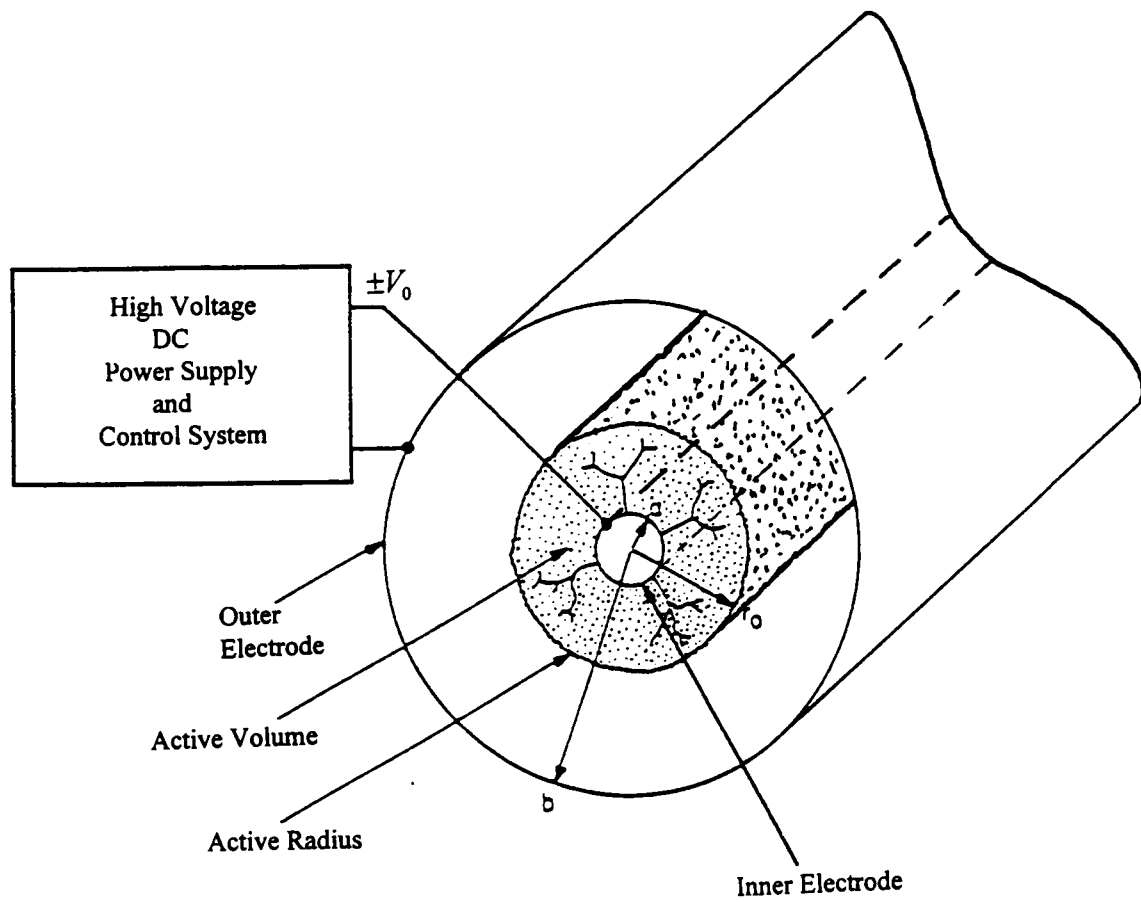


Figure 2.2.1. Fine wire cylinder corona discharge

minimum value at the outer electrode. When the applied voltage, V_0 , is higher than necessary to initiate a corona discharge, the electric field will drop off to the breakdown value given by equation (2.2.1) at a radius r_0 which is called the active radius. This active radius is usually associated with the visible radius of the corona discharge, and it is within this active volume that most corona-initiated plasma chemistry occurs. At or near atmospheric pressure, electrons attach to oxygen quite readily, and therefore the electrical current between the active radius and the outer electrode is carried by either negative or positive ions, depending on the polarity of the voltage applied to the inner electrode. There is little difference in the initiation voltages required to produce a corona current with positive or negative polarity.

There are many industrial applications of the corona discharge. One of the most well-known applications of corona discharge may be xerography. All kinds of copier machine, plain paper fax machine and some others are examples of the successful commercial application of the corona discharge. Electrostatic precipitators are also an important industrial application of the corona discharge. Such precipitators play a major role in industrial air pollution control. Surface treatment of materials by using corona discharge is one of the most attractive applications in the materials processing industry.

Corona can significantly modify the surface properties of treated materials, including the wettability, wickability and printability of fabrics and films. Corona discharge surface treatment can result from either physical modification or electrical modification. Corona antistatic treatment is a typical example of electrical modification of surfaces. Static control is important in movie film, since the untreated film moving at high speed through a camera

may cause electrical discharges which may create visible or ultraviolet radiation. Such radiation will expose the film.

Compared to chemical and some other treatment processes, plasma treatment has many advantages. Greatly reduced chemical pollution and production of unwanted byproducts are among these advantages.

TANDEC (the Textiles and Nonwovens Development Center, the University of Tennessee, Knoxville) recently has successfully used a corona discharge to improve air filtration (Ref. 7). After charging the filter fibrous material by using a corona discharge, the filtration will be increased from normally about 30%(for PP(Polypropylene) filter) to 95% or more. The corona discharges have been playing and continue to play an important role in industrial applications of atmospheric plasma .

2.3 RF Electrical Discharges in Gases and their Applications

From the driving frequency point of view, the generation of plasma can be divided into DC electrical discharge plasma, AC(50/60 Hz) electrical discharge plasma, RF(radio frequency) electrical discharge plasma, microwave plasma and laser plasma. When the power supply is DC, the electrical discharge draws a true current from the power supply in the form of a flow of ions or electrons to the electrodes. It is almost the same for an AC discharge, because the frequency is so low, and the period of the applied frequency is so much longer than the time scale of most physical processes in the plasma, that such discharges can be treated as though they were DC.

RF electrical discharges are operated at much higher frequency and they have different characteristics or advantages in industrial applications. One of the most important advantages is that the currents are displacement currents rather than real currents, so power can be delivered without the requirement

that electrodes be in contact with the plasma. The plasma is not contaminated by electrode material, so it is better for surface treatment, new material production, plasma etching and many other applications. Another important advantage of an RF plasma is that the power supply voltage requirement is usually lower than the voltage needed in a DC electrical discharge. We will discuss this issue in later chapters.

There are two main energy coupling methods which have been used in RF electrical discharges; inductive RF electrical discharges and capacitive RF electrical discharges.

Inductive RF electrical discharges are one of the most commonly used methods of generating plasma in industry. Figure 2.3.1. shows an inductively coupled plasma generator. In inductive coupling, radio frequency power is coupled to the plasma through the oscillating magnetic field. The frequencies used for inductive coupling to plasmas range from below 10 kHz, to as high as 30 MHz or even more. When the frequency is higher, in the microwave frequency range, both the oscillating electric field and magnetic field will be coupled to the plasma. The working gas pressure in inductively coupled plasmas can range from a few torr to a few hundred torr, but can range as high as one atmosphere.

Capacitive RF electrical discharges are another important type of RF discharge. The RF power is transferred to the plasma principally through an oscillating electric field. A unmagnetized, parallel plate plasma reactor schematic is shown on figure 2.3.2. which has two electrodes, separated by the distance d . An RF voltage with a frequency ω is applied to the electrodes. It is usually desirable to have a matching network between the power supply and plasma reactor in order to deliver maximum power to the plasma and also

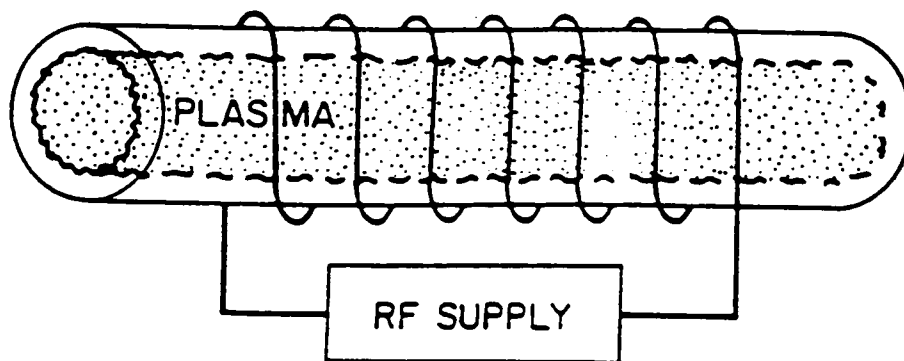


Figure 2.3.1 Schematic of an inductively coupled RF plasma generator(Ref. 1.).

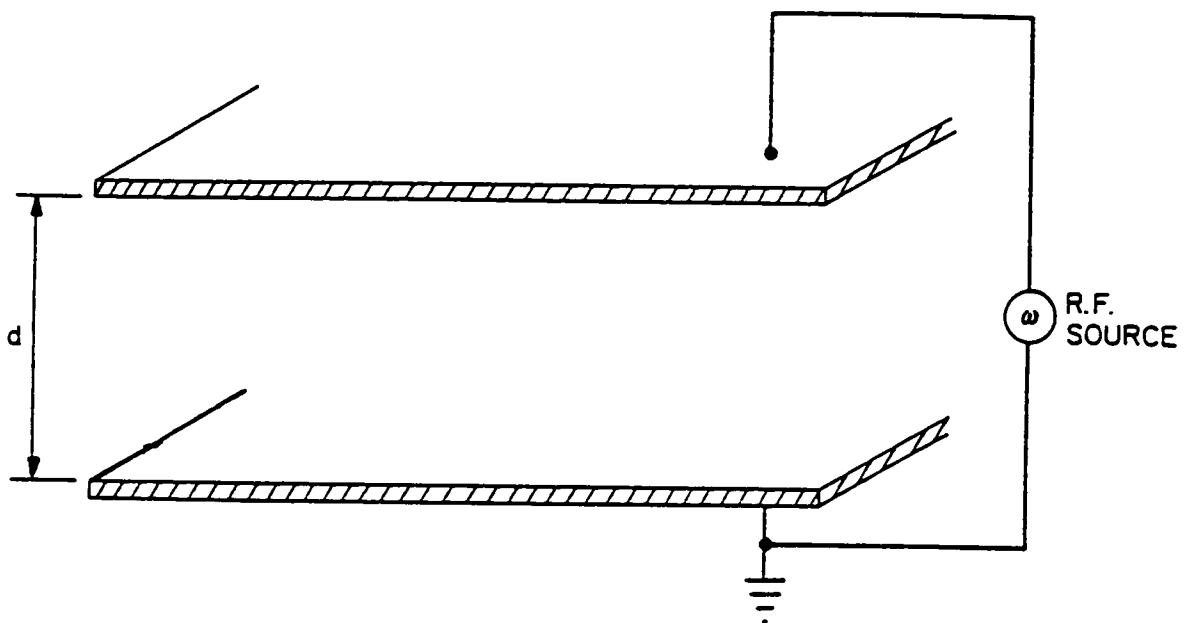


Figure 2.3.2 Schematic of an unmagnetized, parallel plate plasma reactor with plate separation d .

to protect the power supply from reflected power or overloads. The operating pressure can be adjusted over a very wide range.

To review, RF power can be coupled to plasma through either an oscillating magnetic field (inductive coupling), an oscillating electric field (capacitive coupling), or both (quasi-optical, or microwave coupling). Whether and to what extent these oscillating fields interact with the plasma depend on the frequency of the oscillation, the electron plasma frequency, and the electron collision frequency in the plasma.

The electron plasma frequency is a function of the electron number density and is given by(Ref. 2):

$$\nu_{pe} = \frac{e}{2\pi} \sqrt{\frac{n_e}{\epsilon_0 m_e}} \text{ (Hz)} \quad (2.3.1)$$

When the frequency of incident electromagnetic radiation is below the electron plasma frequency, the inertia of the electrons is low enough for them to respond to the electric field in the incident electromagnetic wave, and the electron therefore can absorb energy from the oscillating electric field. This absorbed energy may be converted to heat through collisions, or re-radiated in the form of a reflected electromagnetic wave from the surface of the plasma. If the frequency of the incident electromagnetic radiation is higher than the electron plasma frequency, then the inertia of an electron is too high to allow it to respond strongly to the incident electromagnetic wave. The electromagnetic wave is then free to propagate through the plasma as a quasi-optical dielectric medium, in which the interaction of the

electromagnetic radiation with individual electrons is relatively insignificant, except at the gyroresonance frequency in magnetized plasmas(Ref. 1).

In spite of the comparatively higher cost of RF power relative to DC, there are many industrial applications for which the advantages of RF power outweigh its higher cost. One of the most widely used industrial plasma sources is the inductively coupled RF plasma torch, a schematic of which is illustrated in figure 2.3.1. The plasma can be started with a pulsed high voltage applied to a starting rod, and maintained by RF power coupled through an axisymmetric coil wrapped around the plasma, which is part of an RF tank circuit. This inductive plasma torch is used for the industrial heating and plasma spraying of refractory materials.

The one atmosphere plasma reactor is a capacitive RF electrical discharge. Figure 2.3.3 shows the original MOD-I design of the one atmosphere uniform glow discharge plasma reactor in the UTK(University of Tennessee, Knoxville) Plasma Science Laboratory. This reactor is designed to operate at one atmosphere of pressure at room temperature, and it can generate a steady-state glow discharge plasma. This one atmosphere uniform glow discharge plasma should find many industrial applications. First, it makes unnecessary expensive vacuum systems which enforce batch processing in plasma surface treatment processes. It also makes possible industrial applications which must be operated at one atmosphere. Aircraft boundary layer control by using a one atmosphere glow discharge plasma(Ref. 3) is a good example. Figure 2.3.4 shows a cylindrical surface layer plasma generator which makes creating a layer of plasma at one atmosphere of pressure possible on the surface of aircraft. We will discuss these in detail in a later chapter.

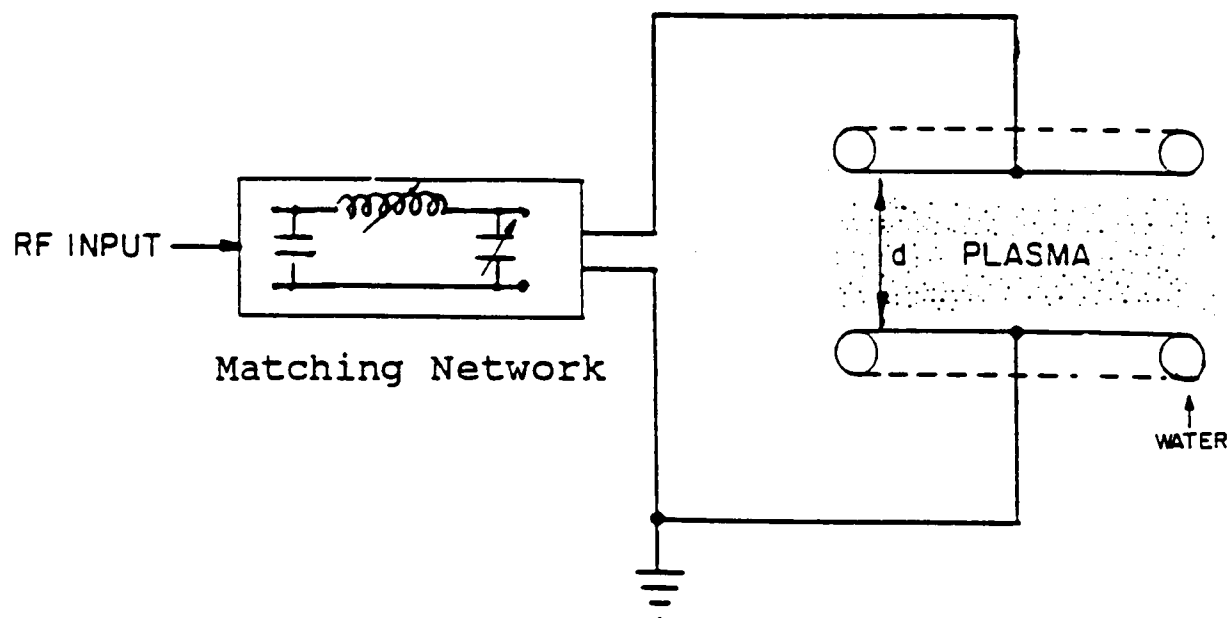


Figure 2.3.3 First design of the one atmosphere plasma reactor in the UTK Plasma Science Laboratory.

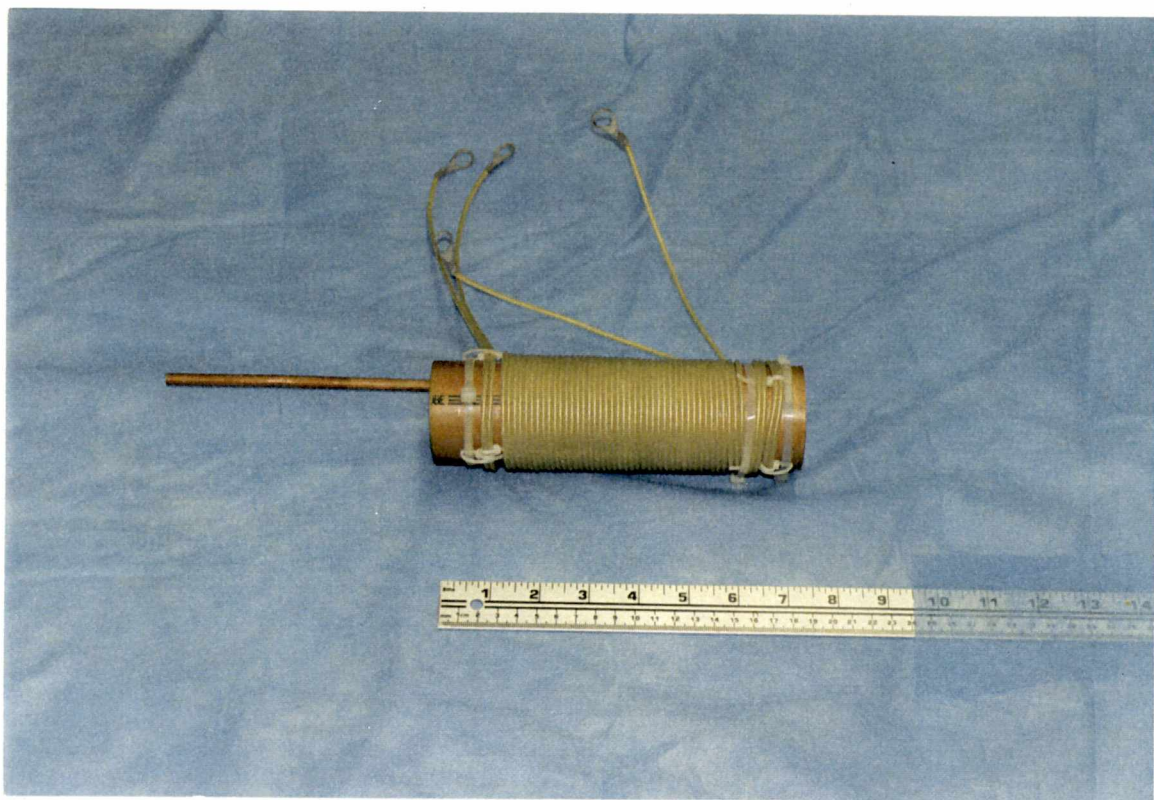


Figure 2.3.4 The surface layer plasma generator on a 5cm diameter metal cylinder .

3. THEORY OF RF UNMAGNETIZED GAS BREAKDOWN AT ONE ATMOSPHERE PRESSURE

3.1 Introduction

The capacitive parallel plate plasma reactor is one of the most common RF glow discharge plasma sources, and is widely used in industrial plasma processing. Our recent development of the one atmosphere uniform glow discharge(Ref. 4) as an industrial plasma source is based on this configuration. Similar research has been done by Kanda, et al. (Ref. 10) on what appears to be a related type of plasma, the dielectric barrier (or filamentary) discharge. A schematic of the MOD-I one atmosphere uniform glow discharge plasma reactor system developed at the UTK (University of Tennessee, Knoxville) Plasma Science Laboratory is shown in figure 3.1.1. As a graduate research assistant, I worked with Prof. Roth to design and build the first one atmosphere glow discharge plasma reactor system. Because of the success of this one atmosphere glow discharge plasma, in early 1992 we started to work on its theoretical analysis(Ref. 13). We have reached good agreement between our experimental work and the theoretical study.

In this chapter I will describe the theoretical analysis for our one atmospheric plasma discharge in a parallel electrode plate configuration.

3.2 RF Unmagnetized Gas Breakdown in a Capacitive Plasma Reactor

The configuration of the parallel plate reactor is shown in figure 3.2.1. In order to produce a glow discharge between these two parallel plate

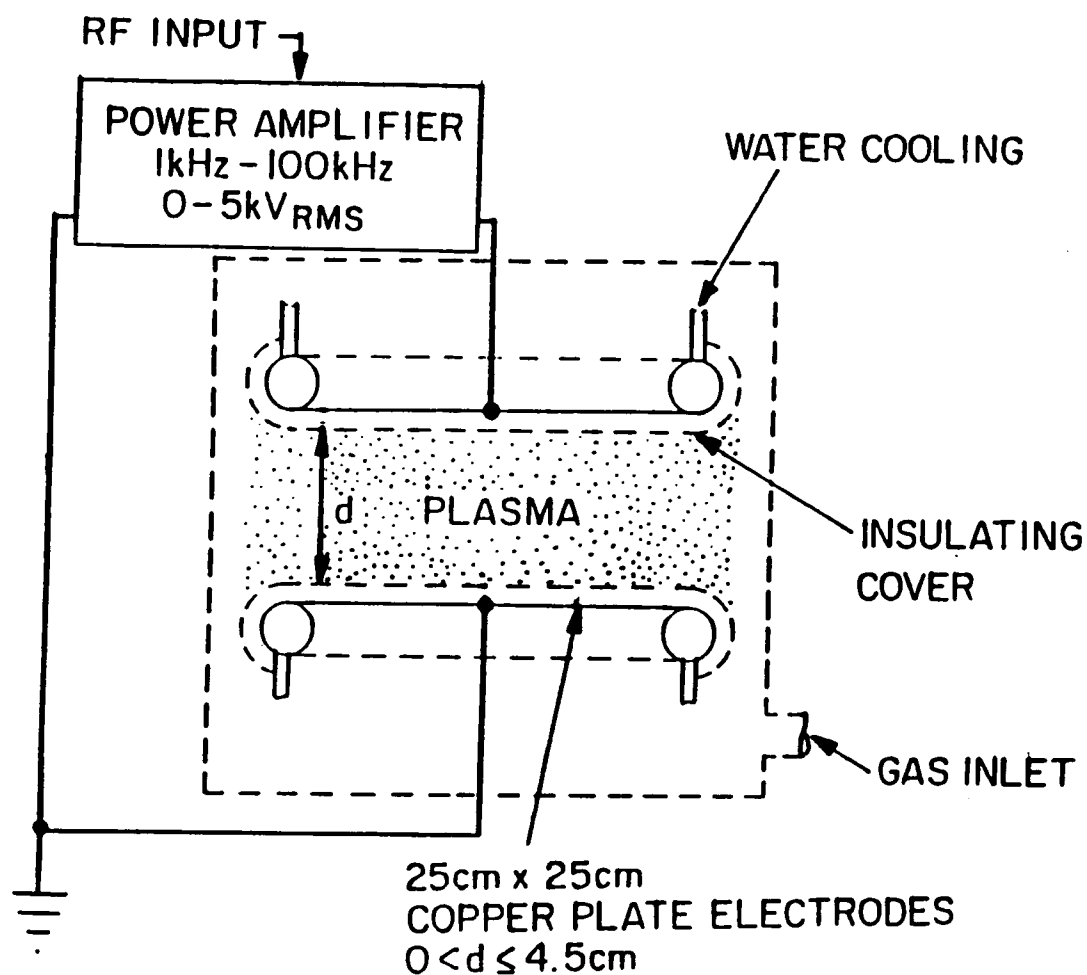


Figure 3.1.1 A schematic drawing of the MOD-I one atmosphere uniform glow discharge plasma reactor system in UTK Plasma Science Laboratory.

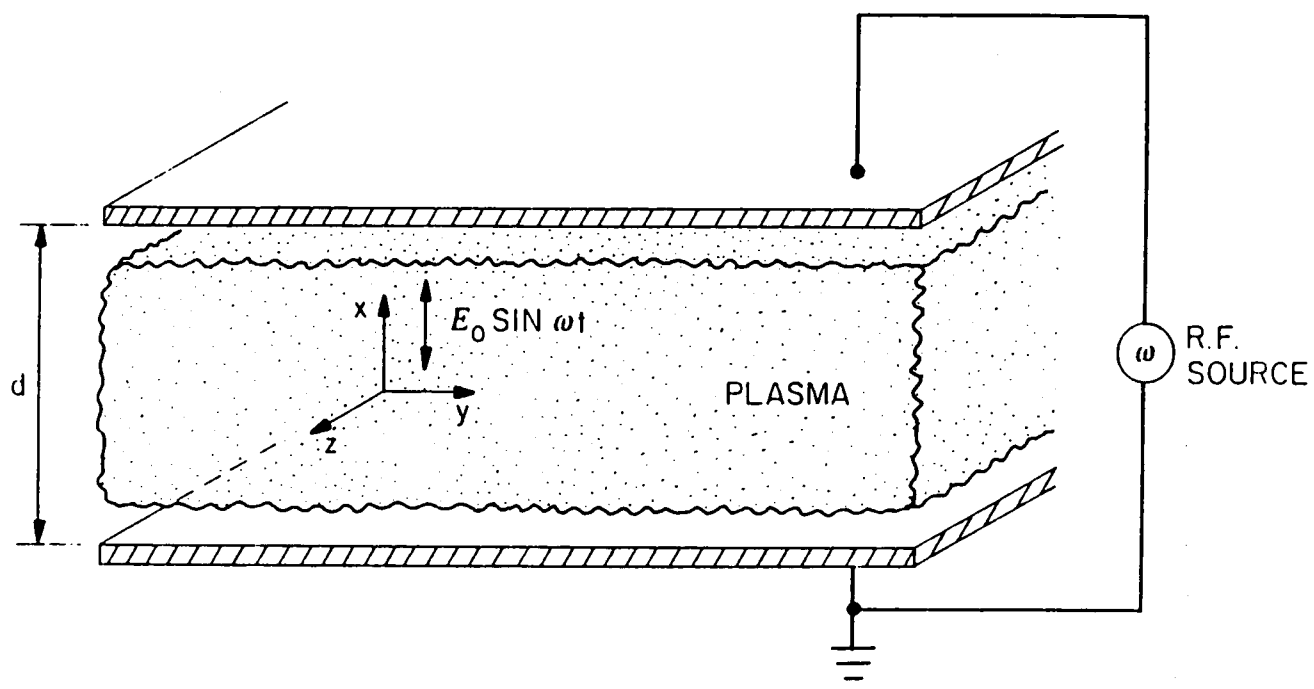


Figure 3.2.1 Schematic of the one atmosphere uniform glow discharge plasma reactor.

electrodes, a certain range of electric field strength, RF frequency, and distance d are required for a given working gas.

In figure 3.2.1, a Cartesian coordinate system is used with the applied electric field in the x direction. The maximum amplitude of the electric field between the grounded electrode and upper electrode is E_0 , and the separation of the two electrodes is d .

The electric field between the electrodes shown on figure 3.2.1 is given by

$$\vec{E} = (E_0 \sin \omega t, 0, 0). \quad (3.2.1)$$

This one atmosphere glow discharge is operated in a magnetic-field-free plasma. The equation of motion for the ions or electrons in the Lorentzian model is given by

$$\vec{F} = m\vec{a} = -m\nu_c \vec{v} - e\vec{E}, \quad (3.2.2)$$

where the first term on the right-hand side is the Lorentzian collision term, according to which the momentum $m\vec{v}$ is lost with each collision that occurs with a collision frequency ν_c . The x component of (3.2.2) is given by

$$m \frac{d^2 x}{dt^2} + m \nu_c \frac{dx}{dt} = eE_0 \sin \omega t, \quad (3.2.3)$$

where the electric field $\vec{E}$ from equation (3.2.1) has been substituted into equation (3.2.2). The general solution to equation (3.2.3) is

$$x = C_1 \sin \omega t + C_2 \cos \omega t \quad (3.2.4)$$

where the constants C_1 and C_2 are given by

$$C_1 = -\frac{eE_0}{m} \frac{1}{(\omega^2 + \nu_c^2)} \quad (3.2.5)$$

and

$$C_2 = -\frac{\nu_c eE_0}{\omega m} \frac{1}{(\omega^2 + \nu_c^2)} \quad (3.2.6)$$

In our experiment, helium is the easiest gas to break down. The one atmosphere helium glow discharge is operated at frequencies between 1 kHz and 30 kHz, where, for helium at one atmosphere (Ref. 11), $\nu_{ci} \approx 6.8 \times 10^9$ ion collisions/s, and $\nu_{ce} \approx 1.8 \times 10^{12}$ electron collisions/s. The collision frequency for ions and electrons is much greater than the RF frequency, $\nu_c \gg \omega$. The relation $\nu_c \gg \omega$ for ions and electrons, implies that C_2 is much greater than the constant C_1 , or

$$C_2 \approx -\frac{eE_0}{\omega m \nu_c} \gg C_1. \quad (3.2.7)$$

The ion or electron position in the x direction can be obtained by substituting equation (3.2.7) into equation (3.2.4), to obtain

$$x(t) \approx -\frac{eE_0}{\omega m \nu_c} \cos \omega t \quad (3.2.8)$$

The rms displacement of the ion or electron during a half-cycle is given by

$$x_{rms} \approx \frac{2}{\pi} \frac{eE_0}{m\omega v_c} \quad (3.2.9)$$

If ν_0 is the driving frequency in Hz, and V_{rms} is the rms supplied voltage, then the radian RF frequency is given by

$$\omega = 2\pi\nu_0 \quad (3.2.10)$$

and the maximum electric field between the plates can be approximated by the maximum voltage V_0 appearing between them,

$$E_0 = \frac{V_0}{d} = \frac{\pi V_{rms}}{2d} \quad (3.2.11)$$

If the particle in question moves across the discharge width from the median plane to one of the electrode plates during one full cycle, then we have

$$x_{rms} \leq d/2. \quad (3.2.12)$$

Substituting equations (3.2.10) to (3.2.12) into equation (3.2.9) yields the relationship of the voltage, frequency and the electrode separation distance:

$$\frac{1}{2} \approx \frac{eV_{rms}}{2\pi m \nu_0 v_c d} \quad (3.2.13)$$

If we now solve for the critical frequency above which the particle should remain in the plasma volume, we have

$$\nu_0 \approx \frac{eV_{rms}}{\pi m v_c d^2} (\text{Hz}) \quad (3.2.14)$$

where ν_c is the collision frequency for ions or electrons.

Because

$$m_e \nu_{ce} < M_i \nu_{ci} \quad (3.2.15)$$

at lower frequency the ion will trap first, and the glow discharge will build up. Once the frequency is high enough to let the electrons be trapped, the filamentary discharge starts. This is exactly what we have seen in our experiment.

We have derived the working relationship between the electrode separation, the driven frequency and the voltage. The conclusion is that the RF frequency, electric field, and the electrode separation given by equation (3.3.14) will be required to maintain the one atmosphere uniform glow discharge.

4. EXPERIMENTAL GENERATION AND MEASUREMENT OF ONE ATMOSPHERE UNIFORM GLOW DISCHARGE PLASMA AT ONE ATMOSPHERE PRESSURE

4.1 Introduction

In industrial plasma engineering, a steady-state atmospheric glow discharge would allow many plasma-related surface modification processes to be carried out under production line conditions, rather than in vacuum systems which require batch processing. 1988, Prof. Roth first proposed a development program which would lead to a one atmosphere glow discharge plasma. After two years, AFOSR(Air Force Office of Scientific Research) support was obtained which made possible the design of the MOD-I reactor in 1990 and the procurement of parts and equipment in 1991. About at the end of summer, 1991 we built our first one atmosphere uniform glow discharge plasma reactor(now we call it MOD-I)(Ref. 4). Figure 4.1.1 shows a picture of the MOD-I reactor with a uniform glow discharge plasma. This first plasma was generated in January, 1992. During the following two years, we conducted a series of exploratory experiments and measurements using the MOD-I reactor. The experiments included generation and measurement of the glow discharge plasma at one atmosphere pressure with various working gases, power supply voltages, driving frequencies and electrode separations. We also tested possible industrial applications of the one atmosphere glow discharge plasma, which include the surface treatment

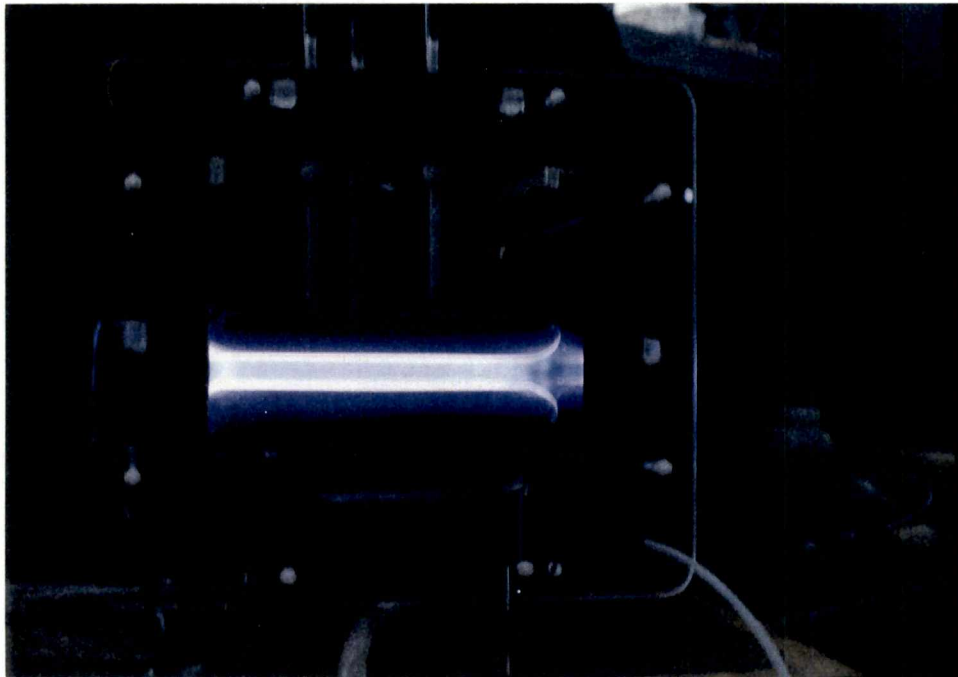


Figure 4.1.1 The one atmosphere MOD-I plasma reactor with a uniform glow discharge plasma generated by RF at 3KV RMS, and frequency equals 10KHz, with helium as working gas.

of nonwoven textiles, a new lighting source, plasma cloaking of aircraft, and initial, exploratory experiments on fluid mechanical boundary layer control by using a plasma surface layer. In July of 1993, Dr. Paul D. Spence joined our one atmosphere glow discharge plasma generation, measurement and application research. A more advanced one atmosphere plasma reactor(Ref. 12), MOD-II, was built based on MOD-I. Figure 4.1.2 shows the MOD-II reactor built by Dr. Spence. It had an advanced working gas flow control and measurement system. An impedance matching system also was added between the power amplifier and the plasma reactor. This system can improve the power amplifier performance, as well as protect the amplifier.

In this chapter, I will describe some details of our atmospheric plasma generator and characteristic measurements based largely on work I did on the MOD-I reactor. Some of the data were taken on the MOD-II reactor.

4.2 Experimental Generation of a Steady-State Uniform Glow Discharge at Atmospheric Pressure

In 1991, we started experiments on a plasma reactor which generates a steady-state uniform glow discharge plasma at one atmosphere pressure, with air as one of several possible working gases. In January, 1992, we successfully generated plasma at one atmosphere pressure in helium gas and a mixture of helium with air. Figure 4.2.1 shows a picture of the power supply system set up. The power amplifier is a Ling AP-3/5 power amplifier, which is capable of supplying up to 5 kilowatts of RF power at RMS voltages up to 10 kV and over a frequency range from 1 KHz to 100 KHz. Figure 4.2.2 is a picture of the steady-state uniform glow discharge plasma generated between the two parallel electrode plates. If you look carefully at this picture you can see the plasma is unlike the plasma in figure 4.1.1. The reason will be

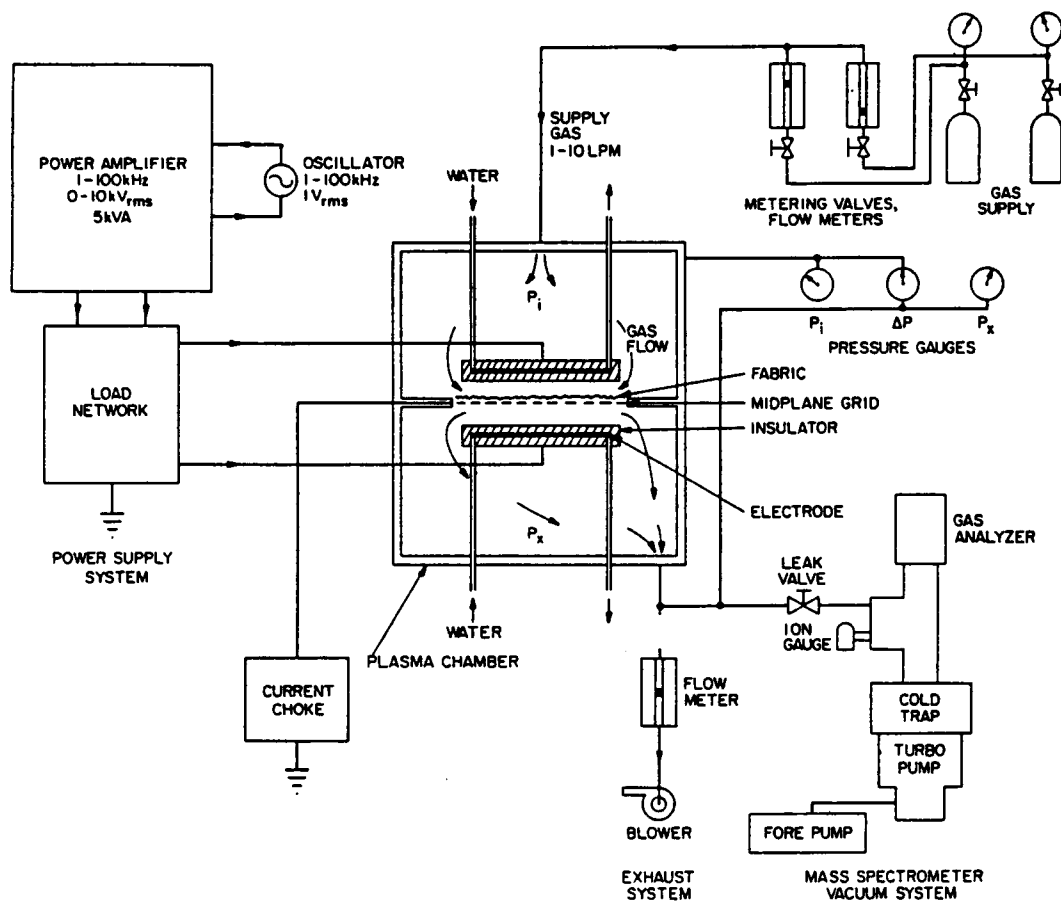


Figure 4.1.2 A schematic drawing of the MOD-II advanced one atmospheric discharge plasma reactor system(Ref. 12).

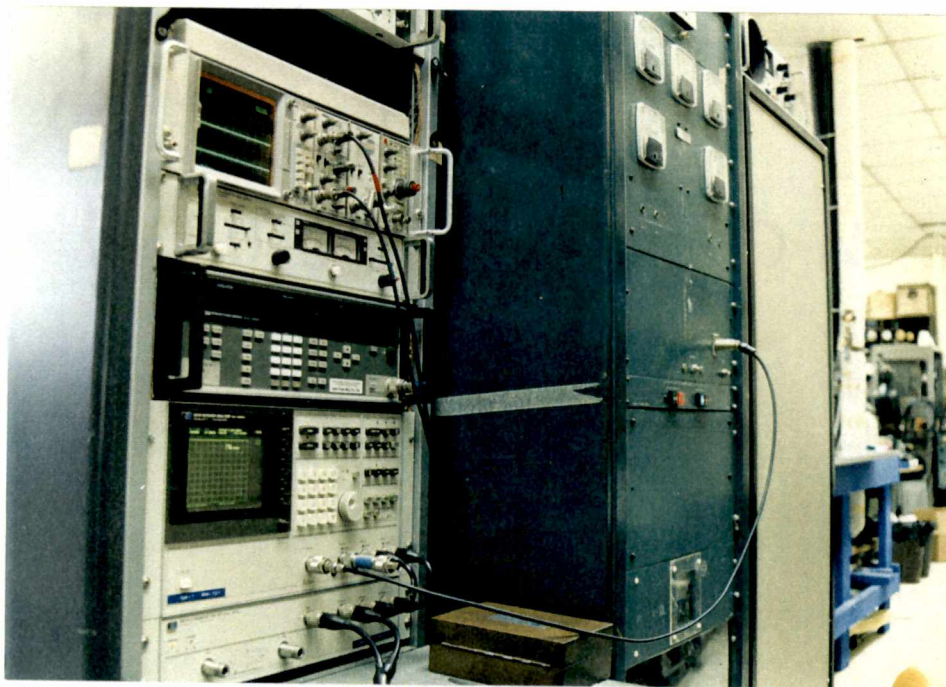


Figure 4.2.1 Power supply system for the MOD-I reactor.

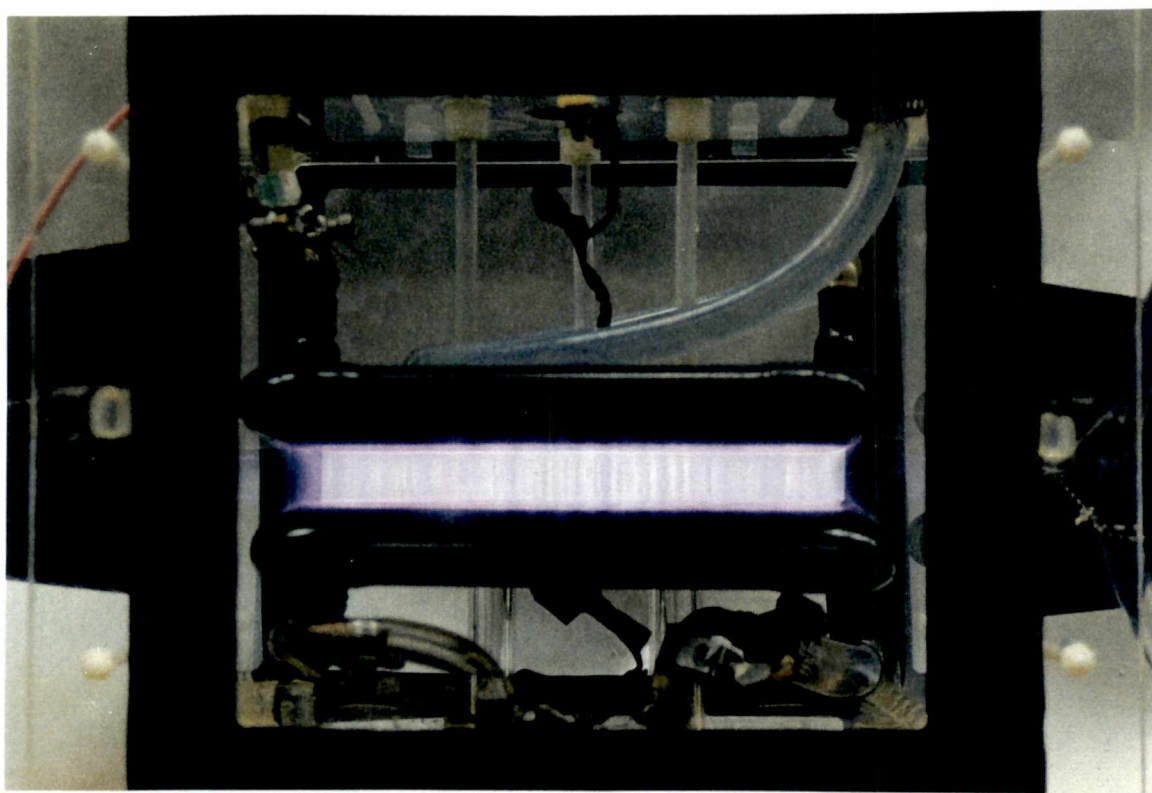


Figure 4.2.2 A steady-state uniform glow discharge plasma in the MOD-I reactor.

explained later. The electrode is rubber insulated and the edge of the reactor is supplied with water cooling, see figure 4.2.3. As shown in figure 4.2.2, the entire reactor was contained in a specially designed Plexiglas box. There are several advantages of having this box in place. Figure 4.2.4 shows the original design of the container box. Because this box is made of Plexiglas, it gives a very good view of the plasma and electrodes from outside. This box also can absorb a big percentage of UV radiation generated by the plasma. Also, this box allows us to change the working gas and makes our working condition more safe if ozone or other harmful active species are generated by the plasma.

Here we show the results of a group of exploratory experiments on one atmosphere uniform glow discharge plasma generation. In the experiment for which the set up is as shown in figure 4.2.2 with two parallel plate electrodes, we generated a glow discharge plasma under the condition of approximately one atmosphere pressure with different working gases and power supply voltages and frequencies.

From figure 4.2.5 to figure 4.2.8 are a series of pictures which show the appearance of the glow discharge plasma as a function of frequency. In this experiment, we used helium as the working gas. The power supply voltage is about 4 KV (RMS). The electrode separation is about 3 cm and the frequency ranges up to 80 KHz. The results of this experiment gave us good qualitative agreement with our one atmosphere unmagnetized gas breakdown theoretical results(Ref. 13). We explain the reason for the different appearance of the plasma as follows: at the lower frequency the ions will be trapped between the electrodes, and the glow discharge will build up; once the frequency is high enough also to trap the electrons, a filamentary

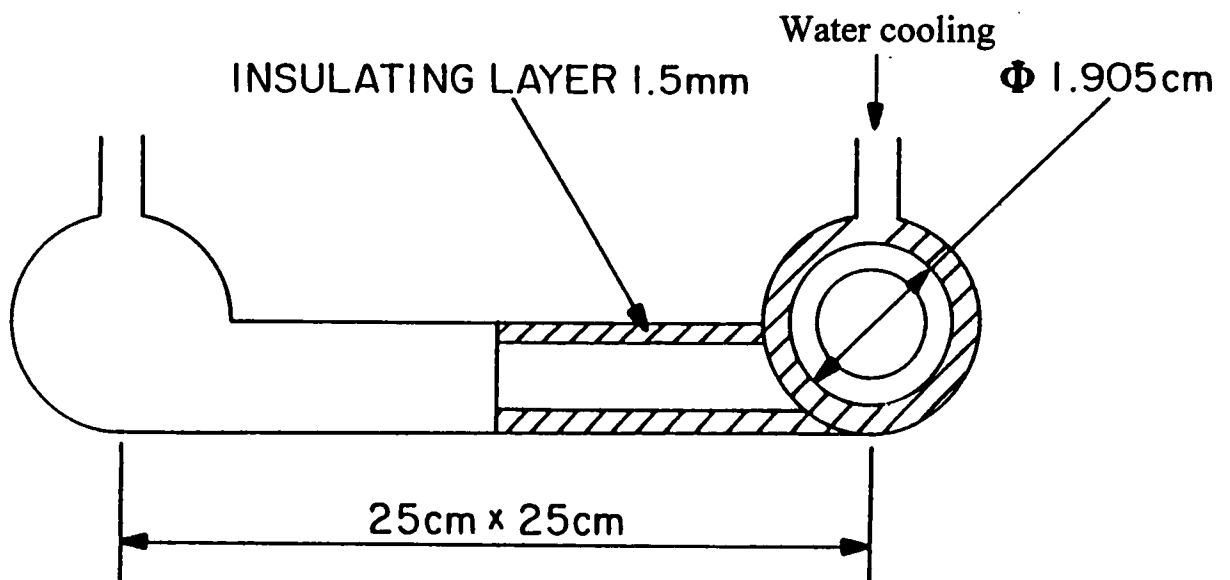


Figure 4.2.3 Rubber insulated electrode with water cooling system on the edge.

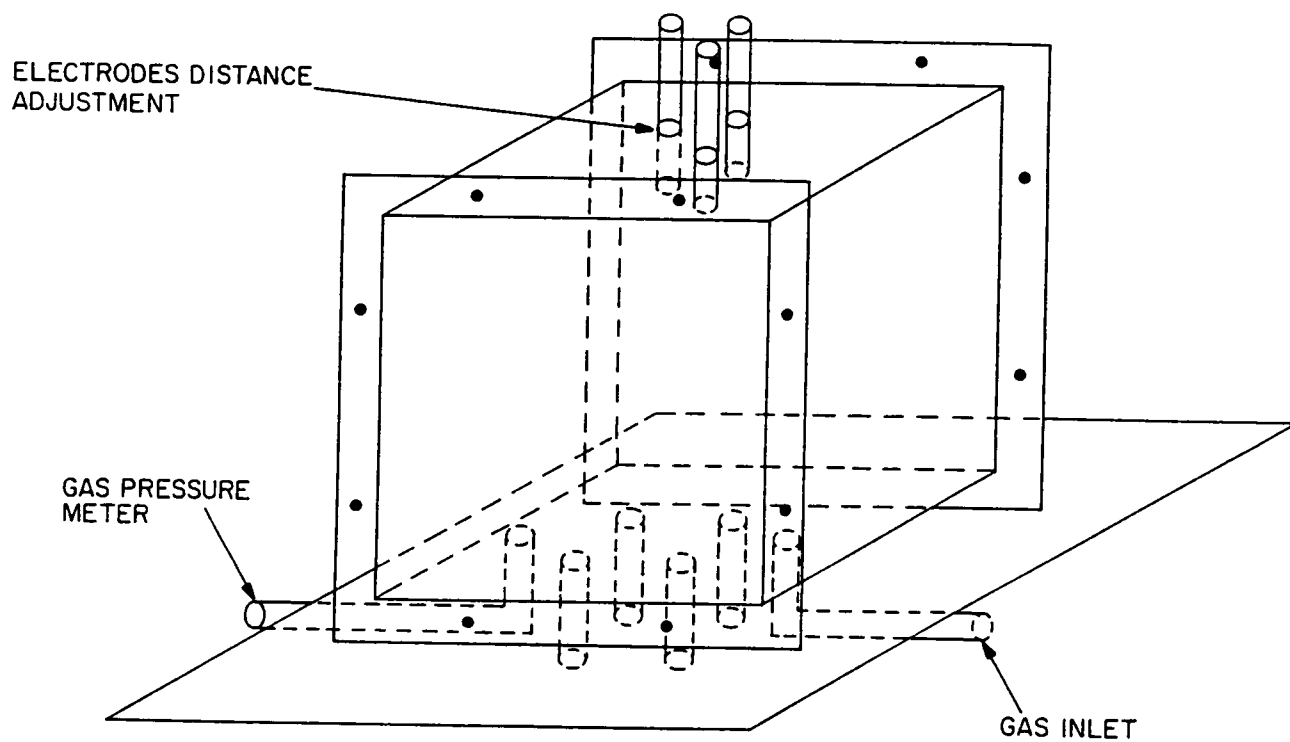


Figure 4.2.4 A schematic drawing of the original design of the MOD-I container box.

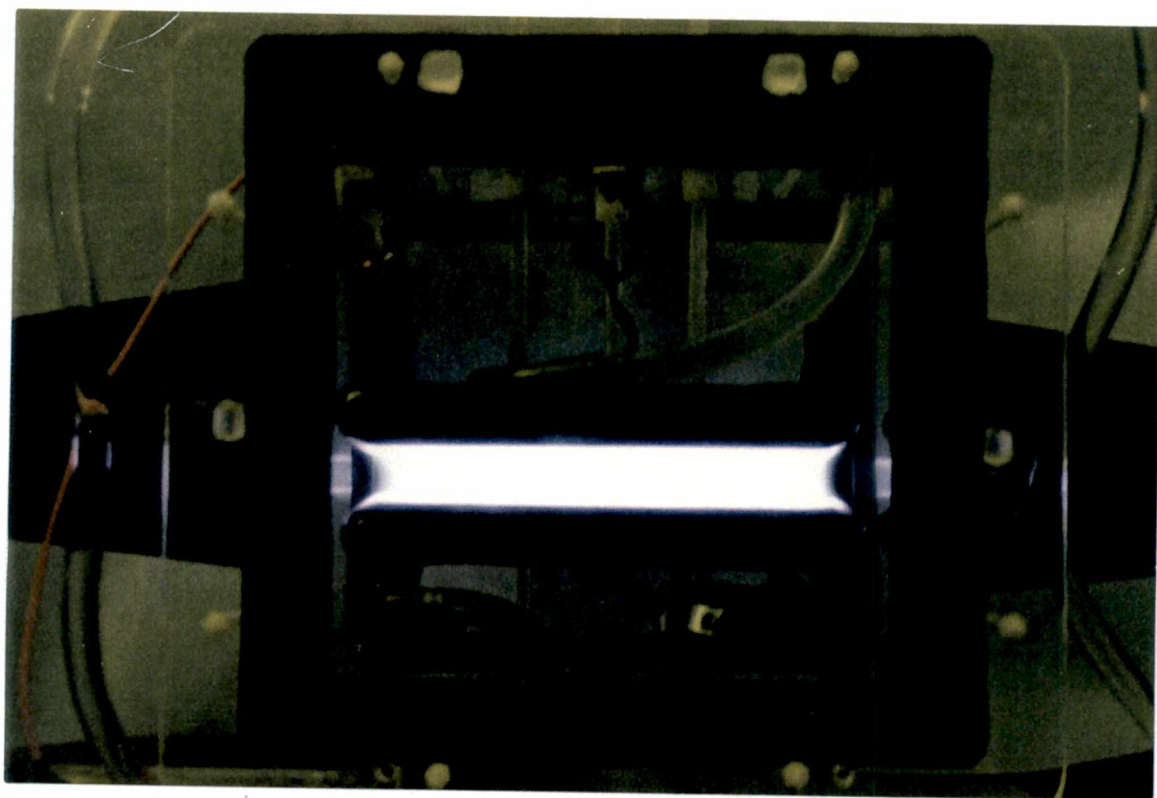


Figure 4.2.5 Appearance of the one atmosphere uniform glow discharge as a function of frequency; the frequency is 20 KHz.

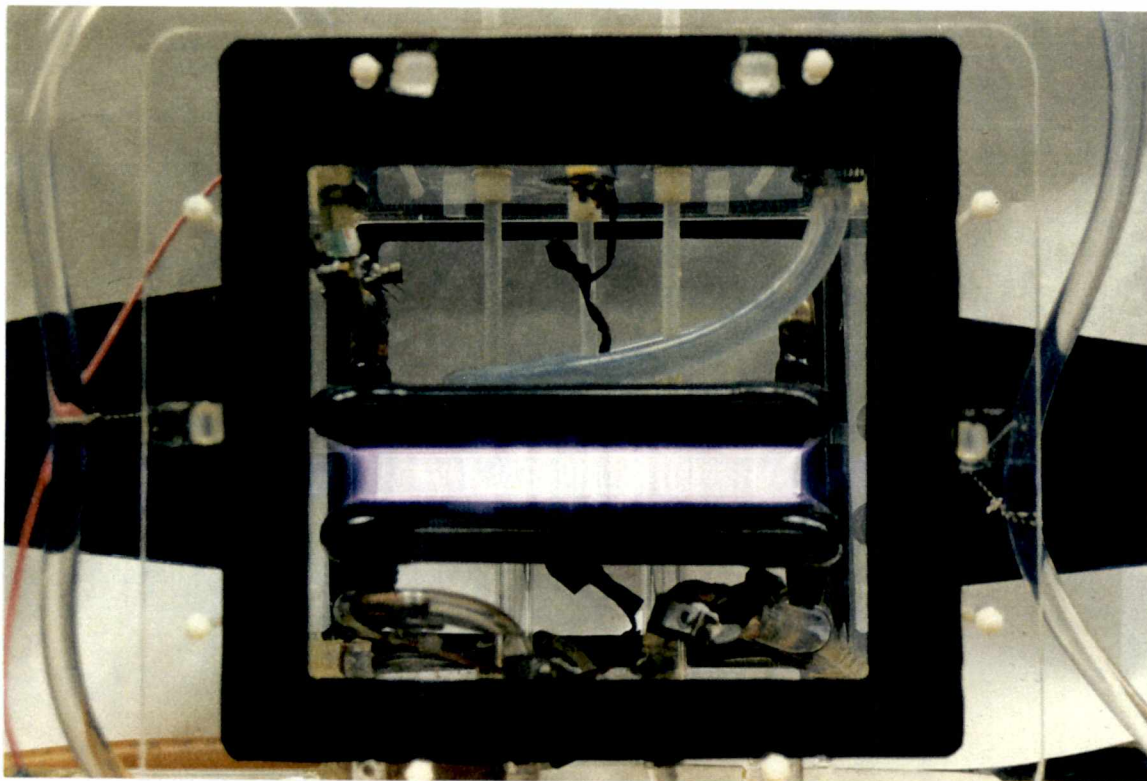


Figure 4.2.6 Appearance of the one atmosphere uniform glow discharge as a function of frequency; the frequency is 40 KHz.

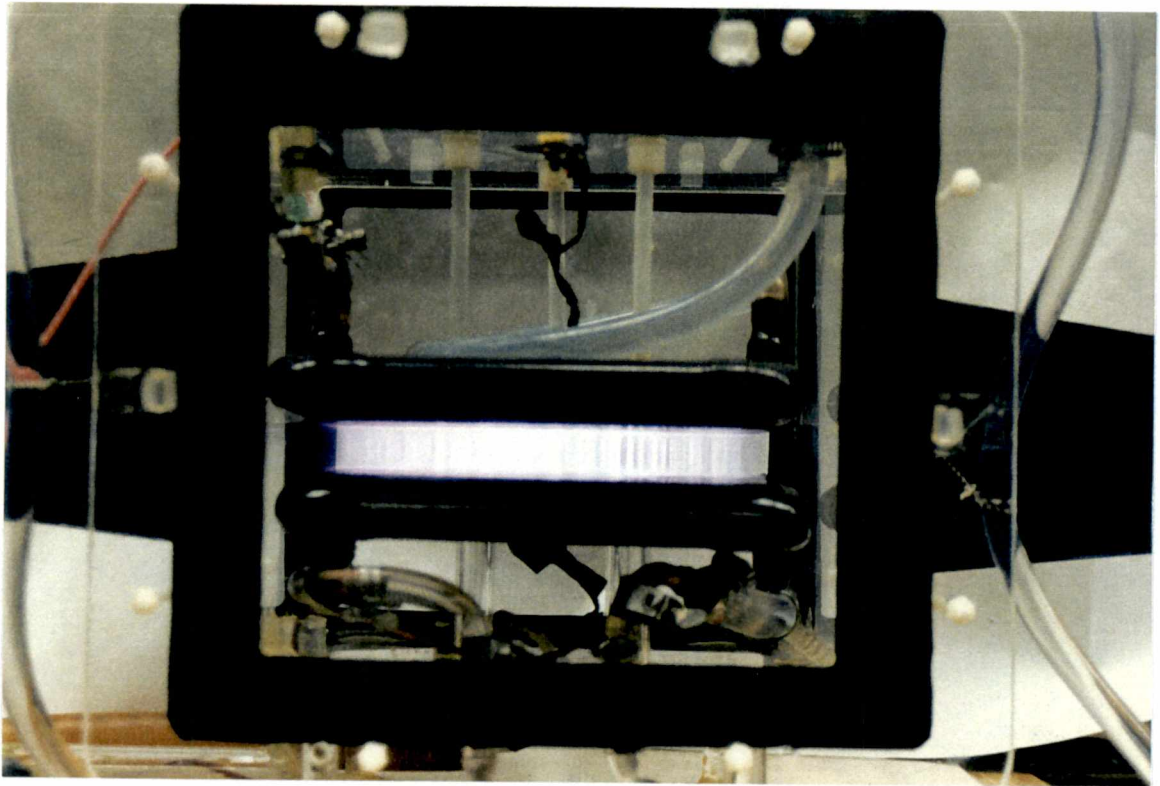


Figure 4.2.7 Appearance of the one atmosphere uniform glow discharge as a function of frequency; the frequency is 60 KHz.

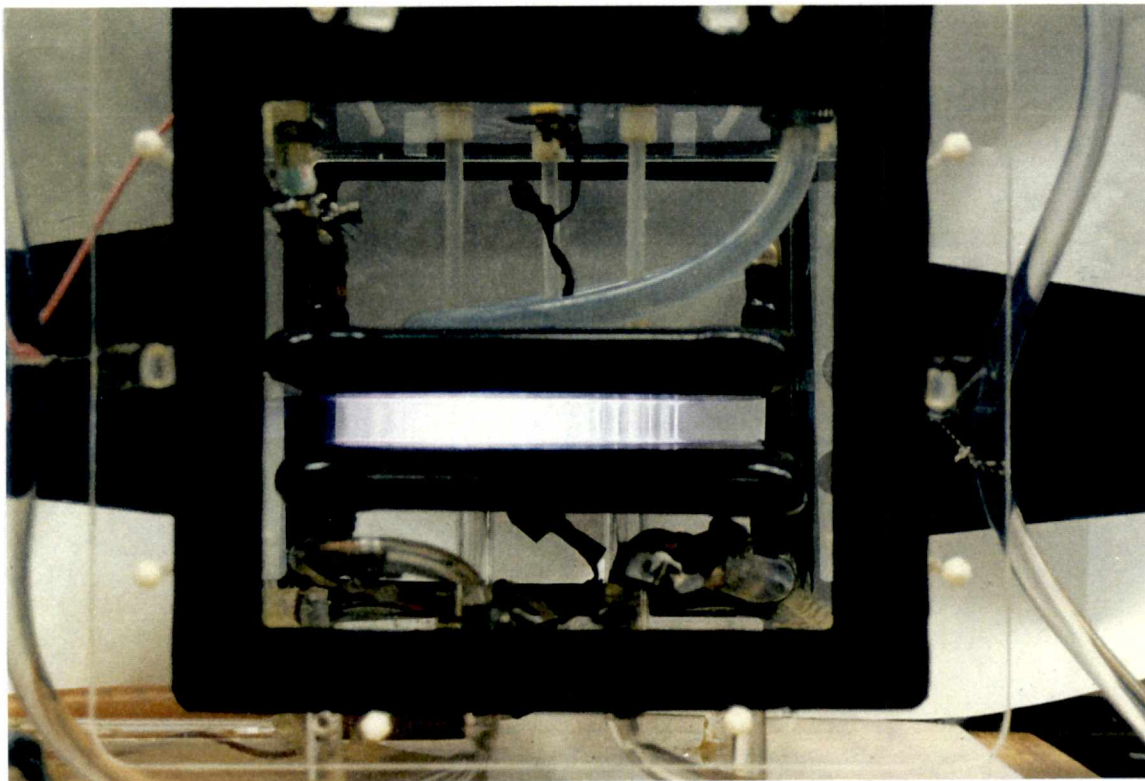


Figure 4.2.8 Appearance of the one atmosphere uniform glow discharge as a function of frequency; the frequency is 80 KHz.

discharge starts which becomes clearer and clearer in figures 4.2.6 to 4.2.8 as the frequency is increased. The conclusion is that in order to get and maintain a uniform glow discharge plasma without a filamentary discharge, the RF frequency must be in the right range to trap ions but not electrons.

Some different geometrical configurations of the one atmosphere plasma generator also were developed during our research. Figure 4.2.9 to figure 4.2.13 shows a group of one atmosphere plasma generators. Some of these may be useful in the future for industrial applications. Figure 4.2.14 is a picture of some of those generators which were made by the author.

Next we introduce a further group of plasma generation experiments which use different geometry electrodes(Ref. 14). In figure 4.2.15, with the configuration of the picture, a uniform glow discharge has been generated between a glass covered tube and a copper plate. The working gas is helium and the gap between the tube and plate is about 1.5 cm. The voltage is about 1.6 KV and the RF frequency is about 2.5 KHz. On figure 4.2.16, two parallel glass covered tube electrodes were tested. A one atmosphere uniform glow discharge plasma has been created between them. The working gas is helium, with RF frequency 2.5 KHz, and RMS electrode voltage of 1.6 KV. Figure 4.2.17 shows that a free-standing one atmosphere uniform glow discharge plasma has been created. The experimental set-up is shown on the picture on figure 4.2.17. The plasma was generated with helium gas as the working gas, the RF frequency equals 2.5 KHz, and the RMS electrode voltage is 1.0 KV, with the base electrically grounded.

During the past several years, we developed several different configurations of the one atmosphere pressure uniform glow discharge plasma generator. We produced a plasma on all the generators we developed. The

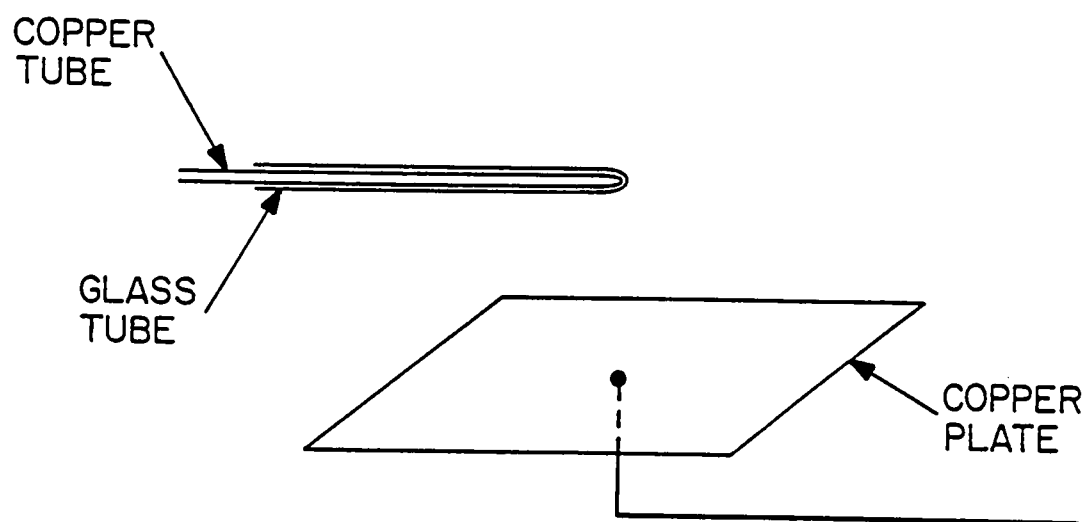


Figure 4.2.9 Single copper tube with a copper plate reactor.

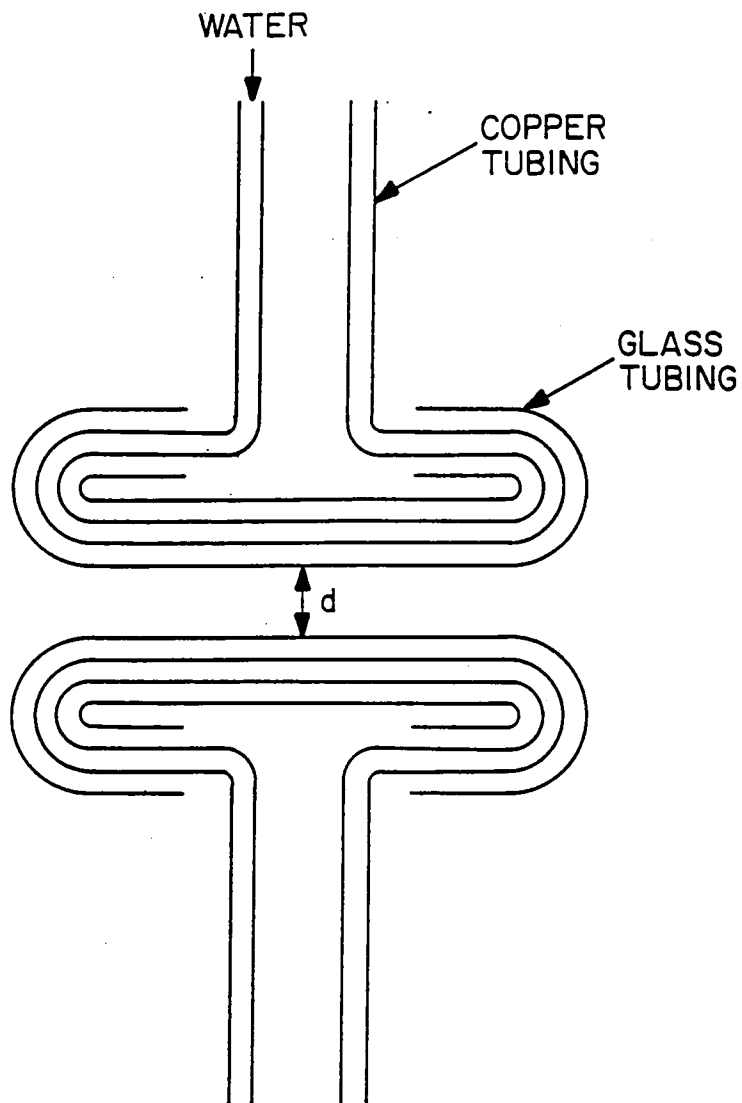


Figure 4.2.10 Two parallel copper tube plasma reactor.

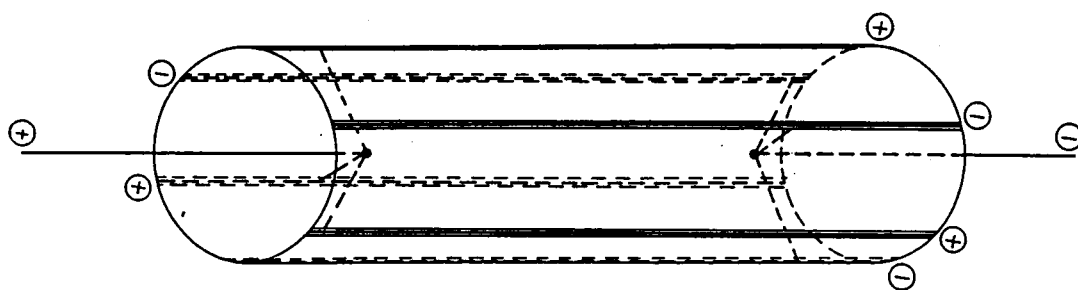


Figure 4.2.11 A free-standing one atmosphere uniform atmosphere plasma generator.

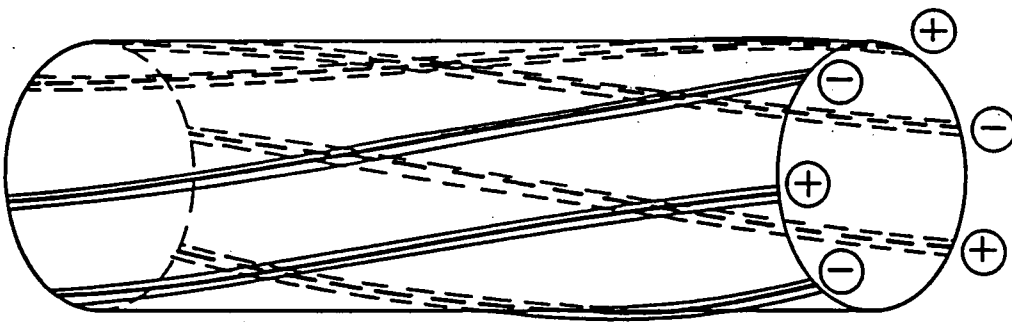


Figure 4.2.12 A free-standing one atmosphere uniform atmosphere plasma generator.

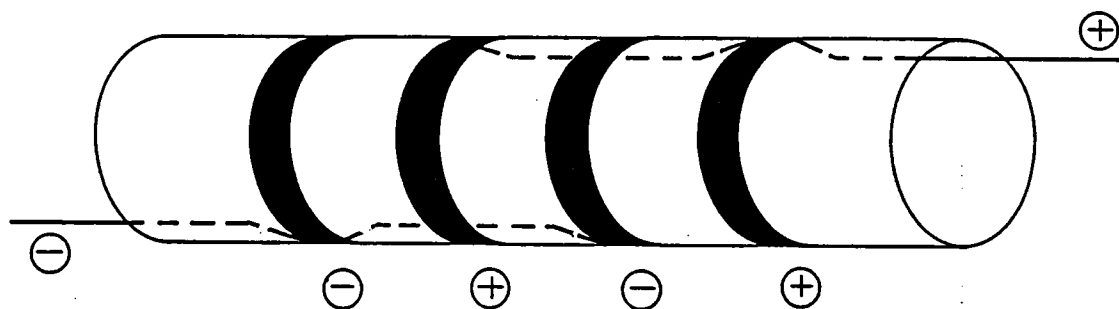


Figure 4.2.13 A free-standing one atmosphere uniform atmosphere plasma generator.

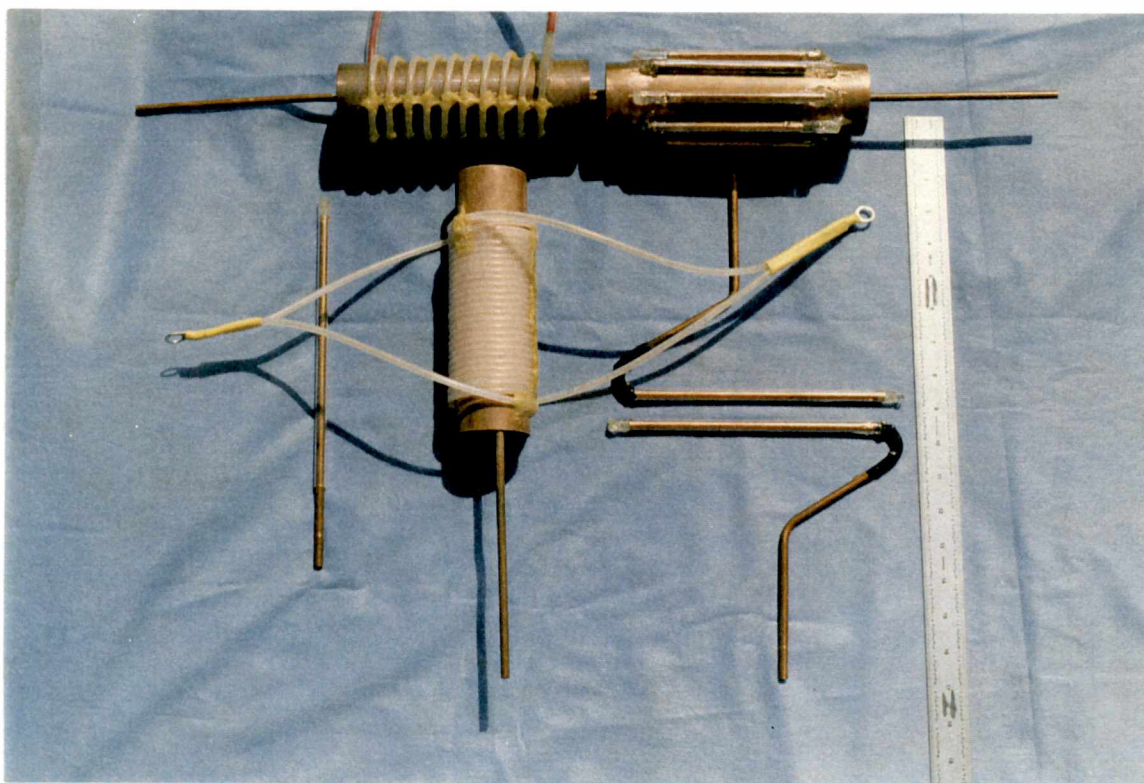


Figure 4.2.14 A group of plasma generators with different geometry.

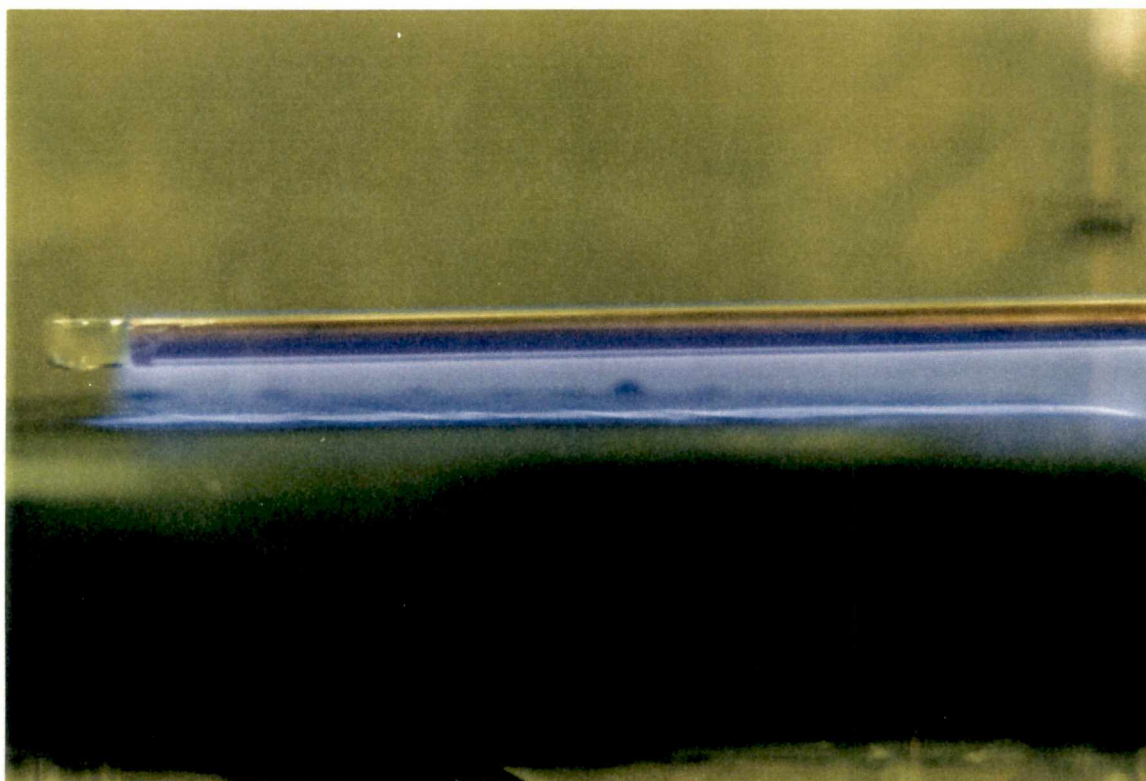


Figure 4.2.15 A Copper tube with a copper plate plasma generator set up and operating.

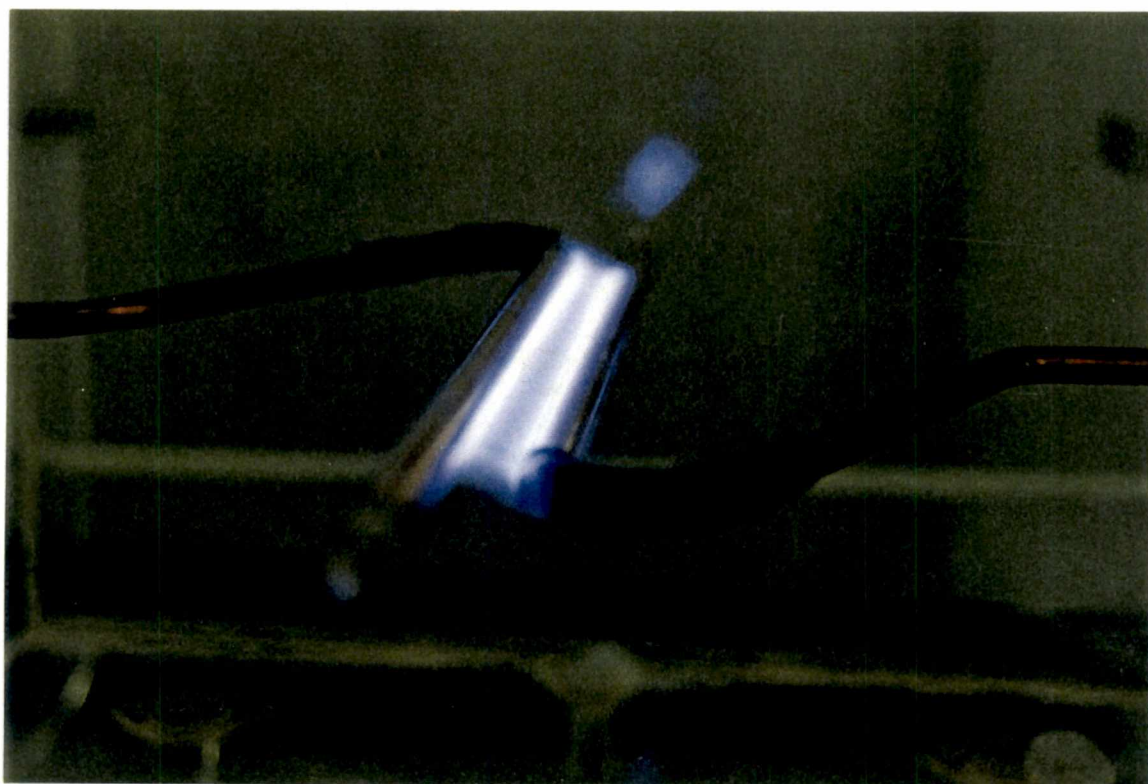


Figure 4.2.16 Two parallel copper tubes plasma generator set up and operating.

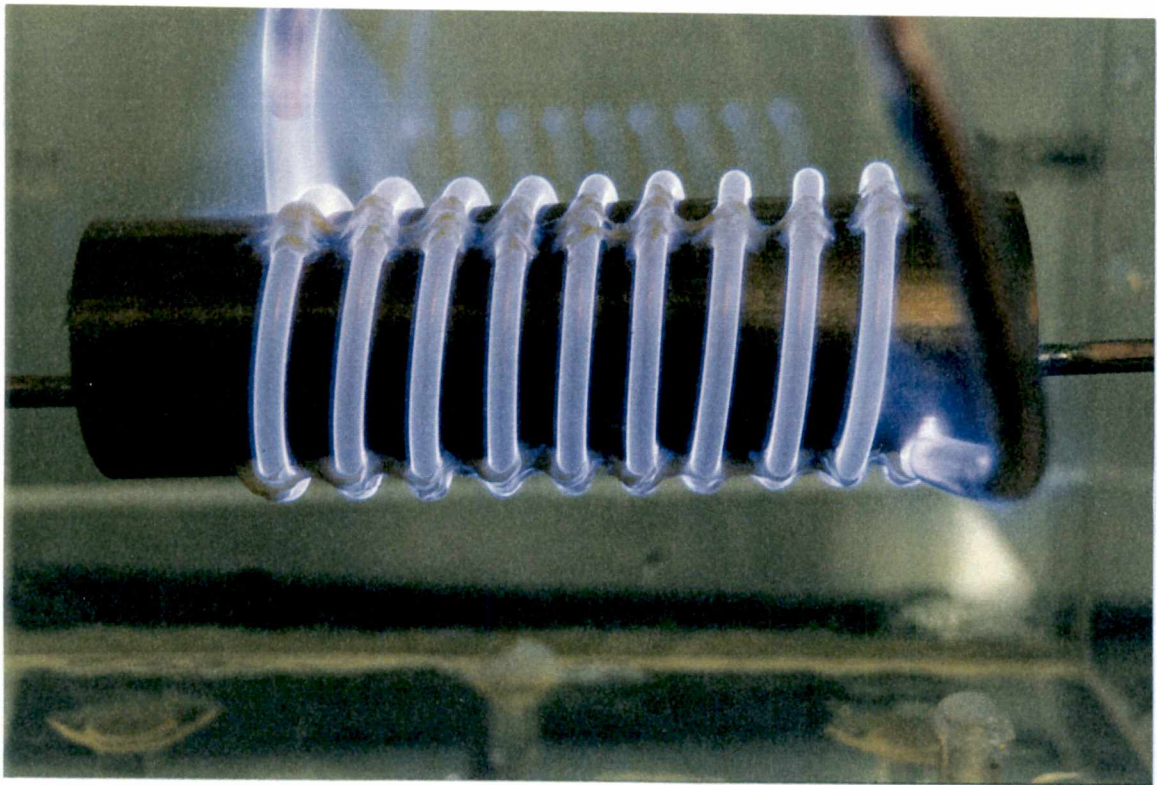


Figure 4.2.17 A free-standing one atmosphere uniform glow discharge plasma generator set up and operating.

plasma can be generated in several working gases including air. These plasmas and the generator should have industrial application potential.

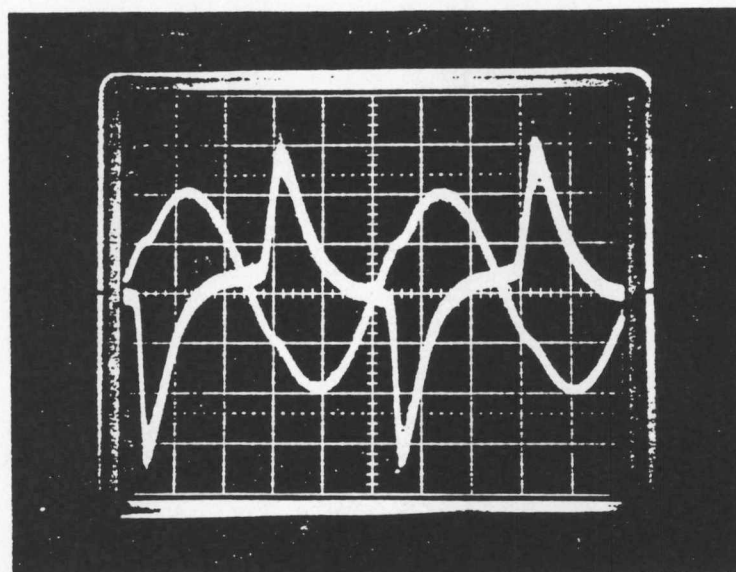
4.3 Plasma-Related Characteristics of a Steady-State, Uniform Glow Discharge at Atmospheric Pressure

We generated a steady-state glow discharge plasma at atmospheric pressure in a parallel plate plasma reactor and some other capacitive reactor configurations. In this section we will discuss the measurement of plasma-related characteristics on a parallel plate plasma reactor.

The most basic measurement we did was to measure the waveforms of the electrode voltage and input current, using a high voltage probe and current probe connected to a digital oscilloscope. Figure 4.3.1 shows the waveform of input voltage and current taken during plasma operation. It is clear to see from this picture that there is a phase angle between the maximum of the voltage waveform and current. In a typical example of plasma operation, the plasma is operated at one atmosphere pressure, helium is the working gas; the electrode separation distance is about 1.0 cm; and the applied voltage on the electrodes is held constant. The RF frequency was the independent variable, and the phase angle between the I-V waveform(peak to peak) was measured. Table 4.3.1(a) reports the corresponding phase angle θ of breakdown current with the RF frequency. Table 4.3.2(a) shows the input power as a function of frequency. This phase angle increased with frequency. We call it the breakdown phase angle(Ref. 4. and Ref. 15.). Figure 4.3.2 shows a graph of the phase angle as a function of frequency.

Taking the same operating conditions as the example we used above , we held the RF frequency constant at 30 KHz, and changed the RMS electrode voltage V. We measured the corresponding relationship between

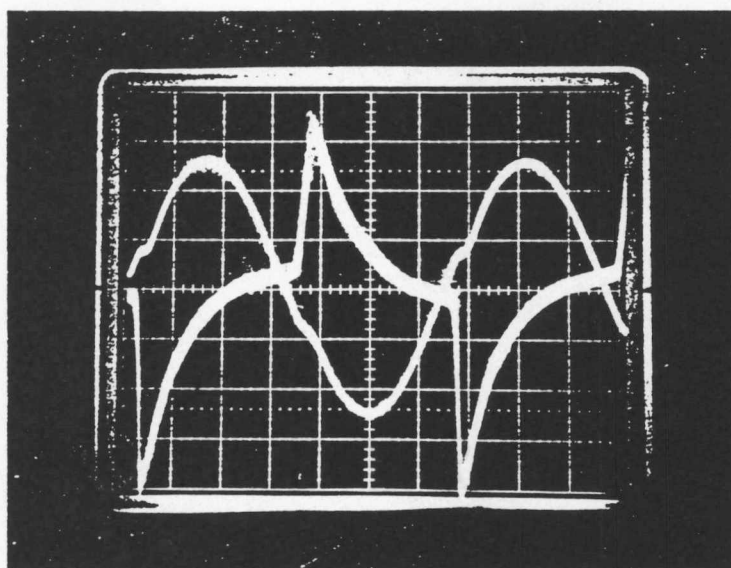
2000V/DIV
25mA/DIV



4 KHZ

20us/DIV

2000V/DIV
40mA/DIV



8 KHZ

20us/DIV

Figure 4.3.1 The waveform of the input voltage and current.

TABLE 4.3.1

a. Phase angle θ as a function of voltage V.

f(KHz)	10	20	30	40	50	60	70	80	90	100
θ (deg)	43	32	43	52	54	61	60	56	45	22.5

b. Phase angle θ as a function of RF frequency f.

V(KV)	1	1.5	2	2.5	3	3.5
θ (deg)	28	40	61	46	65	76.5

TABLE 4.3.2

a. Power input P as a function of RF frequency f.

f(KHz)	10	20	30	40	50	60	70	80	90	100
P(W)	5	8	11	19	35	43	47	57	89	124

b. Power input P as a function of input voltage V.

V(KV)	1	1.5	2	2.5	3	3.5
P(W)	7	13	22	57	50	44.9

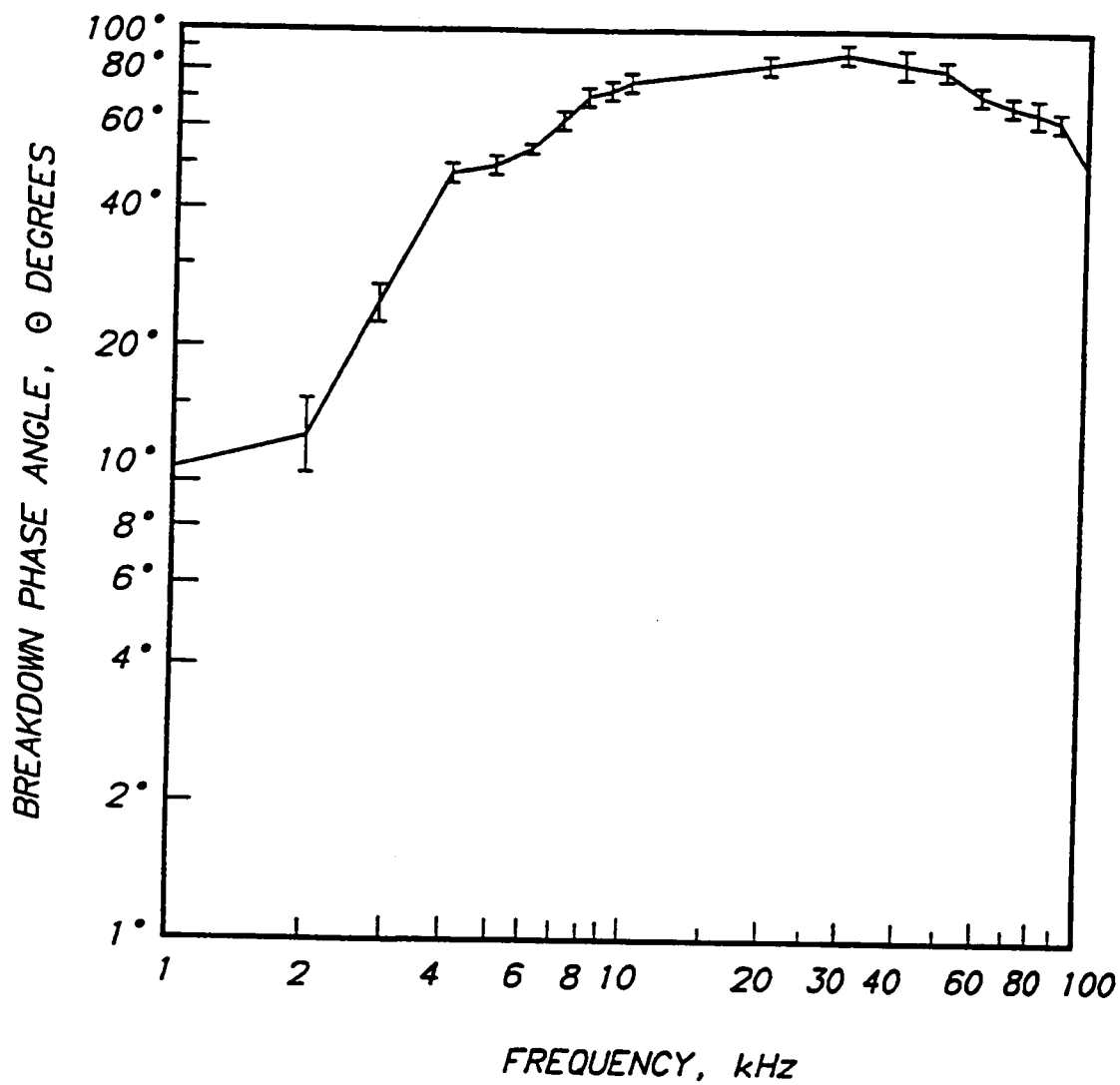


Figure 4.3.2 The phase angle as a function of frequency.

the input potential with the breakdown phase angle, θ , and the power input, W . Table 4.3.1(b) and figure 4.3.3 show our results.

Some conclusions based on our experiments are: the phase angle between the maximum of the voltage waveform and current breakdown increased with frequency; the reactive power density of the glow discharge was approximately several ten to about one hundred mW/cm^3 the actual plasma power densities which were well below this level were good for material surface treatment without damaging the surface.

The plasma floating potentials also were measured during our experimental measurements(Ref. 13). For the operating conditions of RMS electrode voltage $V_{rms} = 3KV$, an electrode separation of $d=2cm$, one atmosphere pressure, and helium working gas, measurements of floating potential were made with a floating potential probe. The floating potential probe was made with tungsten wire (about $\varnothing 0.5mm$) covered by a glass tube and located in the center of the plasma. The experimental set up is shown on figure 4.3.4. Table 4.3.3 shows measurements of the floating potential as a function of frequency. Table 4.3.4 shows the measurements of the floating potential as a function of RF input voltage.

For plasma diagnostics in low pressure plasmas, the diagnostic equipment has been researched and developed for many years. Microwave interferometers, Langmuir probes, and other diagnostic devices for low pressure plasma measurement are off the shelf (Ref. 16). The atmospheric plasma is a new type of plasma. Because it operates at high pressure, traditional plasma diagnostic techniques are not appropriate because of the short electron mean free path and high collision frequency. To develop a new type of plasma diagnostic device in order to measure the atmospheric plasma

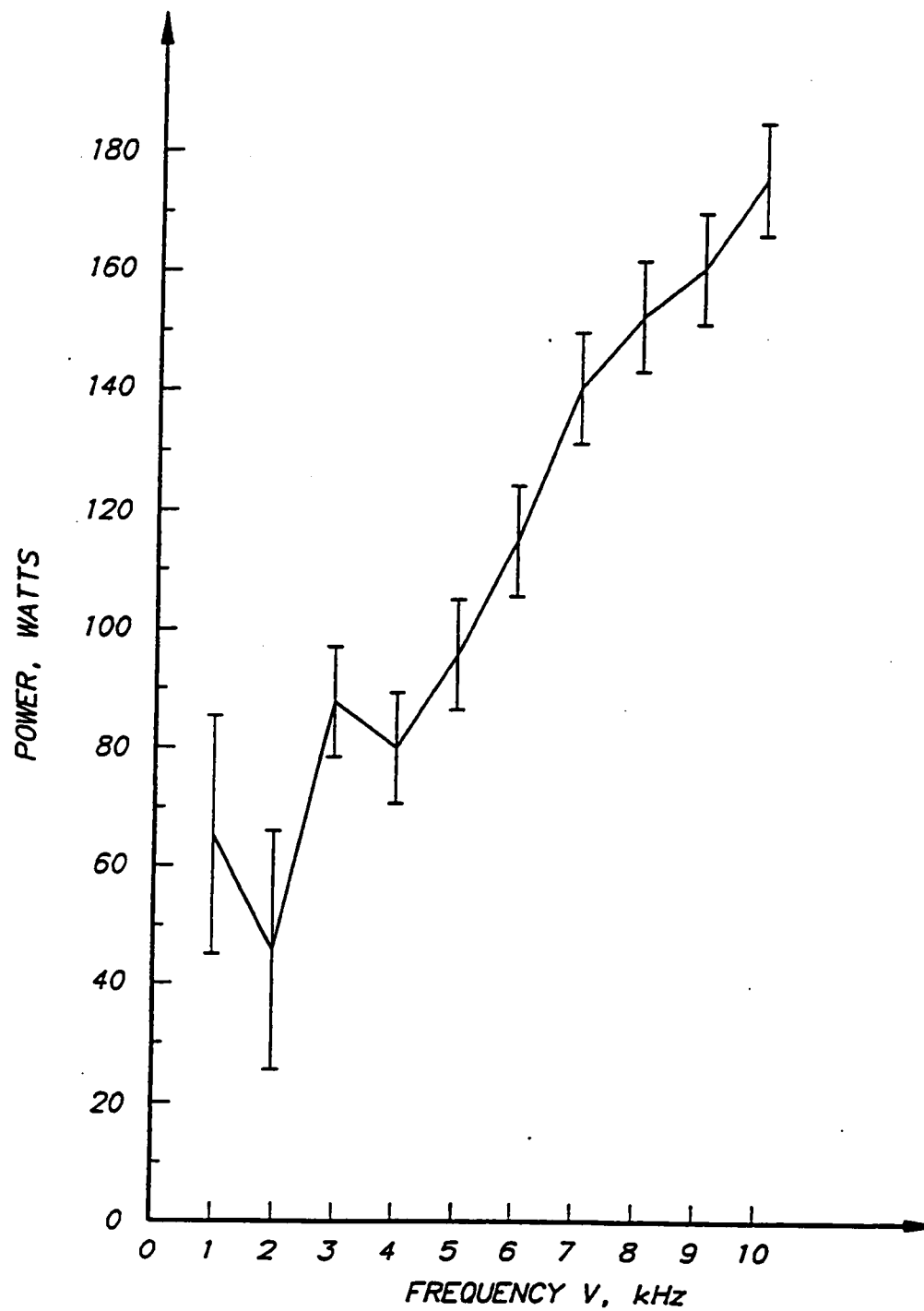


Figure 4.3.3 The total, reactive power input as a function of frequency.

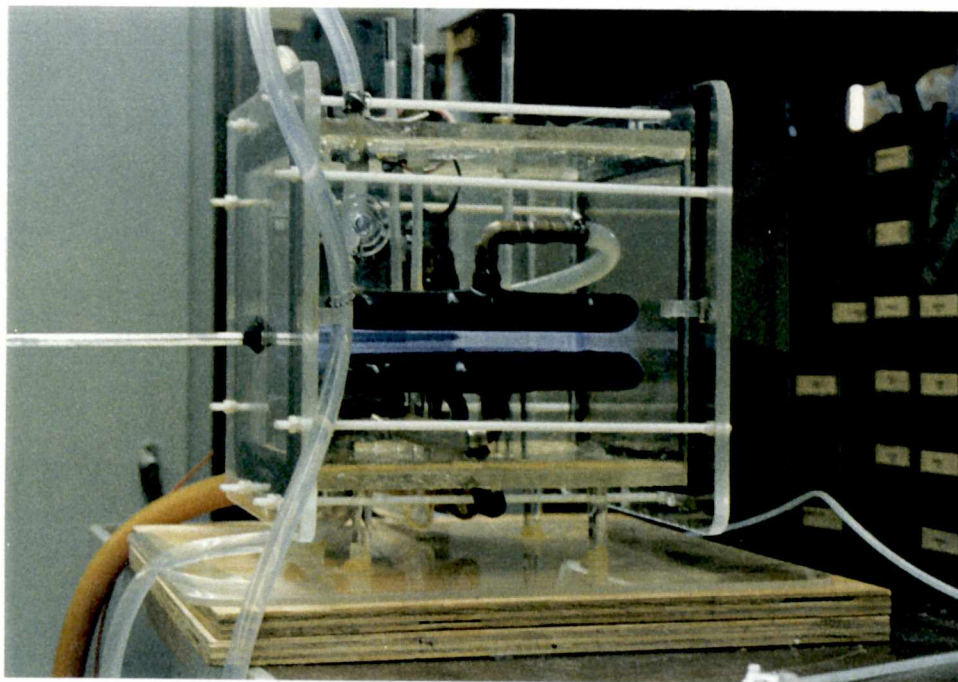


Figure 4.3.4 The experimental setup for the plasma floating potential measurements.

Table 4.3.3 Steady-state glow discharge plasma floating potential is measured as a function of frequency.

ν (KHz)	1	2	3	4	5	6	7	8	9	10
V_p (V)	89.9	110.4	108	108.3	123.6	133.9	132.6	133.7	131.1	94.1

ν (KHz)	11	12	13	14	15	16	17	18	19	20
V_p (V)	106.3	120.9	125.5	124.7	133.5	132.7	132.7	136.9	140.2	131.1

Error: 15%

Table 4.3.4 Steady-state glow discharge plasma floating potential is measured as a function of the RF input voltage.

V_{rms}	1.5	2	2.5	3	3.5	4	4.5	5
V_p (V)	73.6	79.7	109.7	133.1	192.5	241.8	299.8	367.1

Error: 15%

number density, kinetic temperature, etc. is a topic for future research.

5. APPLICATIONS OF THE ONE ATMOSPHERE UNIFORM GLOW DISCHARGE PLASMA

5.1 Introduction

It is possible that one atmosphere uniform glow discharge plasmas will be of great value in industrial applications. They can operate at one atmosphere pressure, which would allow many plasma-related surface modification processes to be carried out under production line conditions. Glow discharge plasmas have a low power density which is useful for non-destructive surface modification of materials. These glow discharge plasmas can produce useful amounts of ultraviolet radiation in the simultaneous presence of active species. They have been used to treat nonwoven textiles, meltblown webs, polymer films and other materials during our research. The wettability and wickability of treated textiles or webs have been permanently improved(Ref. 13 and Ref. 18), and the printability of polymer film has been improved(Ref. 19).

A new type of lighting source based on the one atmosphere uniform glow discharge plasma may bring longer lifetime, lower environmental impact, higher efficiency, greater safety and other advantages(Ref. 20).

The applications to plasma cloaking of ships and aircraft by using a layer of the one atmosphere uniform glow discharge plasma have been studied(Ref. 21). Because this plasma can operate at one atmosphere in air, it may make possible plasma cloaking of aircraft.

An entirely new area of research on drag reduction and aerodynamic boundary layer control on aircraft by using our one atmosphere glow discharge plasma has been proposed and initially studied. This research may have an important impact on aircraft design and operation in the future, particularly in reducing skin friction drag.

In this chapter, we will describe most of the application research related to the one atmosphere RF uniform glow discharge plasma which was carried out by the author at the U.T.K Plasma Science Laboratory. Several illustrative figures and pictures of the design, experimental setups, and experiment for all results will be shown. Some of the pictures are the first time published.

5.2 Treatment of Nonwoven Textiles by a Uniform Glow

Discharge Plasma at One Atmosphere

Plasma treatment was introduced in the textile industry about 10 years ago. It has been used to improve the quality of printing on textiles, and to improve the wettability, wickability, and printability of polymer fabrics by using active species from a plasma discharge to modify the treated textile surface. The active species are directly or indirectly from electron-neutral collisions and subsequent chemical reactions in the plasma(Ref. 23). Plasma generated active species are almost without exception produced in larger numbers than the same species would be by purely chemical reactions in a chemical reactor. These active species make it possible to do things to the surface of materials which can be done in no other way, or which are economically impracticable by other methods. Active species can be produced by corona or glow discharges, which have power densities that range from below 10^{-4} to slightly over 1 watt per cubic centimeter.

A definition of plasma surface treatment is the use of active species from a plasma to modify the normal surface characteristics of materials. Plasma surface treatment does or may remove adsorbed monolayers; it may add adsorbed monolayers; it may add or remove molecular fragments to or from the surface molecules of the material; plasma surface treatment may add or remove surface charge, and it may also otherwise change the chemical or physical state of the surface monolayers of a material(Ref. 23).

Currently, most plasma treatments of textiles are carried out by either corona discharges or low pressure glow discharges. Figure 5.2.1 shows an example of a low pressure glow discharge plasma surface treatment for wool(Ref. 22). In this example, wool treatment using a low pressure glow discharge plasma was introduced. Because the plasma is operated at low pressure, the process is relatively difficult and expensive, although superior to the conventional "chlorination" process(Ref. 22).

Our steady-state uniform glow discharge plasma is capable of operating at one atmosphere, which potentially allows the textile treatment processes to be carried out under production line conditions. Figure 5.2.2 shows an experimental design for one atmosphere textile treatment. As an example of many experimental sample testing runs we did, the PET(Poly ethyleneterephthalate), PP(PolyPropylene), Nylon and LLDPE(Linear Low Density PolyEthylene) polymer treatment will be used as examples(Ref. 13). This experiment was done with the cooperation of Dr. P. P-Y. Tsai, an Assistant Research Professor from TANDEC(Textiles and Nonwovens Development Center, College of Human Ecology, University of Tennessee, Knoxville). The experimental setup and process are shown in figure 5.2.3. These sample testing runs were done in the MOD-I reactor. Figure 5.2.3

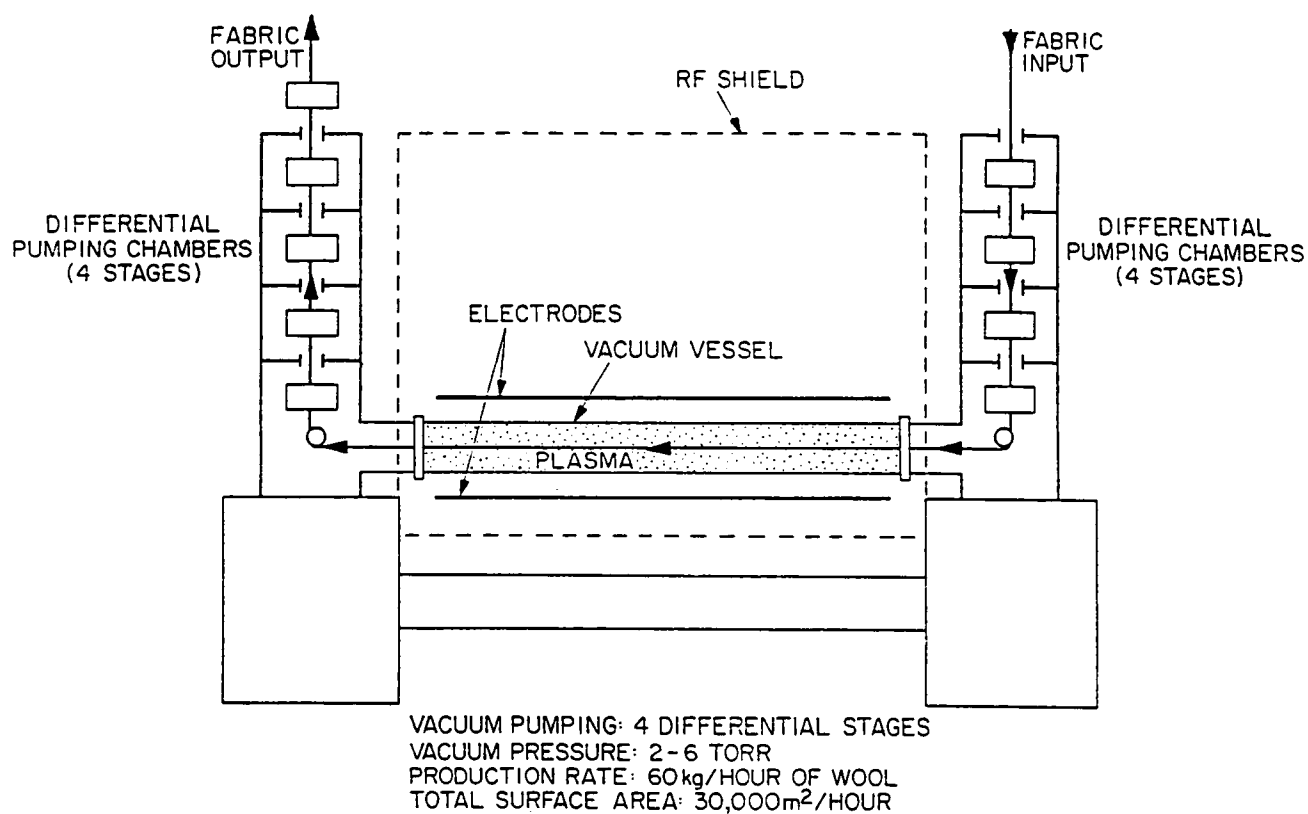


Figure 5.2.1 Apparatus for low pressure plasma treatment of wool(Ref. 22).

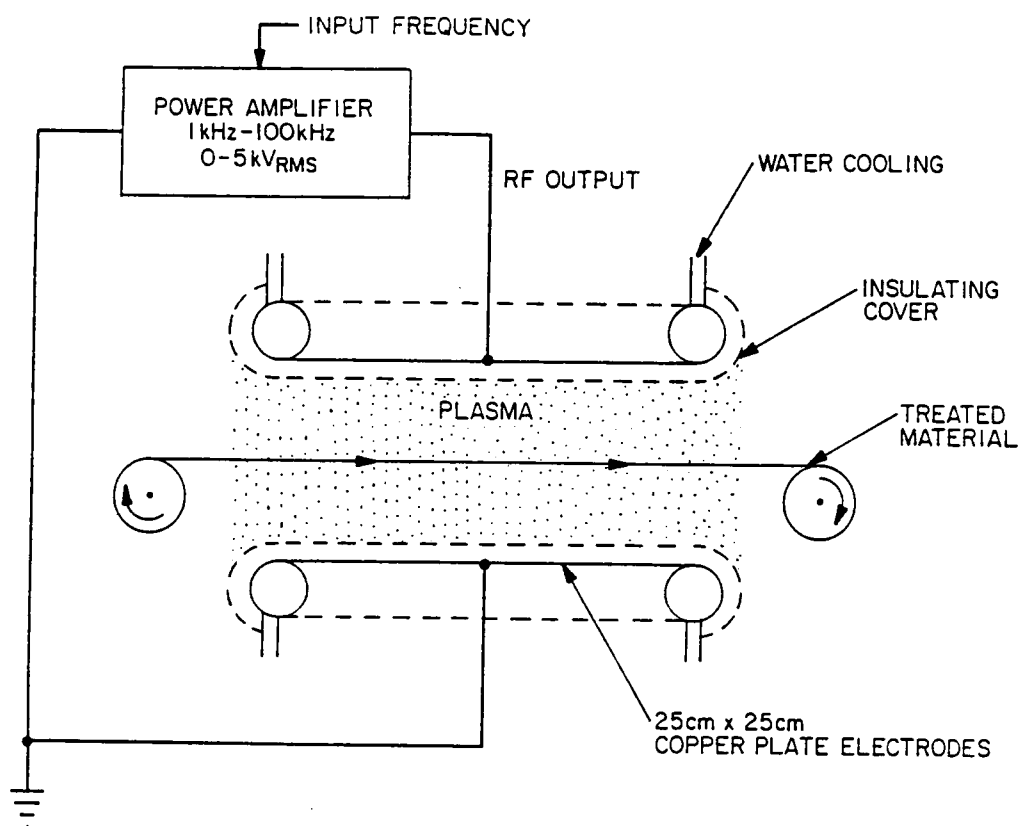


Figure 5.2.2 An experimental design for one atmosphere plasma treatment of textiles.

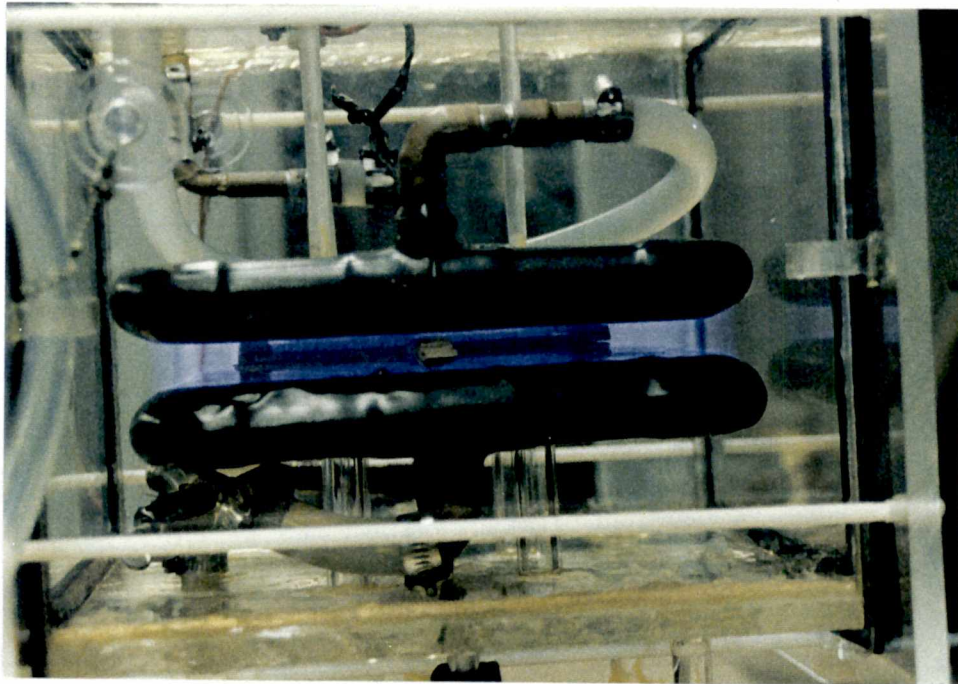


Figure 5.2.3 An experiment setup and process for plasma treatment of textiles.

shows how the test sample was held by a sample holder located in the center of the plasma. The working gas used for this group of polymers was helium. The RF frequency was 10KHz, the RF voltage was between 2-3KV RMS, and the electrode plate separation was about 1.5cm. The treatment time was between 30 sec to 3 min.. Table 5.2.1 gives the treated and untreated meltblown web properties. After treatment, we examined the treated meltblown web wettability by measuring the contact angle and wicking rate.

The important surface tension related characteristics include(Ref. 23) the **wettability**, one of the most important surface tension related characteristics. It is the ability to adsorb a liquid on a solid surface, or to absorb the liquid in the bulk of fibrous materials such as fabrics. Wettability implies a low contact angle and high wicking rate. **Wickability** is a measure of the bulk absorptivity of porous or fibrous materials, such as fabrics. It is usually defined in terms of the upward speed of the wetted boundary in a vertical sample dipped in the liquid of interest. Alternatively, the wickability can be defined as the distance a wetted boundary travels upwards in a vertical sample in a fixed time. **Contact angle** the angle measured between the horizontal and the liquid surface of a drop of liquid in contact with a material. It is a simple but widely used test for the measurement of surface characteristics.

Returning to the experimental example we discussed above, table 5.2.2 gives the measurements of the treated meltblown web wettability. It is not difficult to see how large the changes are. The wettability and wickability of the treated meltblown webs are greatly improved. Figure 5.2.4 shows the change in water contact angle of untreated and plasma treated polypropylene(Ref. 12), and figure 5.2.5 shows a photograph of treated and

TABLE 5.2.1

Meltblown Web Properties*

Sample	Basis Weight (g/m^2)	Thickness (μm)	Fiber Diameter (μm)	Porosity (%)	Pore Size (μm)
Nylon	37.1	334	4.13	90	41.90
PET	31.1	281	2.43	92	31.61
PP	26.0	291	1.95	90	22.08
LLDPE	52.0	469	5.52	88	42.60

* See Reference 13.

TABLE 5.2.2**Treated Meltblown Web Wettability***

Sample	Duration of Treatment (minutes)	Contact Angle Control (degree)	Contact Angle Treated (degree)	Wicking Rate* Control (seconds)	Wicking Rate** Treated (seconds)
Nylon	1	high	0	∞	16.61
PET	2.5	high	0	∞	37.37
PP	2.5	high	greatly reduced	∞	+
LLDPE	2.5	high	greatly reduced	∞	+

*See Reference 13.

** Experiment done according to INDE Standard Test.

+ Has not been measured.

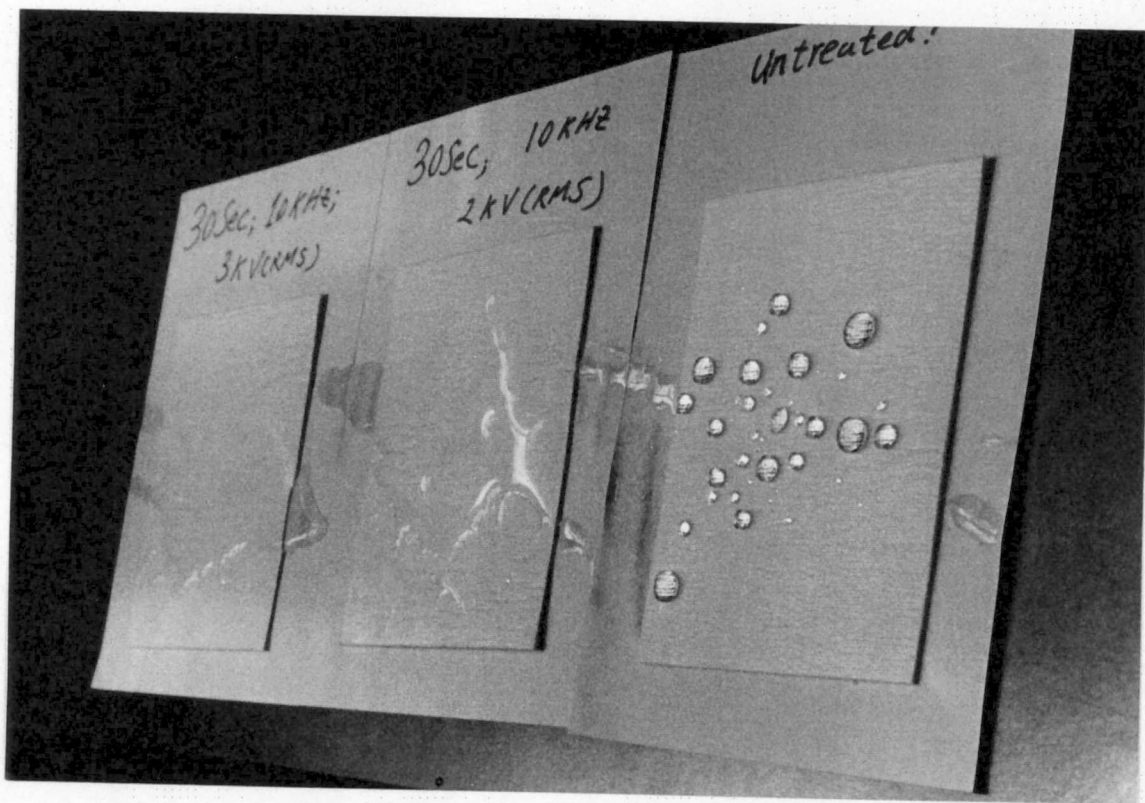


Figure 5.2.4 Photograph of water contact angle of untreated and plasma treated polypropylene.

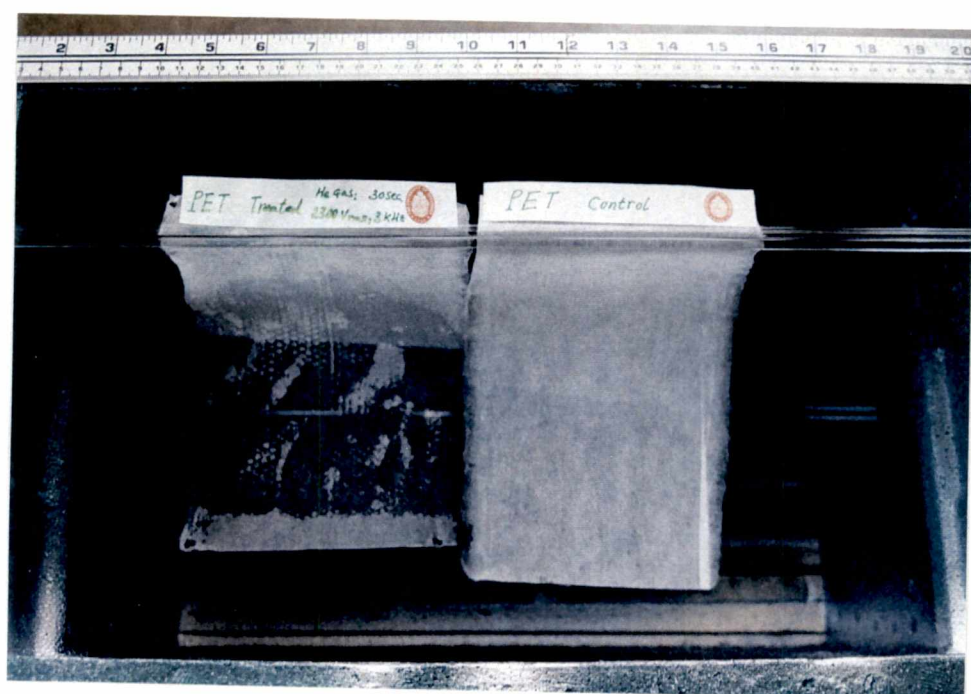


Figure 5.2.5 Photograph of wettability of untreated and plasma treated PET.

untreated samples of PET illustrating the improvement in wettability of the treated sample brought into contact with the surface of a container of water. The untreated sample is floating on the water at the right. The treated sample was exposed to a helium one atmosphere uniform glow discharge plasma for 30 sec. at an RMS voltage of 2.3KV, a RF frequency of 3KHz, and a plate separation of 1.27cm. Changes of the properties of the tested textile materials are directly related to the characteristics of the plasma parameters and the exposure time.

The one atmosphere uniform glow discharge plasma in industrial textile treatment applications has a very good future. It is safer, cheaper, and convenient. More research appears to be warranted.

5.3 Research on a New Lighting Source using the One

Atmosphere Uniform Glow Discharge Plasma for Illumination

Plasma used as a lighting source has a long history. One lighting source is the fluorescent light tube, which uses plasma generated active species which radiate ultraviolet. These photons excite fluorescence of phosphors to produce visible light. Another type of lighting source uses directly the plasma luminosity of arcs. This luminosity is produced by the plasma when the electron energy and number density are high enough to generate visible light by excitation collisions. Such plasma lighting sources as the above are operated at low pressure (about 1 Torr). In order to produce higher fluxes of ultraviolet radiation to excite the phosphors, mercury is normally used.

The one atmosphere uniform glow discharge plasma is visible. It produces a large flux of photons at visible wavelengths. Also, because it operates at relatively high pressure, the active species density produced by this plasma is high and the flux of ultraviolet radiation may be higher. We

tested a new method of illumination, which uses a surface layer of steady-state uniform glow discharge plasma to excite fluorescence on a layer of phosphor below the plasma layer. It may have advantages compared to existing plasma light sources. Research on this new lighting source has been done, using our one atmosphere uniform glow discharge plasma. We report some exploratory results below.

Figure 5.3.1 shows a free-standing plasma generator which was used as the plasma generator for a lighting source(Ref. 3). We coated green and blue phosphors on the outer cylindrical surface of the generator. The phosphors we used in the test were #2461 blue and #2211 green supplied by Dr. Gerald L. Rogoff, Engineering Manager, GTE products Corp.; and Dr. Barry G. Deboer of Osram Sylvania Inc., Towanda, PA. They are acknowledged here. Figure 5.3.2 shows the experimental arrangement. The phosphor used here is #2211 green, and the RF frequency is 6KHz, and the RF electrode voltage is 2KV RMS. The plasma excited fluorescence is on the surface layer of phosphor below the plasma layer.

There are several advantages of this one atmosphere uniform glow discharge plasma lighting source. It is safer and causes less environmental impact than lighting devices which require mercury, and must be operated at low pressure. It probably would have a much longer lifetime. Additional research should be carried out, if funding sources can be identified.

5.4 Plasma Cloaking of Aircraft in the Atmosphere

Plasma cloaking is a technology which can protect aircraft or space vehicles from directed microwave energy weapons and/or radar detection. This technology is called plasma cloaking because it uses a plasma generated on or around the surface of a target to absorb microwave radiation. Plasma

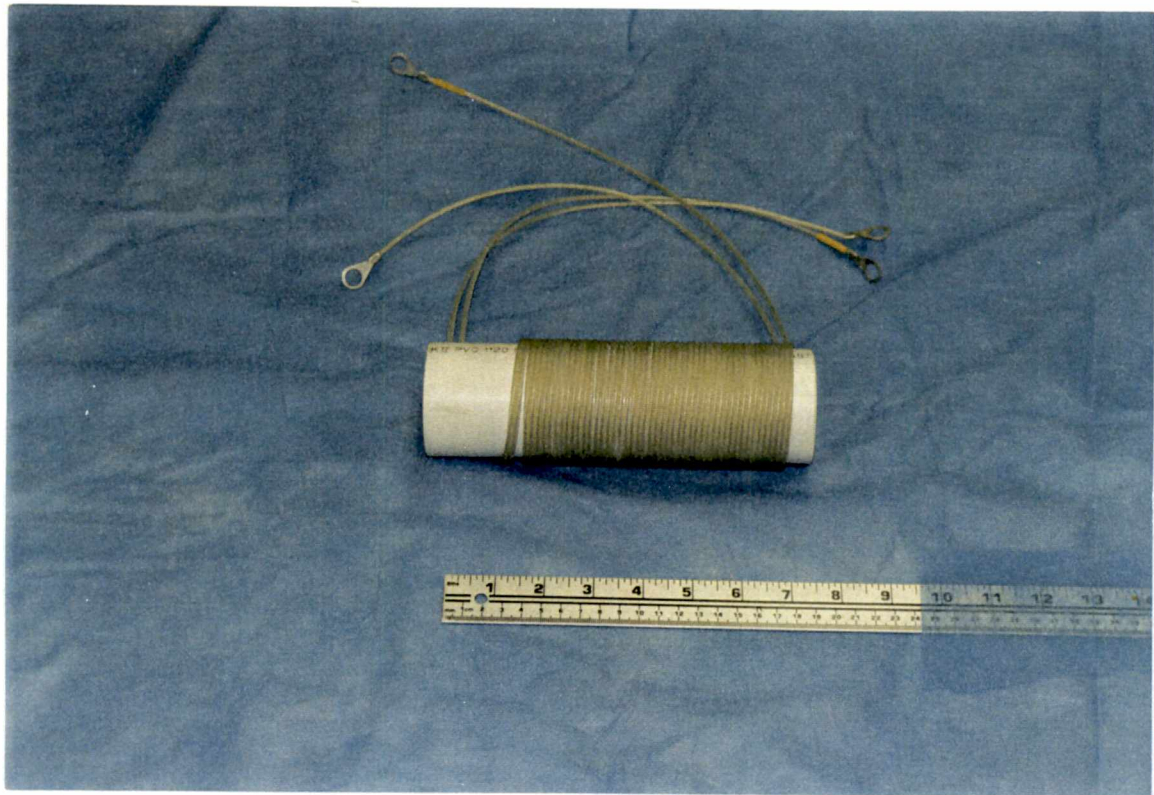


Figure 5.3.1 The surface layer plasma generator on a 5cm diameter plastic cylinder.

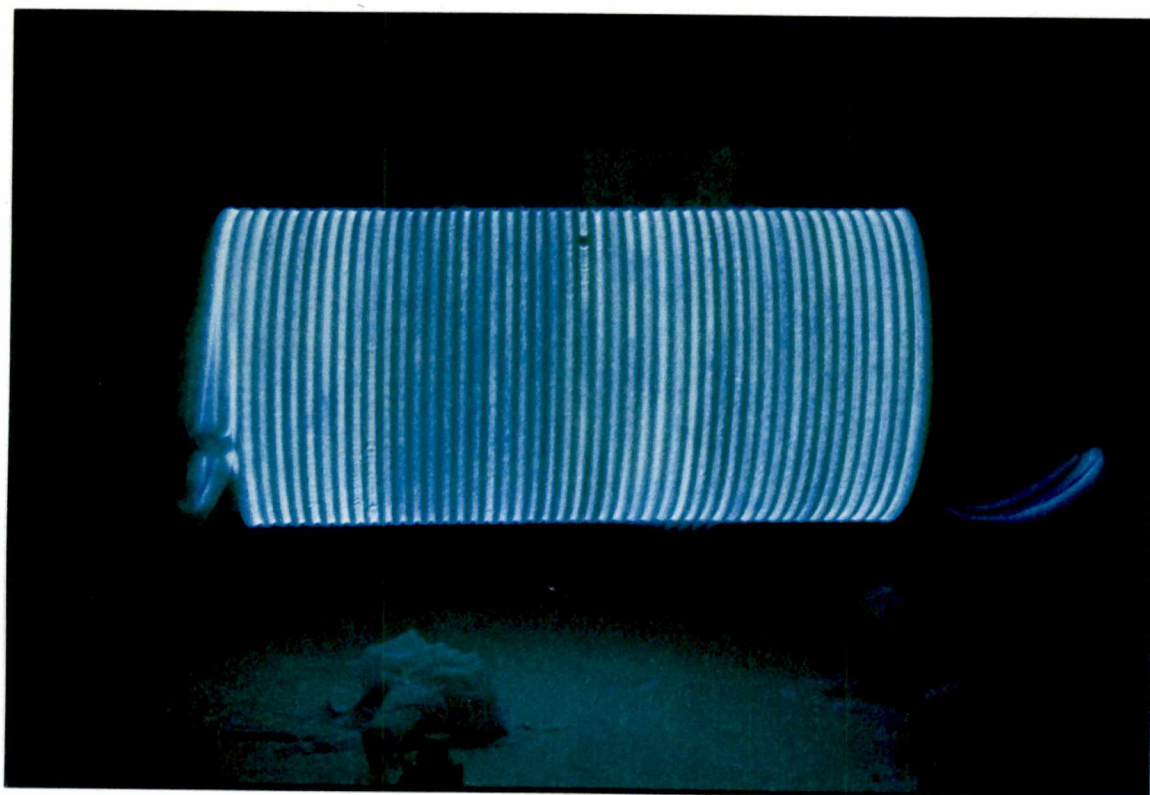


Figure 5.3.2 Photograph of one atmosphere uniform glow discharge plasma lighting source experiment, taken with an RF frequency 6KHz, RF electrode voltage of 2KV RMS.

cloaking research has been underway at the UTK Plasma Science Laboratory for many years(Ref. 24). One of the major problems for the application of plasma cloaking is how to create a plasma layer at atmospheric pressure. Now, with our one atmosphere uniform glow discharge plasma generation, we have a solution.

Figure 5.4.1 shows the first design of a simulated aircraft fuselage. The experimental setup was shown on figure 5.4.2. In this example, the plasma created on the simulated aircraft fuselage surface was operated with a working gas of air mixed with helium; the RF frequency was 3 KHz; the electrode power supply RMS voltage was 2KV. From the picture on figure 5.4.3, it is evident that a thin layer of plasma was created on the surface of the simulated fuselage. Figure 5.4.4 shows the plasma operated at the same power supply condition but with pure air.

To meet the requirements of the plasma cloaking application, there still is a long way to go. It is encouraging however, that the one atmosphere uniform glow discharge has been successfully generated on the surface of a simulated fuselage in the laboratory.

5.5 A one Atmosphere Uniform Glow Discharge for Fluid Mechanical Control Using a Plasma Surface Layer

Fluid mechanical boundary layer control is a major concern in the design of an aircraft. If a surface plasma can be applied to the fluid mechanical boundary layer control of an aircraft, it may bring many advantages for the aircraft, including reduction of vorticity, skin friction drag, and aircraft turbulent noise production.

Our uniform glow discharge which can be operated at atmospheric pressure may make this possible. The parallel plate geometry plasma

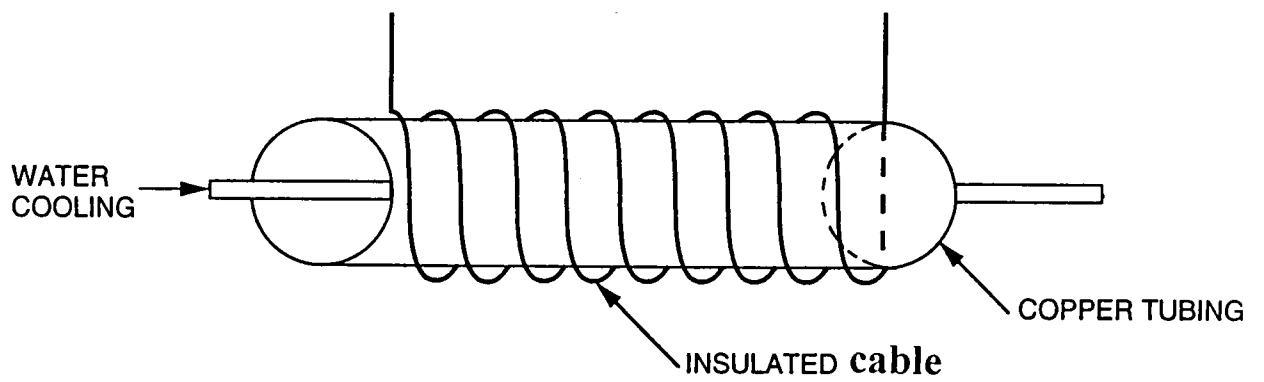


Figure 5.4.1 The first design of a simulated aircraft fuselage.

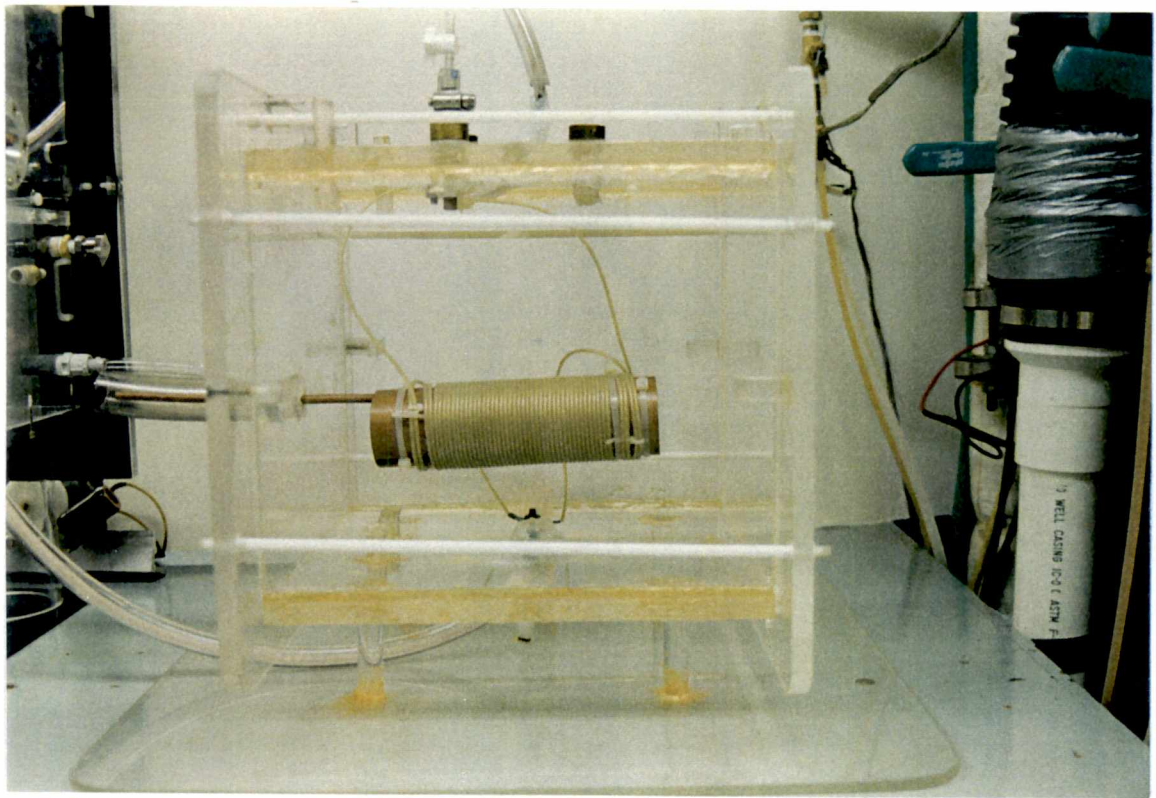


Figure 5.4.2 Plasma cloaking experimental setup.

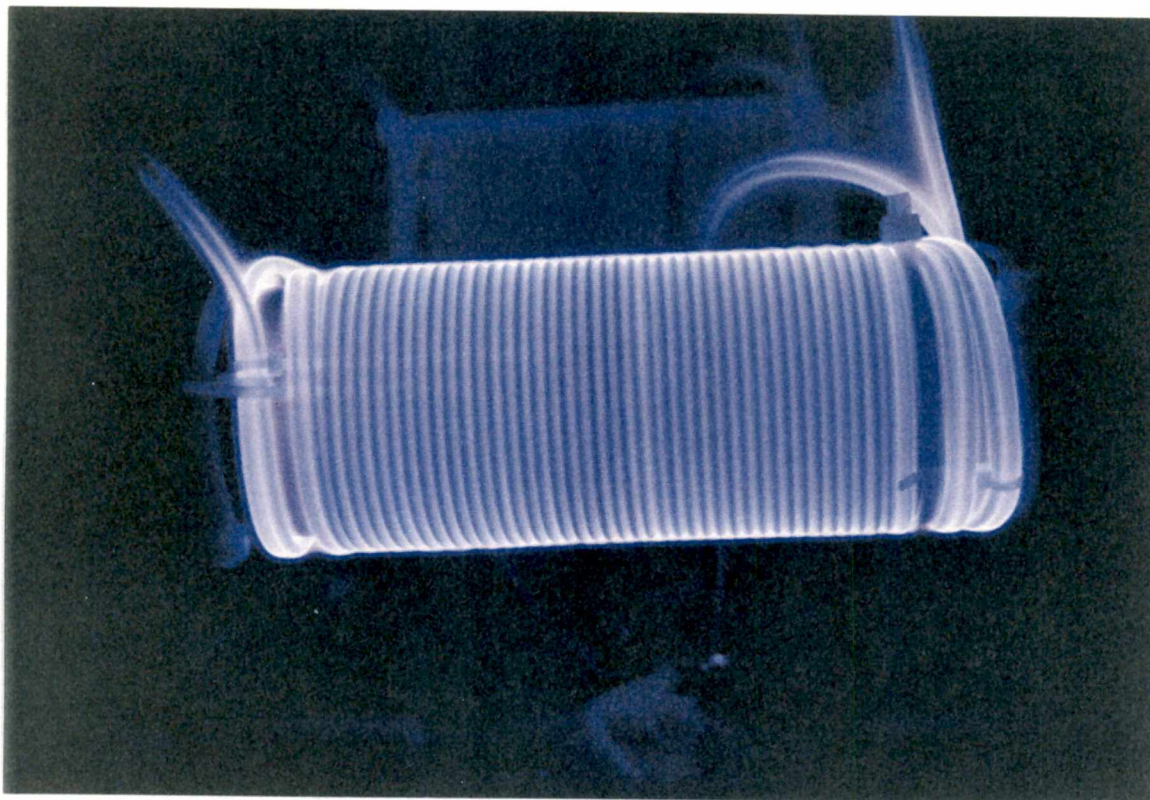


Figure 5.4.3 Plasma was generated on the surface of a simulated aircraft fuselage in mixed air with helium; the RF frequency was 3 KHz; the electrode power supply RMS voltage was 2KV.

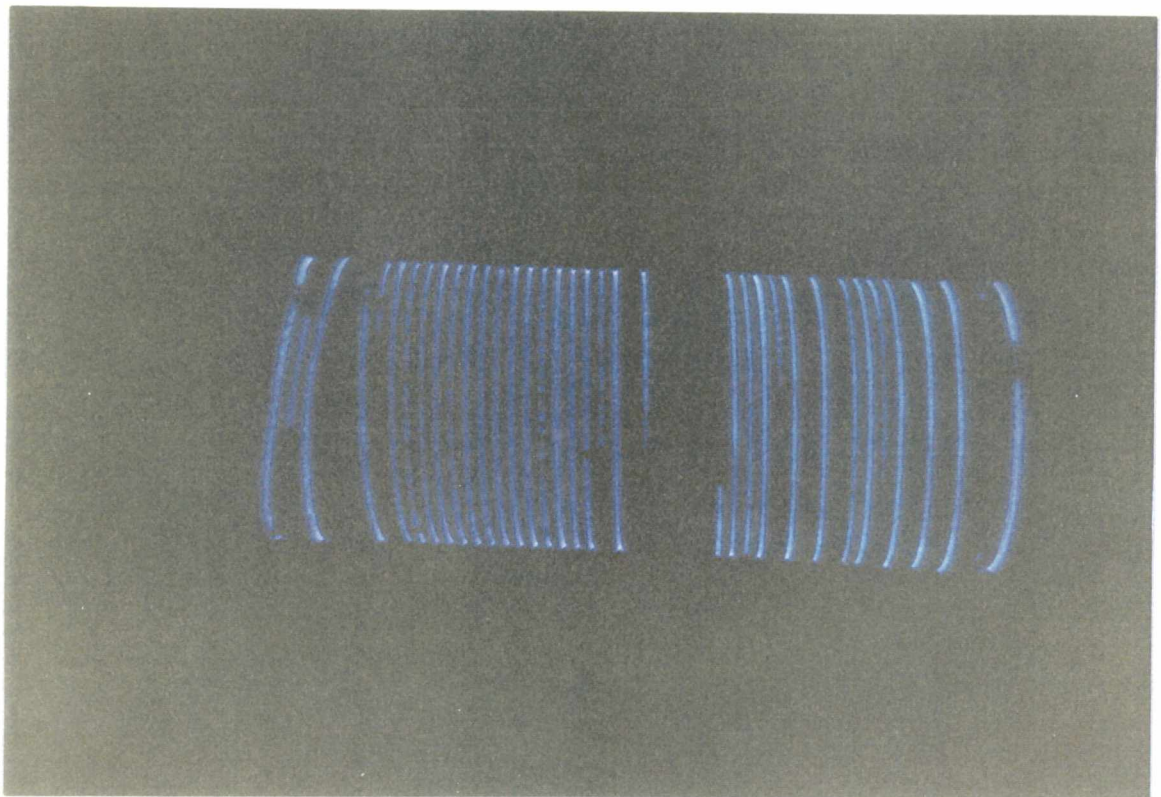


Figure 5.4.4 Plasma was generated on the surface of a simulated aircraft fuselage in air; the RF frequency was 3 KHz; the electrode power supply RMS voltage was 2KV.

generator has been geometrically transformed and made to operate on a planar surface(see fig. 5.4.2), producing a uniform glow discharge plasma layer up to several millimeters thick(see fig. 5.4.3). The electric field on this surface plasma layer can provide a body force per unit area of up to several thousand pascals(Ref. 26). Some speculative applications to boundary layer control, turbulence suppression, drag reduction and noise reduction will be discussed.

As we discussed before, we have capability to create a layer of uniform glow discharge plasma on a simulated aircraft fuselage. If we apply it to the fluid mechanical boundary layer control for the aircraft, the first question will be " what conditions will the trapped-ion plasma be "blown away" by the airflow? The worst case is if the ions cannot replenish themselves during a half-period of oscillation, and are convected the distance between electrodes in a half-period. The best case is if the ions are produced rapidly enough that they are convected away only when the convection velocity is equal to the ion drift velocity in the electric field.

Figure 5.5.1 shows our experiment set up for the testing if the glow discharge plasma will blow away by the airflow. We used a high-velocity air jet which driven by a 2.5 atmosphere pressure to perturb the plasma layer on the simulated aircraft fuselage as shown on figure 5.5.2. The plasma survived(Ref. 20).

This project study is new and requires cooperation with other research organizations. There will be more scientists and researchers from other fields joining our research. I am looking forward to continue to work on it.

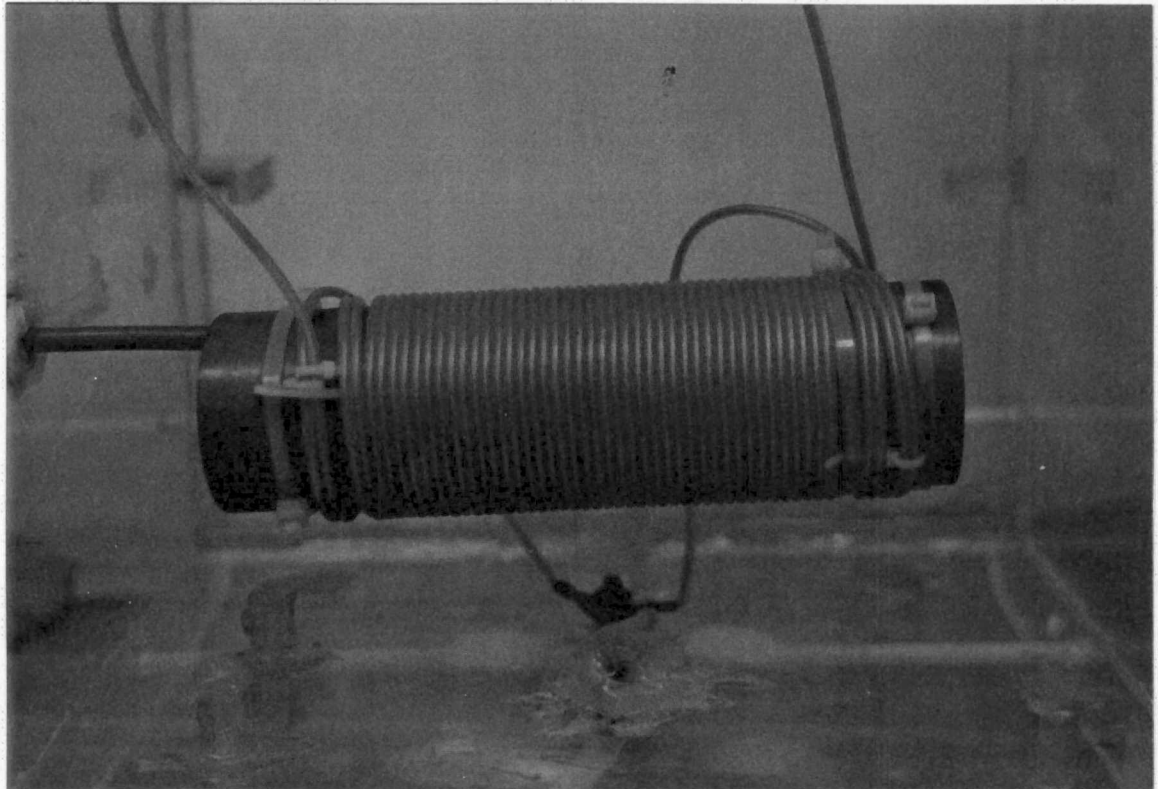


Figure 5.5.1 The experimental set up for the airflow testing.

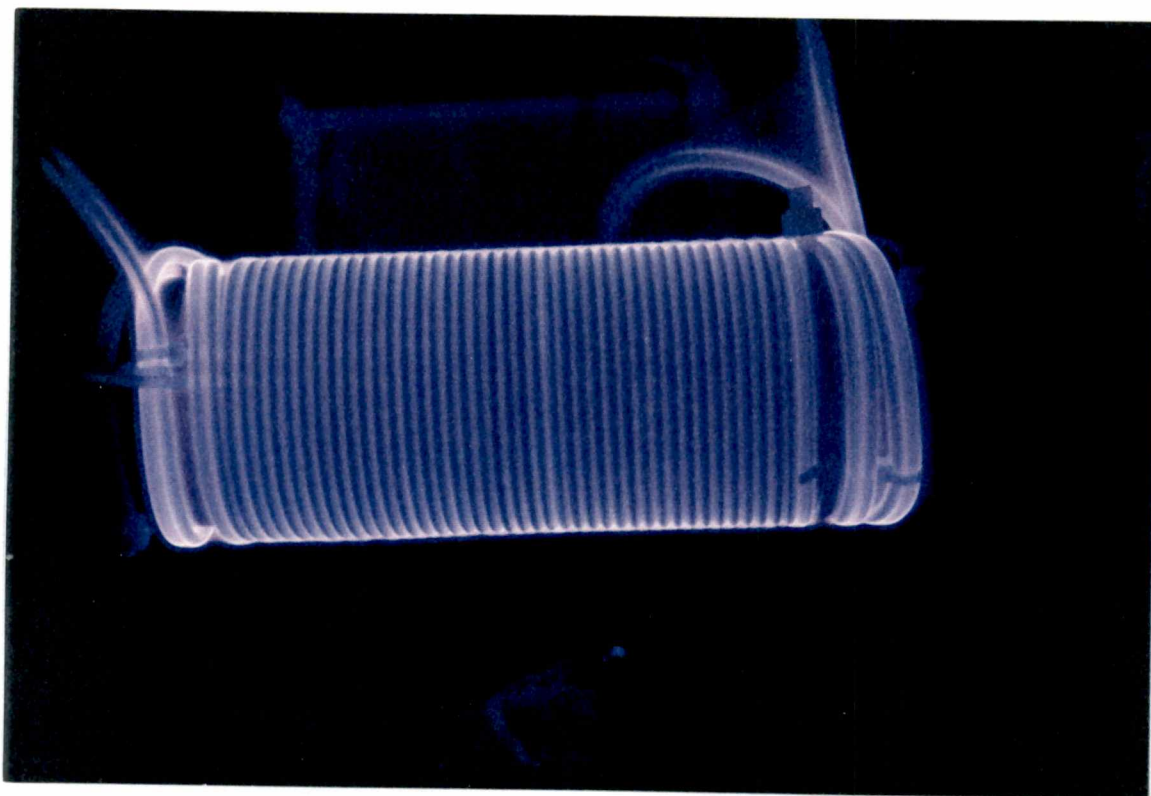


Figure 5.5.2 The plasma layer on the simulated aircraft fuselage survived during the exposure to a high-velocity air jet driven by a 2.5 atmosphere pressure differential.

6. CONCLUSIONS

A new type of atmospheric glow discharge plasma has been generated at the UTK Plasma Science Laboratory. This plasma is capable of operating at one atmosphere pressure, in helium, argon and some other gases, including air. It has been named the One Atmosphere Uniform Glow Discharge Plasma. It is the first time a uniform glow discharge plasma has been generated at one atmosphere in our laboratory. This is a capacitive RF plasma which can be generated in a parallel plate geometry. It is neither a corona discharge nor a filamentary discharge. This plasma can generate useful active species for industrial applications.

Both experimental and theoretical investigations for the one atmosphere uniform glow discharge plasma have been conducted. The physical processes which make this discharge possible are based on an ion trapping mechanism at lower RF frequency, and both ion and electron trapping at higher RF frequencies which results in a filamentary discharge. A certain RF frequency, electric field, and electrode separation, will be required to maintain this discharge. In our experiment, we generated up to 2.4 liters of uniform glow discharge plasma by using about 530 cm^2 parallel plate electrodes, our power supply can provide up to 5 kilowatts RF power at RMS voltages up to 10 kV, and over a frequency range from 1 KHz to 100 KHz.

Some of the important plasma-related characteristics of the steady-state uniform glow discharge plasma at atmospheric pressure have been measured. These measurements show this plasma is of low power density, related not

only to the reactive power input but also the RF frequency applied. Since it operated at high pressure, the collision mean free paths of all charged species are much smaller than the electron Debye distance, the thickness of any sheath which would surround a Langmuir probe or any electrode put into the plasma. For this reason alone, traditional plasma diagnostic techniques like the Langmuir probe are inappropriate. New plasma diagnostic methods have to be developed for this new kind of plasma.

From the point of industrial applications, it is not excessive to say a new page has been opened by the successful generation of a steady-state uniform glow discharge plasma at atmospheric pressure. First of all, the traditional low pressure generated industrial application plasma will be replaced by an atmospheric plasma. Plasma surface treatment processes will no longer enforce batch processing. Some plasma-related processes which can only be operated at atmosphere pressure by a glow discharge plasma will become possible. The experiments we did for the polymer material surface treatment has shown important changes of the surface properties. The modified polymer fabrics have shown that the wettability, wickability, and printability were significantly improved. To take this plasma as a new kind of lighting source research also presents a quite attractive possibility for industrial lighting, because this new type of lighting source may be safer, and have a longer life compared to other plasma lighting devices.

In order to extend the application of our atmospheric steady-state uniform glow discharge plasma to various industrial applications, several different geometries of free-standing atmosphere plasma generators which can produce a surface layer of plasma at atmosphere pressure have been developed. This experiment brings the application of plasma cloaking to aircraft closer. The

surface layer of plasma may absorb microwave radiation to prevent radar detection. Furthermore, the application of an atmospheric glow discharge to drag reduction and aerodynamic boundary layer control on the aircraft by using the electrical body force are more interesting and remarkable. This may bring a revolution of aircraft design and operation.

We just starting at the beginning of the one atmosphere uniform glow discharge plasma research and study. There is much more to learn. To bring the laboratory study to industrial application, there still a long way to go. As one of the principal researchers, I would like to get a chance to continue working on this research project in the future. Also, I am very glad during my graduate study and research, I have had the chance to work on this project which brought me four issued US patents and many publications.

REFERENCES

REFERENCES

1. Roth, J. R. (1995): Industrial Plasma Engineering: Volume I-Principles. Institute of Physics Press, Bristol, U. K. ISBN 0-7503-0318-2.
2. Chen F. F. (1974) Introduction to Plasma Physics and Controlled Fusion Vol. I; Plasma Physics; 2nd edn. (New York: Plenum)
3. Liu, Chaoyu and Roth, J. Reece; Applications of the One Atmosphere Glow Discharge Plasma to Illumination and Aerodynamic Boundary Layer Control. The 36th Annual Meeting, APS Division of Plasma Physics; 7-11 November 1994, Minneapolis, MN. APS Bulletin, Vol. 39, No. 7, Nov. 1994. p.1730.
4. Roth, J. R.; Liu, Chaoyu; and Laroussi, M.: Experimental Generation of a Steady-State Glow Discharge at Atmospheric Pressure. Paper 5P-21, Proc. 1992 IEEE International Conference on Plasma Science, Tampa, FL, IEEE Catalog No. 92-Th0460-6, ISBN 0-7803 0716-X (1992). pp. 170-171.
5. Roth, J. R. Ball Lightning: What Nature is Trying to Tell the Plasma Research Community. Fusion Technology, Vol.27, (May 1995) pp. 225-269.

6. Bartnikas, R. and McMahon, E. J. Engineering Dielectrics
Vol I. Corona Measurement and Interpretation
ASTM Special Technical Publication No. 669 (1979)
(Philadelphia, PA: ASTM) LCCCN 78.72477,
ISBN 04-669000-21
7. Cobine, J. D. Gaseous Conductors (1958) (New York: Dover
Publications)
8. Tsai, Peter P. and Wadsworth, Larry C. Air Filtration
Improved By Electrostatically Charging Fibrous
Materials, Particulate Science and Technology, Volume
12 (1994), pp. 323-332.
9. Gross B, Grycz B and Miklossy K. 1969 Plasma Technology
(New York: American Elsevier) LCCCN 68-27535.
10. Kanda, N., Kogoma, M., Jinno, H., Uchigama, H., and
Okazaki, S.: Atmospheric Pressure Glow Plasma and Its
Application to Surface Treatment and Film Deposition II
Development of a New Method for Lower Cost. 1991 Proc.
10th Int. Symp. on Plasma Chemistry vol.3 (International
Union of Pure and Applied Chemistry) pp. 3.2-20.
11. Anon. 1991 Plasma Processing of Materials: Scientific
Opportunities and Technological Challenges (National
Academy Press, Washington DC) ISBN 0-309-04597-5.

12. Roth, J. R., Spence, Paul D., and Liu, Chaoyu: Preliminary Measurements of the Plasma Properties of a One Atmosphere Glow Discharge Plasma. APS Bulletin, Series II, Vol. 38 No.10 (1993) p.1901.
13. Liu, Chaoyu, Tsai, P. P. and Roth, J. R.: Plasma-Related Characteristics of a Steady-State Glow Discharge at Atmospheric Pressure. IEEE Catalog No. 93 Ch3334-0 (1993), p. 129.
14. Liu, Chaoyu and Roth, J. R.: An Atmospheric Glow Discharge Plasma for Aerodynamic Boundary Layer Control. IEEE Catalog No. 94CH3465-2, ISBN 0-7803-2207-7 (1994) p. 97-98.
15. Roth, J. R.; Tsai, P. P.; Liu, Chaoyu: Steady-State, Glow Discharge Plasma. US. Patent No.: 5,387,842; Date of Patent: Feb. 7, 1995.
16. Neumann, G. and Banziger U.; Kammeyer, M.; Lange, M.: Plasma-Density Measurements by Microwave Interferometry and Langmuir Probes in an rf Discharge. Rev. Sci. Instrum. Vol. 64, No. 1, (January 1993) pp. 19-25.
17. Roth, J. R.; Wadsworth, L. C.; Tsai, P. P.; Spence, P. D. and Liu, Chaoyu: One Atmosphere Glow Discharge Plasma for Surface Treatment of Nonwoven Textiles. Proc. 3rd Annual TANDEC Conference, Nov. 1-3, Knoxville, TN (1993).

18. Wadsworth, L. C.; Roth, J. R.; Tsai, P. P.; and Spence, P. D.:
Fiber Surface Modifications Using One Atmosphere Glow Discharge Plasma. Proc. 20th ACS National Meeting, San Diego, CA March 13-18, 1994.
19. Liu, Chaoyu: The Research and Study of One Atmosphere Plasma Generation and Applications in U.T.K. Plasma Science Laboratory. Presented at the UTK Plasma Science Seminar, Dec. 3, 1993.
20. Liu, Chaoyu and Roth, J. R.: Applications of the One Atmosphere Glow Discharge Plasma to Illumination and Aerodynamic Boundary Layer Control. APS Bulletin, Vol. 39, No. 7, Nov. 1994. p. 1730.
21. Liu, Chaoyu: Plasma Cloaking of Aircraft in the Atmosphere. Presented at the UTK Plasma Science Seminar, April 8, 1994.
22. Rakowski, W. (1989): Plasma Modification of Wool Under Industrial Conditions. Melliand Textilberichte, Vol. 70, pp. 780-785.
23. Roth, J. R. (1995): Industrial Plasma Engineering: Volume II-Applications. Institute of Physics Press, Bristol, U.K. (to be published April, 1997).
24. Combs, L. H.; Liu, Chaoyu and Roth, J. R.: Microwave

- Interactions with a Magnetized Plasma near the Electron Cyclotron Frequency. 1990 IEEE International Conference on Plasma Science, IEEE Catalog #90CH2857-1(1990) p. 151.
25. Laroussi, M.; Liu, Chaoyu and Roth, J. R.: Numerical and Experimental Investigations of the Attenuation of microwaves by a Non-Uniform Plasma. APS Bulletin, Series II, Vol. 36 (1991) p. 2485.
26. Roth, J. R.: Boundary Layer Control By a One Atmosphere Uniform Glow Discharge Plasma Layer. Presented at the 22nd IEEE International conference on Plasma Science, Madison, Wisconsin, June 5-8, 1995.
27. Roth, J. R.: Microwave Absorption system. US. Patent; US. Patent No.: 4,989,006; Date of Patent: Jan. 29, 1991.

APPENDICES

Appendix A: United States Patent

Steady-State, Glow Discharge Plasma

Inventors: J. R. Roth; P. P. Tsai; Chaoyu Liu.

Patent No. 5,387,842; Date of Patent: Feb. 7, 1995.



US005387842A

United States Patent [19]

[11] Patent Number: 5,387,842

Roth et al.

[45] Date of Patent: Feb. 7, 1995

[54] STEADY-STATE, GLOW DISCHARGE PLASMA

[75] Inventors: John R. Roth; Peter P. Tsai; Chaoyu Liu, all of Knoxville, Tenn.

[73] Assignee: The University of Tennessee Research Corp., Knoxville, Tenn.

[21] Appl. No.: 68,508

[22] Filed: May 28, 1993

[51] Int. Cl.⁶ H01J 7/24; H05B 31/26

[52] U.S. Cl. 315/111.21; 118/723 E; 313/231.31; 204/298.04

[58] Field of Search 315/111.21, 111.51; 118/723 E; 204/298.04; 219/121.1; 313/231.31

[56] References Cited

U.S. PATENT DOCUMENTS

4,469,932 9/1984 Spiegelberg et al. 313/231.31
 4,687,573 8/1987 Miller et al. 210/143
 4,771,730 9/1988 Tezuka 118/723 E
 4,919,968 4/1990 Buhl et al. 219/121.11
 5,213,658 5/1993 Ishida 118/723 E

FOREIGN PATENT DOCUMENTS

0200529 11/1983 Japan 315/111.21
 1177374 8/1986 Japan 118/723 E
 0312223 12/1990 Japan 315/111.21

OTHER PUBLICATIONS

Authors: Naoki Kanda, Masuhiro Kogoma, Hiroshi Jinno, Hiroshi Uchiyama and Sachiko Okazaki Title: Atmospheric Pressure Glow Plasma and Its Application to Surface Treatment and Film Disposition Publication: International Union of Pure and Applied Chemistry, 10th International Symposium on Plasma Chemistry, Symposium Proceedings, vol. 3, Bochum, Germany, Aug. 4-9, 1991.

Authors: M. Kogoma, H. Kasai, K. Takahashi, T. Moriwaki and S. Okazaki Title: Wettability Control of a Plastic Surface by CF₄-O₂ Plasma and Its Etching Effect Publication: J. Phys. D: Appl. Phys., vol. 20, (1987).

Authors: S. Kanazawa, M. Kogoma, T. Moriwaki and S. Okazaki Title: Stable Glow Plasma at Atmospheric

Pressure Publication: J. Phys. D: Appl. Phys., vol. 21 (1988).

Author: Chaoyu Liu Title: Plasma-Related Characteristics of a Steady-State Glow Discharge at Atmospheric Pressure Publication: Presented at the 1993 Sigma XI Graduate Student Paper Competition, The University of Tennessee, Knoxville, Tenn., Mar. 4, 1993.

Authors: C. Liu and J. R. Roth Title: Characteristics of a Steady-State, Low Power Glow Discharge at Atmospheric Pressure Publication: Bulletin of the American Physical Society Series II, vol. 37, No. 6, Nov., 1992. Authors: J. Reece Roth, Mounir Laroussi and Chaoyu Liu Title: Experimental Generation of a Steady-State Glow Discharge at Atmospheric Pressure Publication: 1992 19th IEEE International Conference on Plasma Science, Conference Record-Abstracts-1-3 Jun., 1992. Author: Witold Rakowski Title: Effect and Cost of Plasma Treatment of Polypropylene Melt Blown Webs Publication: Second Annual TANDEC Conference, 13-16 Oct., 1992.

Authors: Von Engel, A.; Seeliger, R.; and Steenbeck, M. Title: On the Glow Discharge At High Pressure. Publication: Ziet, fur Physik, vol. 85 (1993) pp. 144-160. English language translation of above from original German.

(List continued on next page.)

Primary Examiner—Robert J. Pascal

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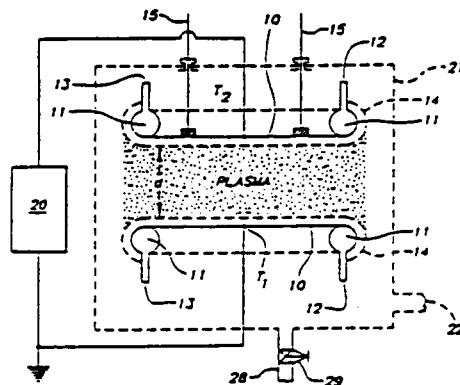
Attorney, Agent, or Firm—Weiser & Associates

[57]

ABSTRACT

A steady-state, glow discharge plasma is generated within the volume between a pair of parallel, insulated metal plate electrodes spaced up to 5 cm apart and R.F. energized with an rms potential of 1 to 5 KV at 1 to 100 KHz. The electrodes are located within an enclosure capable of maintaining an atmosphere other than atmospheric air between the electrode surfaces. Space between the electrodes is occupied by a noble gas such as helium, neon, argon, etc. or mixtures thereof.

22 Claims, 4 Drawing Sheets



OTHER PUBLICATIONS

- EPO Publication No. 0,068,775 Issue Date: Jan. 5, 1983
Inventor: Hata, et al.
- EPO Publication No. 0,117,561 Issue Date: Sep. 5, 1984
Inventor: Akagi, et al.
- Authors: Wakida, T.; Kawamura, H.; Han L.: Hwan Kim, K.; Goto, T.; and Takagishi, T. Title: Changes In Surface Properties of Poly(ethylene terephthalate) Treated With Low Temperature Plasma: Effect of Pretreatment With Dimethylformamide Publication: *Sen-I-Gakkaishi*, vol. 42, No. 2 (1986).
- Authors: Wakida, T.; Kawamura, H.; Song, J. C.; Goto, T.; Takagishi, T. Title: Surface Free Energy of Poly(ethylene Terephthalate) and Nylon 6 Films Treated With Low Temperature Plasma Publication: *Sen-I-Gakkaishi*, vol. 43, No. 7 (1987).
- Japanese Patent No. 62-235,339 Issue Date: Oct. 15, 1987 Inventors: M. Kogoma et al.
- Author: Rakowski, W. Title: Plasma Modification of Wool Under Industrial Conditions Publication: *Melliand Textilberichte*, vol. 70 (1989) pp. 780-785.
- Authors: Roth, J. R.; Spence, P. D.; Liu, C. Title: Plasma-Related Characteristics of a Steady-State Glow Discharge at Atmospheric Pressure Publication: Paper 2P-18, *Proc. 1993 IEEE International Conference on Plasma Science*, Vancouver, B.C. IEEE Catalog No. 93-CH3334-0, ISBN 0-7803-1360-7 (1993), p. 129.
- Authors: Roth J. R.; Spence, P. D.; Liu, C. Title: Preliminary Measurements of the Plasma Properties of a One Atmosphere Glow Discharge Plasma. Publication: Paper presented at *35th Annual Meeting of the APS Division of Plasma Physics*, St. Louis, Mo., Nov. 1-5, 1993; *APS Bulletin*, Series II, vol. 38, No. 10 (1993), p. 1901.

Appendix B: United States Patent

**Method And Apparatus for Glow Discharge Plasma
Treatment of Polymer Materials At Atmospheric Pressure**

Inventors: J. R. Roth; P. P. Tsai; Larry C. Wadsworth;
Chaoyu Liu; Paul D. Spence.

Patent No. 5,403,453; Date of Patent: Apr. 4, 1995.

United States Patent [19]

Roth et al.

US005403453A

[11] Patent Number: 5,403,453

[45] Date of Patent: Apr. 4, 1995

- [54] METHOD AND APPARATUS FOR GLOW DISCHARGE PLASMA TREATMENT OF POLYMER MATERIALS AT ATMOSPHERIC PRESSURE
- [75] Inventors: John R. Roth; Peter P. Tsai; Larry C. Wadsworth; Chaoya Liu; Paul D. Spence, all of Knoxville, Tenn.
- [73] Assignee: The University of Tennessee Research Corporation, Knoxville, Tenn.
- [21] Appl. No.: 145,349
- [22] Filed: Oct. 29, 1993

Related U.S. Application Data

- [63] Continuation-in-part of Ser. No. 68,739, May 28, 1993.
- [51] Int. Cl.⁴ _____ H05F 3/00
- [52] U.S. Cl. _____ 204/164
- [58] Field of Search _____ 204/164

References Cited

FOREIGN PATENT DOCUMENTS

- 0068775 1/1983 European Pat. Off. .
- 0117561 9/1984 European Pat. Off. .
- 200133 9/1986 Japan .
- 62-235339 10/1987 Japan .

OTHER PUBLICATIONS

- Authors: Von Engel, A.; Seeliger, R.; & Steenbeck, M. Title: On the Glow Discharge at High Pressure Publication: *Ziet. fur Physik*, vol. 85 (1933) pp. 144-160.
- Authors: Wakida, T.; Kawamura, H.; Han, L.; Hwan Kim, K.; Goto, T.; & Takagishi, T. Title: Changes in Surface Properties of Poly(Ethylene Terephthalate) Treated with Low Temperature Plasma: Effect of Pre-treatment with Dimethylformamide Publication: *sen-i gakkaiishi*, vol. 43, No. 7 (1987).
- Authors: Wakida, T.; Kawamura, H.; Song, J. C.; Goto, T.; Takagishi, T. Title: Surface Free Energy Poly-(Ethylene Terephthalate) and Nylon 6 Films Treated with Low Temperature Plasma Publication: *Seni-I Gakkaishi*, vol. 43, No. 7 (1987).
- Authors: Kogoma, M.; Kasai, H.; Takahashi, K.; Moriwaki, T. & Okazaki, S. Title: Wettability Control of a Plastic Surface by CF₄ Plasma and Its Etching

Effect Publication: *J. Phys. D: Appl. Phys.*, vol. 20 (1987).

Authors: Kanazawa, S.; Kogoma, M.; Moriwaki, T.; & Okazaki, S. Title: Stable Glow Plasma at Atmospheric Pressure Publication: *J. Physics D: Appl. Phys.*, vol. 21 (1988) pp. 838-840.

Author: Rakowski, W. Title: Plasma Modification of Wool Under Industrial Conditions Publication: *Melliand Textilberichte*, vol. 70 (1989) pp. 780-785.

Authors: Yokoyama, T.; Kogoma, M.; Moriwaki, T.; & Okazaki, S. Title: The Mechanism of the Stabilisation of Glow Plasma at Atmospheric Pressure Publication: *J. Physics D: App. Phys.*, vol. 23 (1990) pp. 1125-1128.

Authors: Naoki Kanda; Masuhiro Kogoma; Hiroshi Jinno; Hiroshi Uchiyama & Sachiko Okazaki Title: Atmospheric Pressure Glow Plasma and Its Application to Surface Treatment and Film Deposition. Publication: *International Symposium on Plasma Chemistry*, Symposium Proceedings, vol. 3, Bochum, Germany, Aug. 4-9, 1991.

Authors: Liu, C.; Chen, D.; & Roth, J. R. Title: Characteristics of a Steady-State, Low Power Glow Discharge at Atmospheric Pressure Publication: *APS Bulletin*, vol. 37, No. 6 (1992) p. 1563.

Authors: Roth, J. R.; Liu, C.; & Laroussi, M. Title: Experimental Generation of a Steady-State Glow Discharge at Atmospheric Pressure Publication: Paper

(List continued on next page.)

Primary Examiner—John Niebling

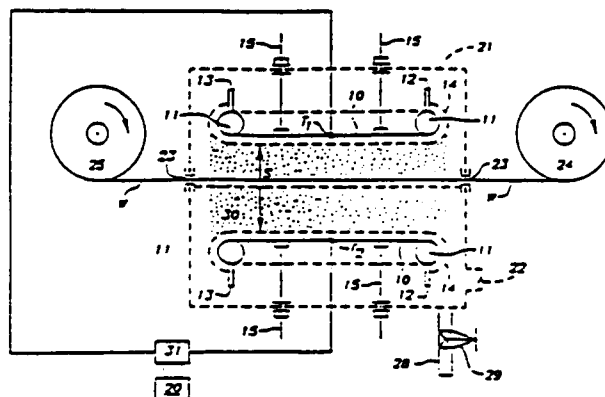
Assistant Examiner—Kishor Mayekar

Attorney, Agent, or Firm—Weiser & Associates

[57] ABSTRACT

Polymer materials such as film and fabrics, woven, non-woven and meltblown, may be non-destructively surface treated to improve water wettability, wickability, and other characteristics by exposure to a glow discharge plasma sustained at substantially atmospheric pressure in air or modified gas atmospheres comprising helium or argon.

12 Claims, 11 Drawing Sheets



OTHER PUBLICATIONS

5P-21, *Proc. 1992 IEEE International Conference on Plasma Science*, Tampa, Fla., IEEE Catalog No. 92-TH0460-6, ISBN 0-7803 0716-X (1992), pp. 170-171.

Authors: Reitz, H.; Schwarz, R.; Salge, J. G. H. Title: Power Modulation for Dielectric Barrier-Discharges Publication: Paper presented at *Twentieth Power Modulator Symposium*, 1992.

Author: Rakowski, W. Title: Effect and Cost of Plasma Treatment of Polypropylene Melt Blown Webs Publication: *Second Annual TANDEC Conference*, 13-16 Oct., 1992.

Author: Liu, C. Title: Plasma-Related Characteristics of a Steady-State Glow Discharge at Atmospheric

Pressure Publication: Presented at the 1993 Sigma XI Graduate Student Paper Competition, The University of Tennessee, Knoxville, Tenn. on Mar. 4, 1993.

Authors: Roth, J. R.; Spence, P. D.; Liu, C. Title: Plasma-Related Characteristics of a Steady-State Glow Discharge at Atmospheric Pressure Publication: Paper 2P-18, *Proc. 1993 IEEE International Conference on Plasma Science*, Vancouver, B. C. IEEE Catalog No. 93-CH3334-0, ISBN 0-7803-1360-7 (1993), p. 129.

Authors: Roth, J. R.; Spence, P. D.; Liu, C. Title: Preliminary Measurements of the Plasma Properties of a One Atmosphere Glow Discharge Plasma Publication: Paper present at 35th Annual Meeting of the APS Division of Plasma Physics, St. Louis, Mo., Nov. 1-5, 1993; *APS Bulletin*, Series II, vol. 38, No. 10 (1993), p. 1901.

Appendix C: United States Patent

One Atmosphere, Uniform Glow Discharge Plasma

Inventors: J. R. Roth; P. P. Tsai; Chaoyu Liu; Mounir Laroussi;

Paul D. Spence.

Patent No. 5,414,324; Date of Patent: May 9, 1995.



US005414324A

United States Patent [19]

[11] Patent Number: 5,414,324

Roth et al.

[45] Date of Patent: * May 9, 1995

[54] ONE ATMOSPHERE, UNIFORM GLOW DISCHARGE PLASMA

[75] Inventors: John R. Roth; Peter P. Tsai; Chaoyu Liu; Mounir Laroussi; Paul D. Spence, all of Knoxville, Tenn.

[73] Assignee: The University of Tennessee Research Corporation, Knoxville, Tenn.

[*] Notice: The portion of the term of this patent subsequent to Feb. 7, 2012 has been disclaimed.

[21] Appl. No.: 145,786

[22] Filed: Oct. 29, 1993

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 68,508, May 28, 1993.

[51] Int. Cl.⁶ H01J 7/24

[52] U.S. Cl. 315/111.21; 315/111.51; 118/723 E

[58] Field of Search 315/111.21, 111.51; 118/723 E, 723 I; 313/231.31

[56] References Cited

U.S. PATENT DOCUMENTS

5,147,493 9/1992 Nishimura 315/111.51
5,272,417 12/1993 Ohmi 315/111.21
5,309,063 5/1994 Singh 315/111.51

FOREIGN PATENT DOCUMENTS

0068775 1/1983 European Pat. Off. .
0117561 9/1984 European Pat. Off. .
0292582 8/1991 Germany 315/111.21
0200529 11/1983 Japan 313/111.21
1177374 8/1986 Japan 118/723 E
0002544 1/1987 Japan 118/723 E
62-235339 10/1987 Japan .
4225226 8/1992 Japan 315/111.21

OTHER PUBLICATIONS

Authors: Von Engel, A.; Seeliger, R.; & Steenbeck, M.
Title: On The Glow Discharge At High Pressure Publication: *Zeit. fur Physik*, vol. 85 (1933) pp. 144-160.

Authors: Wakida, T.; Kawamura, H.; Han, L.; Hwan Kim, K.; Goto, T.; & Takagishi, T. Title: Changes In Surface Properties Of Poly(Ethylene Terephthalate)

Treated With Low Temperature Plasma: Effect Of Pretreatment With Dimethylformamide Publication: *sen-i gakkaishi*, vol. 43, No. 7 (1987).

Authors: Wakida, T.; Kawamura, H.; Song, J. C.; Goto, T.; Takagishi, T. Title: Surface Free Energy Poly-(Ethylene Terephthalate) And Nylon 6 Films Treated With Low Temperature Plasma Publication: *Sen-i Gakkaishi*, vol. 43, No. 7 (1987).

Authors: Kogoma, M.; Kasai, H.; Takahashi, K.; Moriwaki, T. & Okazaki, S. Title: Wettability Control Of A Plastic Surface By CF₄ Plasma And Its Etching Effect Publication: *J. Phys. D: Appl. Phys.*, vol. 20 (1987).

Authors: Kanazawa, S.; Kogoma, M.; Moriwaki, T.; & Okazaki, S. Title: Stable Glow Plasma At Atmospheric Pressure Publication: *J. Physics D: Appl. Phys.*, vol. 21 (1988) pp. 838-840.

Author: Rakowski, W. Title: Plasma Modification Of Wool Under Industrial Conditions Publication: *Melliand Textilberichte*, vol. 70 (1989) pp. 780-785.

Authors: Yokoyama, T.; Kogoma, M.; Moriwaki, T.; & Okazaki, S. Title: The Mechanism Of The Stabilisation Of Glow Plasma At Atmospheric Pressure Publication: *J. Physics D: App. Phys.*, vol. 23 (1990) pp. 1125-1128.

Authors: Naoki Kanda; Masuhiro Kogoma; Hiroshi (List continued on next page.)

Primary Examiner—Robert J. Pascal

Assistant Examiner—Darius Gambino

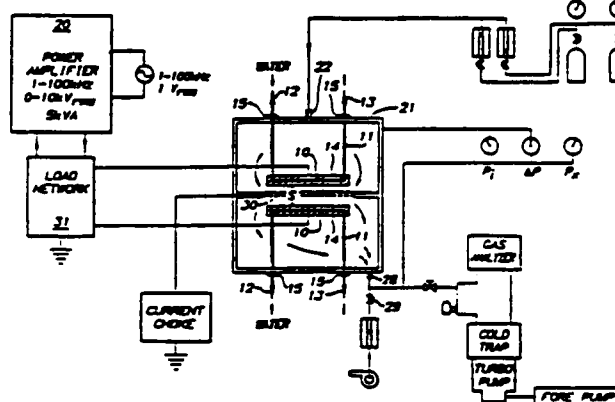
Attorney, Agent, or Firm—Weiser & Associates

[57]

ABSTRACT

A steady-state, glow discharge plasma is generated at one atmosphere of pressure within the volume between a pair of insulated metal plate electrodes spaced up to 5 cm apart and R.F. energized with an rms potential of 1 to 5 KV at 1 to 100 KHz. Space between the electrodes is occupied by air, nitrous oxide, a noble gas such as helium, neon, argon, etc. or mixtures thereof. The electrodes are charged by an impedance matching network adjusted to produce the most stable, uniform glow discharge.

27 Claims, 10 Drawing Sheets



OTHER PUBLICATIONS

- Jimmo; Hiroshi Uchiyama & Sachiko Okazaki Title: Atmospheric Pressure Glow Plasma And Its Application To Surface Treatment And Film Deposition. Publication: *International Symposium on Plasma Chemistry*, Symposium Proceedings, vol. 3, Bochum, Germany, Aug. 4-9, 1991.
- Authors: Liu, C.; Chen, D.; & Roth, J. R. Title: Characteristics Of A Steady-State, Low Power Glow Discharge At Atmospheric Pressure Publication: *APS Bulletin*, vol. 37, No. 6 (1992) p. 1563.
- Authors: Roth, J. R.; Liu, C.; & Laroussi, M. Title: Experimental Generation Of A Steady-State Glow Discharge At Atmospheric Pressure Publication: Paper 5P-21, *Proc. 1992 IEEE International Conference on Plasma Science*, Tampa, Fla., IEEE Catalog No. 92-TH0460-6, ISBN 0-7803 0716-X (1992), pp. 170-171.
- Authors: Reitz, H.; Schwarz, R.; Salge, J. G. H. Title: Power Modulation For Dielectric Barrier-Discharges Publication: Paper presented at *Twentieth Power Modulator Symposium*, 1992.
- Author: Rakowski, W. Title: Effect And Cost Of Plasma Treatment Of Polypropylene Melt Blown Webs Publication: *Second Annual TANDEC Conference*, 13-16 Oct., 1992.
- Authors: Liu, C. Title: Plasma-Related Characteristics Of A Steady-State Glow Discharge At Atmospheric Pressure Publication: Presented at the 1993 *Sigma XI Graduate Student Paper Competition*, The University of Tennessee, Knoxville, Tenn. on Mar. 4, 1993.
- Authors: Roth, J. R.; Spence, P. D.; Liu, C. Title: Plasma-Related Characteristics of A Steady-State Glow Discharge At Atmospheric Pressure Publication: Paper 2P-18, *Proc. 1993 IEEE International Conference on Plasma Science*, Vancouver, B. C. IEEE Catalog No. 93-CH3334-0, ISBN 0-7803-1360-7 (1993), p. 129.
- Authors: Roth, J. R.; Spence, P. D.; Liu, C. Title: Preliminary Measurements Of The Plasma Properties Of A One Atmosphere Glow Discharge Plasma Publication: Paper Present at *35th Annual Meeting of the APS Division of Plasma Physics*, St. Louis, Mo., Nov. 1-5, 1993; *APS Bulletin*, Series II, vol. 38, No. 10 (1993), p. 1901.

Appendix D: United States Patent
Method and Apparatus for Glow Discharge Plasma
Treatment of Polymer Materials at Atmospheric Pressure
Inventors: J. R. Roth; L. C. Wadsworth; P. P. Tsai; Chaoyu Liu
Patent No. 5,456,972; Date of Patent: Oct. 10, 1995



US005456972A

United States Patent [19]

[11] Patent Number: 5,456,972

Roth et al.

[45] Date of Patent: Oct. 10, 1995

[54] METHOD AND APPARATUS FOR GLOW DISCHARGE PLASMA TREATMENT OF POLYMER MATERIALS AT ATMOSPHERIC PRESSURE

[75] Inventors: John R. Roth; Peter P. Tsai; Larry C. Wadsworth; Chaoya Liu, all of Knoxville, Tenn.

[73] Assignee: The University of Tennessee Research Corporation, Knoxville, Tenn.

[21] Appl. No.: 68,739

[22] Filed: May 28, 1993

[51] Int. Cl.⁶ D03D 3/00

[52] U.S. Cl. 428/224; 156/272.2; 156/272.6; 264/483; 428/903; 429/247; 429/249; 429/250

[58] Field of Search 429/247, 249, 429/250; 428/224, 903; 264/22; 156/272.2, 272.6

[56] References Cited

U.S. PATENT DOCUMENTS

5,227,172 7/1993 Deeds 264/22
5,270,137 12/1993 Kubota 429/249

OTHER PUBLICATIONS

Authors: Von Engel, A.; Seeliger, R.; and Steenbeck, M., Title: On The Glow Discharge At High Pressure. Publication: *Ziet. fur Physik*, vol. 85 (1993) pp. 144-160. English language translation of above from original German.

EPO Publication No. 0,068,775 Issue Date: Jan. 5, 19083, Inventor: Hata, et al.

EPO Publication No. 0,117,561, Issue Date: Sep. 5, 1984, Inventor Akagi, et al.

Authors: Wakida, T.; Kawamura, H.; Han, L.; Hwan Kim, K.; Goto, T.; and Takagishi, T., Title: Changes In Surface Properties of Poly(Ethylene Terephthalate) Treated With Low Temperature Plasma: Effect of Pretreatment With Dimethylformamide, Publication: *Sen-I Gakkaishi*, vol. 42 No. 2 (1986).

Authors: Wakida, T.; Kawamura, H.; Song, J. C.; Goto, T.; Takagushi, T., Title: Surface Free Energy Of Poly(Ethylene

Terephthalate) And Nylon 6 Films Treated With Low Temperature Plasma, Publication: *Sen-I Gakkaishi*, vol. 43, No., 7 (1987).

Japanese Patent No. 62-235,339, Issue Date: Oct. 15, 1987, Inventors: M. Kogoma, et al.

Author: Rakowski, W. Title: Plasma Modification Of Wool Under Industrial Conditions, Publication: *Melliand Textilberiche*, vol. 70 (1989) pp. 780-785.

Authors: Reitz, H.; Schwarz, R.; Salge, J. G. H., Title: Power Modulation For Dielectric Barrier-Discharges, Publication: Paper presented at *Twentieth Power Modulator Symposium*, 1992.

Authors: Roth, J. R.; Spence, P. D.; Liu, C., Title: Plasma-Related Characteristics Of A Steady-State Glow Discharge At Atmospheric Pressure, Publication: Paper 2P-18, *Proc. 1993 IEEE International Conference on Plasma Science*, Vancouver, B.C. IEEE Catalog No. 93-CH3334-0, ISBN 0-7803-1360-7 (1993), p. 129.

Authors: Roth, J.R.; Spence, P. D.; Lin, C., Title: Preliminary Measurements Of The Plasma Properties Of A One Atmosphere Glow Discharge Plasma, Publication: Paper presented at *35th Annual Meeting of the APS Division of Plasma Physics*, St. Louis, MO., Nov. 1-5, 1993, *APS Bulletin*, Series II, vol. 38, No. 10 (1992), p. 1901.

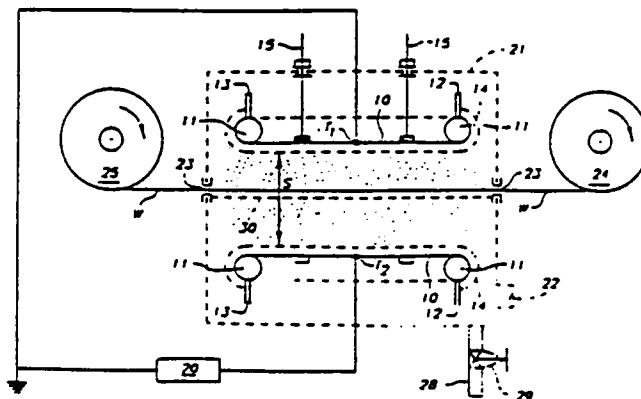
(List continued on next page.)

Primary Examiner—James J. Bell
Attorney, Agent, or Firm—Weiser & Associates

[57] ABSTRACT

Polymer materials such as film and fabrics, woven, non-woven and meltblown, may be non-destructively surface treated to improve water wettability by exposure to a glow discharge plasma sustained at substantially atmospheric pressure in a modified gas atmosphere comprising helium or argon.

29 Claims, 5 Drawing Sheets



OTHER PUBLICATIONS

Author: Noiki Kanda, Mashuhiro Kogoma, Hiroshi Hinno, Hiroshi Uchiyama and Sachiko Okazaki, Title: Atmospheric Pressure Glows Plasma and Its Application to Surface Treatment and Film Disposition, Publication: International Union of Pure and Applied Chemistry, 10th International Symposium on Plasma Chemistry, Symposium Proceedings, vol. 3, Bochum, Germany, Aug. 4-9, 1991.

Authors: M. Kogoma, H. Kasai, K. Takahashi, T. Moriwaki and S. Okazaki, Title: Wettability Control of a Plastic Surface by CF_4 - O_2 Plasma and Its Etching Effect, Publication: J. Phys. D: Appl. Phys., vol. 20, (1987).

Authors: S. Kanazawa, M. Kogoma, T. Moriwaki and S. Okazaki, Title: Stable Glow Plasma at Atmospheric Pressure, Publication: J. Phys. D: Appl. Phys., vol. 21 (1988).

Author: Chaoyo Liu, Title: Plasma-Related Characteristics

of a Steady-State Glow Discharge at Atmospheric Pressure, Publication: Presented at the 1993 Sigma XI Graduate Student Paper Competition, The University of Tennessee, Knoxville, Tenn. Mar. 4, 1993.

Authors: C. Liu and J. R. Roth, Title: Characteristics of a Steady-State, Low Power Glow Discharge at Atmospheric Pressure, Publication: Bulletin of the American Physical Society Series II, vol. 37, No. 6, Nov. 1992.

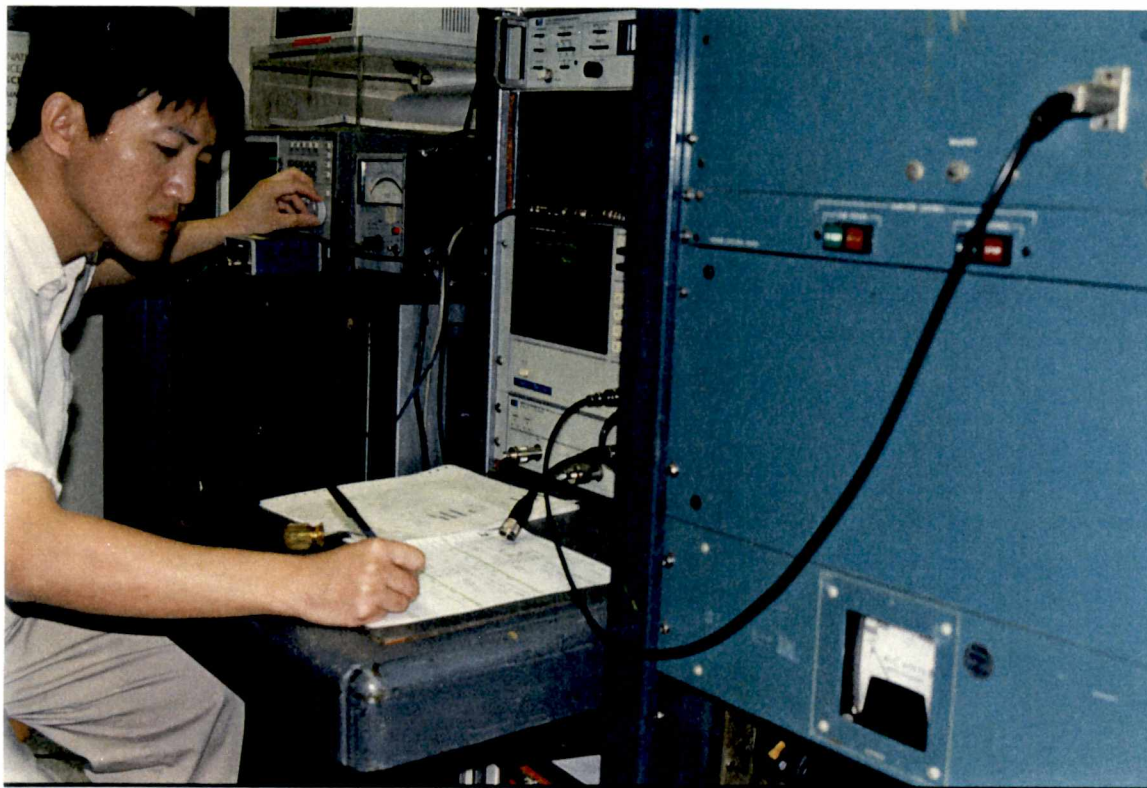
Authors: J. Reece Roth, Mounir Laroussi and Chaoyo Liu, Title: Experimental Generation of a Steady-State Glow Discharge at Atmospheric Pressure, Publication: 1992 19th IEEE International Conference on Plasma Science, Conference Record -Abstracts -1-3 Jun. 1992.

Author: Witold Rakowski, Title: Effect and Cost of Plasma Treatment of Polypropylene Melt Blown Webs, Publication: Second Annual TANDEC Conference. 13-16 Oct. 1992.

VITA

Chaoyu Liu was born in Chengdu, Sichuan, P. R. China on Dec. 9, 1962. He attended schools in the public system of Chengdu, Sichuan, where he graduated from the 21st High School in June, 1979. He entered the Chengdu Institute of Radio Engineering(CIRE) and graduated in July 1983 with a BS. degree. After graduating from college, he attended the Microcomputer Research Institute in CIRE as a research engineer. During this time, he received the 1987 National Award(P.R.China) for "Microcomputer Control System on Medicine Production in the Sichuan Medicine Co.". In August 1987, he entered the University of Electronic Science and Technology of China(UESTC) as a graduate student in the field of high energy electronics and graduated at December 1987 with a Master's degree in engineering. His thesis topic was "The Study of A New Structure Orottron". In 1990, he entered the University of Tennessee, Knoxville, and worked as a Graduate Research Assistant in the Plasma Science Laboratory of the Department of Electrical and Computer Engineering under Prof. Roth's direction.

Up to now, he has co-authored more than 20 papers and patents on the plasma and its applications. Four US patents have been issued with him as a joint inventor. Currently, he is interested in the theory of the one atmosphere uniform glow discharge plasma and its applications. Also, he is present as the president of SINCO., in Knoxville, Tennessee. SINCO. is a company which service for the international corporations.



The author, Chaoyu Liu, was working in the UTK Plasma Science Laboratory.

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