Prospective Industrial Applications of the One Atmosphere Uniform Glow Discharge Plasma

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Abstract - The majority of industrial plasma processing is conducted with glow discharges at pressures below 10 torr. This tends to limit plasma processing applications to high value workpieces as a result of the high capital cost of vacuum systems and the production constraints of batch processing. It has long been recognized that glow discharges would play a much larger industrial role if they could be generated at one atmosphere, and in ambient air. The One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™) has these capabilities. The OAUGDP™ is a non-thermal, "cold" RF glow discharge plasma that operates on displacement currents and has the time-resolved characteristics of a classical low pressure DC normal glow discharge. Exploratory tests have been conducted on a variety of prospective industrial applications of the OAUGDP™, all at one atmosphere using air as the working gas. These applications include subsonic plasma aerodynamic effects, including flow acceleration and flow re-attachment and the electrohydrodynamic (EDH) flow control of neutral working gas; increasing the surface energy and wettability of fabrics, films, and solid surfaces; sterilizing medical equipment and air filters; stripping of photoresist and directional etching of possible microelectronic relevance; and plasma deposition.

I. Introduction

Those participating in industrial research and development need to be aware of new enabling technologies that may provide new market opportunities, or improved manufacturing methods for current products. In the recent past, Very Large Scale Integration (VLSI) and Information Technology (IT) were the focus of Research and Development in industrial and academic circles; now, bioengineering and nanotechnology have taken their place. What is likely to be the next major trend in industrial enabling technology? I would like to make a case that *Industrial Plasma Engineering* is likely to become such an enabling technology.

The industrial use of plasma has a history of well over 100 years, and examples such as ozone generation for public water supplies, arc welding, fluorescent lamps, thin film vacuum coating, and microelectronic deposition and etching are familiar industrial enabling technologies.

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Industrial Plasma Engineering has a bright future in these and other, entirely new areas for the following reasons: 1.) Plasma-related methods can often accomplish industrially relevant results more efficiently and cheaply than competing technologies; 2.) Plasma-related methods can perform tasks that can be accomplished in no other way; 3.) Plasma-related processes usually accomplish results without producing large volumes of unwanted by-products or waste materials; and 4.) Plasma-related processes usually accomplish results without producing significant quantities of toxic wastes. Examples can be found in References 1 and 2. These characteristics of plasma processing have always been advantageous; they are becoming increasingly essential in the current, progressively more stringent regulatory atmosphere, and in an international competitive environment in which the United States must depend on its high-tech skills to compete economically with low-wage, lower-tech overseas competitors.

A major reason why Industrial Plasma Engineering may be ready to take off as an enabling technology is the recent development of plasmas, including glow discharge plasmas, that can operate in air and at one atmosphere. In the past, much industrial plasma processing has utilized glow discharges at pressures below 10 torr, limiting applications to high value workpieces as a result of the high capital cost of vacuum systems and the production constraints of batch processing. I will use the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™), developed at the University of Tennessee's Plasma Sciences Laboratory [3-6], as an example of the industrial potential of plasma operation at one atmosphere. Exploratory tests have been conducted on a variety of prospective industrial applications of the OAUGDP™, which include subsonic plasma aerodynamic flow control; the electrohydrodynamic (EHD) flow control of neutral working gas; increasing the surface energy and wettability of fabrics, films, and solid surfaces; sterilizing medical equipment and air filters; stripping of photoresist and directional etching of possible microelectronic relevance; and plasma deposition.

II. The Physics of the OAUGDP™

Von Engle, et al, [7] appear to have been the first (in 1933) to report the operation of a DC normal glow discharge in air at one atmosphere. However, their procedure required initiation of the discharge under vacuum, followed by a gradual increase in pressure to one atmosphere. It also

required aggressive cooling of the cathode to suppress the glow-to-arc transition. This discharge was not stable with respect to the glow-to-arc transition at one atmosphere, and it found few if any industrial or laboratory applications.

The cathode heating responsible for the glow-to-arc instability of the von Engle discharge arises from ion bombardment of the cathode. To avoid this cathode heating, the *ion trapping mechanism* was conceived [5, 6], in which RF is applied to the electrodes at such a frequency that ions, but not electrons, are trapped between the electrodes [1, 2], and a dielectric plate is used to further suppress the glow-to-arc transition. This transformation of the von Engle discharge from DC to RF greatly reduces cathode heating, reduces sputtering and erosion of the electrodes, reduces contamination of the plasma, stabilizes the plasma against the glow-to-arc transition, and provides a form of electrodynamic trapping that increases the ion number density available for Lorentzian collisions and flow acceleration. This form of discharge was called the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™).

Numerical simulations of the OAUGDP™ by Ben Gadri and measurements of axial luminous intensity by Massines et al. [8-10], both in helium gas, showed that the OAUGDP™ has all the classic structures of the DC normal glow discharge, and that these reverse with each half-cycle of the RF. These structures include the cathode dark space, the negative glow, the Faraday dark space, the positive column, and the anode glow. The identification of the OAUGDP™ as a normal glow discharge is valuable from a plasma-physical and a phenomenological point of view, because it brings into play nearly two hundred years of accumulated observations and understanding from electrical discharge research. An important example is knowledge that the OAUGDP™, like all normal glow discharges, operates at the Stoletow point. This provides assurance that the generation of ion-electron pairs at one atmosphere in the OAUGDP™ cannot be done more efficiently. For air, this minimum energy cost is 81 electron volts per ion-electron pair formed in the plasma.

A photograph of the Mod VI OAUGDP™ reactor operating in air at one atmosphere is shown in Figure 1, and its schematic is shown in Figure 2. This reactor was designed to expose samples to the OAUGDP™ for biological (sterilization) or surface energy testing in three modes of operation: Direct exposure – the workpiece is placed on an electrode, and directly exposed to the plasma; Single pass remote exposure – the workpiece is located outside the plasma volume on a platform in the exit manifold, and active species from the plasma are convected over its surface only once. Recirculating remote exposure - the workpiece is located outside the plasma volume, and active species from the plasma are convected over its surface by a gas flow that recirculates through the OAUGDP™ multiple times.In our research with this reactor, we performed paired comparisons of direct exposure and recirculating remote exposure of samples to the OAUGDP™ active species, and found that direct exposure to the OAUGDP™ is much more effective in increasing the surface energy of materials than single or multiple pass remote exposure. Also,

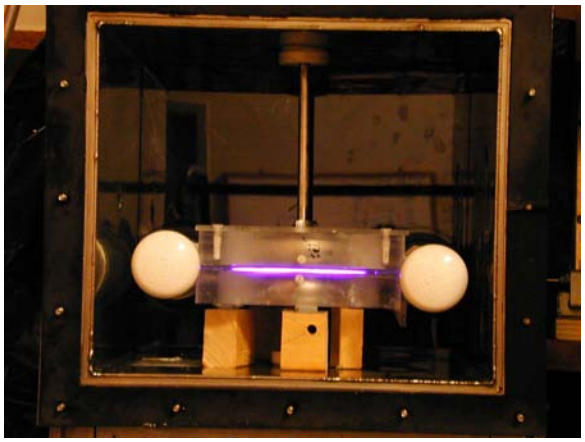
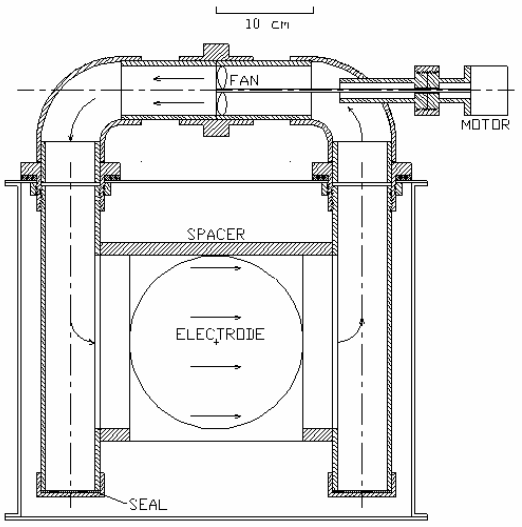
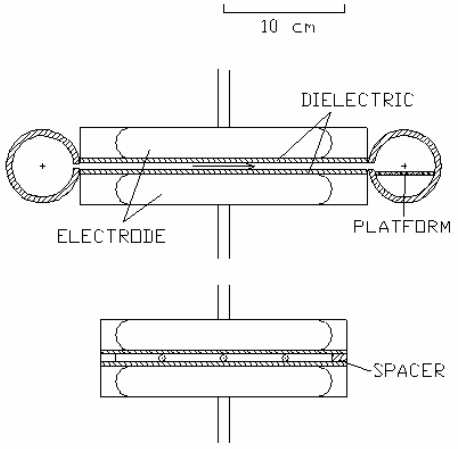


Figure 1 The Mod VI OAUGDP™ reactor operating in air at one atmosphere of pressure.



(a) Plan view of Mod VI Reactor



(b) Elevation views of Mod VI Reactor

Figure 2. Mod VI OAUGDP reactor. a) Plan view from top. b) Elevation view from front, with remote exposure platform in plenum on left, and elevation view from the side.

sterilization of surfaces by remote exposure remains highly effective by comparison with direct exposure to the plasma. We also found that microbial killing/sterilization is more effective for multiple-pass recirculating remote exposure than for single-pass remote exposure. the active species responsible for sterilization build up (or at least become more effective) upon recirculation of the working gas through the plasma.

III. Subsonic Plasma Aerodynamics and Plasma Actuators

The Industrial Plasma Engineering Group of the UT Plasma Sciences Laboratory has been engaged in research relevant to subsonic plasma aerodynamics and plasma actuators since early 1994. Two basic patents [11, 12] and a series of papers on plasma actuators for subsonic plasma aerodynamic applications originated at the UT Plasma Sciences laboratory [13-23]. A plasma actuator consists of two parallel electrode strips, one on each side of a dielectric panel, with a small displacement gap between them. An array of plasma actuators that accelerates neutral gas flow to the right is illustrated in Figure 3. Each actuator acts like a

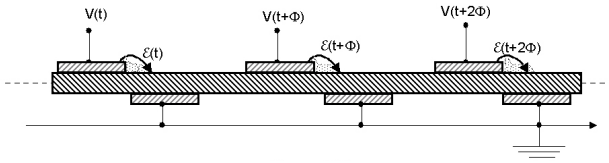


Figure 3 An array of combined paraelectric and peristaltic plasma actuators on a flat panel that accelerate flow to the right.

Glauert wall jet [24] that adds momentum, but not mass, to the boundary layer flow.

Plasma actuators can be based upon corona discharges, dielectric barrier discharges (DBDs), and the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™). In our research, we have emphasized the physics and phenomenology of the OAUGDP™, because, as a normal glow discharge, it operates at the Stoletow point [1] where the energy cost of producing an ion-electron pair is a minimum.

An early example of plasma actuators in action is shown in Figure 4 below. Figure 4a shows a smoke jet flowing tangentially to a 5 cm diameter cylinder in static air with the plasma actuators off. In Figure 4b, the actuators are energized, adding tangential momentum to the boundary layer gas flow which attaches the smoke flow to the cylinder.

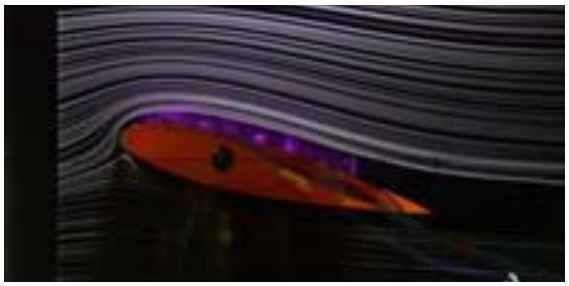
A more sophisticated example is shown in Figure 5, in which a series of eight plasma actuators have been mounted on a flexible panel attached to the upper surface of a NACA 0015 airfoil operated at an angle of attack of 12 degrees [20-23]. The images of Figure 5 were taken in the NASA Langley 7 x 11 Low Speed Wind Tunnel at a free stream velocity of 2.6 meters/second. The re-attachment of the flow is very evident, as is the stabilization of the instability of the separated boundary, which grows to form vortices and increase drag.

a) Plasma actuator off

b) Plasma actuators energized

Figure 4 Flow of tangential smoke jet over a 5cm diameter cylinder in still air. a) Plasma actuator off. b) Plasma actuators energized.

a) Plasma actuators off



b) Plasma actuators on

Figure 5. Flow re-attachment by plasma actuators on a NACA 0015 airfoil at an angle of attack of 12 degrees, actuator electrode voltage V = 3.6 kV , driving frequency f = 4.2 kHz, stream flow velocity 2.6 meters/sec.

In our experiments so far, a wide range of boundary layer and flow control phenomena have been demonstrated. The boundary layer flow can be accelerated, slowed, stopped, and diverted; the boundary layer thickness can be increased or

thinned; and we have seen suggestive evidence that the flow can not only be tripped into the turbulent regime by these EHD body forces, but also that the laminar regime can be extended by OAUGDP™-based EHD effects [12-18]. As a flow control device, the OAUGDP™ plasma actuator can accelerate a flow and maintain a bulk velocity at least 3 centimeters from the surface, thus acting as a plasma pump. We have increased the drag with an OAUGDP™ layer in the laminar regime by a factor of at least ten, and have shown drag reductions (in the vicinity of 4 meters/second) of up to 50% [16-18].

Results thus far include demonstration of flow re-attachment on the NACA 0015 airfoil illustrated in Figure 5; demonstration that the plasma actuators are best located at the leading edge of the airfoil, and not at the location of the flow separation bubble; and modeling of the plasma actuator flow as a Glauert wall jet [22]. Thus far, EHD-induced flow velocities up to 10 meters/sec have been achieved. Potential applications of EHD plasma actuators on aircraft include increasing or decreasing the lift of airfoils, increasing the stall angle of airfoils, altering the effective shape (camber) of airfoils, flow attachment for external aerodynamics on airfoils and fuselages, flow attachment for internal aerodynamics on turbine blades and ducts, and vortex suppression by active feedback control.

There are many possible applications of EHD to subsonic plasma aerodynamics, including contributions to the basic physics of EHD flow acceleration, EHD pumping and flow control for internal aerodynamics, EHD-reduced flow separation to increase stall angle, global airflow modification around aircraft fuselage for pseudo-streamlining, and shortened take-off distances and increased maneuverability of aircraft. If velocities of 30-60 meters/sec can be achieved, plasma actuators might be used to replace the hydraulic-mechanical control surfaces used for takeoff and landing.

IV. Increasing the Surface Energy of Films and Fabrics

Almost any plasma processing operation that can be performed in low pressure glow discharge plasmas below 10 Torr can also be performed at one atmosphere with the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™), provided that long mean free paths are not required. We have shown that exposure to an air OAUGDP™ is a very effective way to increase the surface energy and wettability of metals, polymeric materials, and fabrics [25-28]. Surface energies up to 70 dynes/cm can be achieved with less than one second of exposure in the Mod VI OAUGDP™ Reactor shown in Figures 1 and 2. The surface energy can display an aging effect, in which it decays over periods of days to weeks, depending on the material. If oxygen or other polar groups are added to polymeric materials, the wettability can be durable [29]. We have also observed durable surface energies above 60 dynes/cm for periods of more than a year in OAUGDP™ treated meltblown polyurethane (PU) fabrics [30, 31]. The surface energy of 1 oz/yd^2 (34 g/m^2) meltblown (MB) polypropylene (PP) fabric is shown in Figure 6 as a function of the duration of direct exposure to the OAUGDP™, and for selected times after exposure (the ageing effect).

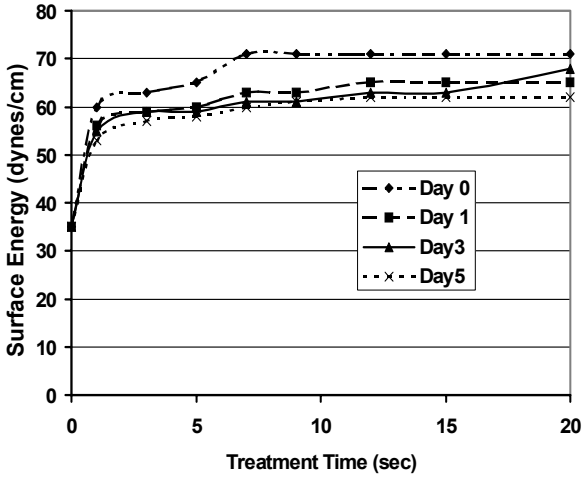
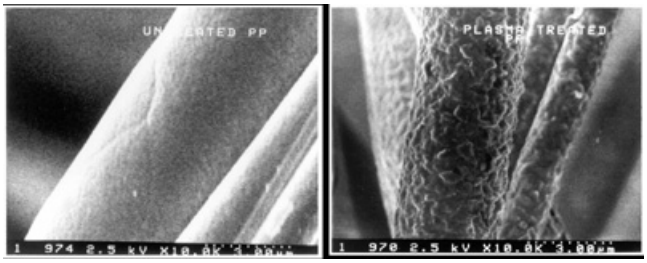


Figure 6. The surface energy as a function of the duration of direct exposure to the OAUGDP, and the aging effect for selected times after exposure for 1 oz/yd^2 (34 g/m^2) meltblown (MB) polypropylene (PP) fabric.

The physical process responsible for increasing the surface energy is believed to be the removal of the last few tightly-adsorbed monolayers of surface contaminants. The etching of the surface of individual fibers in a meltblown polypropylene (PP) fabric is illustrated in Figure 7. This surface etching mechanism is consistent with the observation that iron alloys form a coating of rust within a few minutes of plasma exposure. We have made Teflon wettable after a five minute exposure to an air plasma (water contact angle of 20 degrees). The high surface energies imparted by direct exposure to the OAUGDP™ can make it possible to use water-based inks for printing, to improve the adhesion of paints on plastics, the adhesion of electroplated layers to metals, and to make fabrics wettable and wickable. Applications to the improvement of composite materials such as automobile tires, Fiberglas, and dielectric aircraft fuselages should be possible.



a) Untreated. b) 30-second plasma exposure

Figure 7 Scanning electron micrographs (SEM) of polypropylene (PP) fibers. (a) untreated and (b) fibers exposed for several minutes to an OAUGDP CO_2 plasma. Note three-micron fiduciary scale at the lower right.

V. Decontamination and Sterilization of Surfaces with the OAUGDP™

In June 1995, the Industrial Plasma Engineering Group of the UT Plasma Sciences Laboratory began

collaborating with the Toxins Laboratory of the UT Department of Microbiology, and the UT Textiles and Nonwovens Development Center (TANDEC) to determine whether exposure to the OAUGDP™ was effective in sterilizing and decontaminating fabrics and other surfaces. Preliminary results that year [32-34] with an air plasma were very promising, and led to a series of contracts with the NIH, the EPA, and the DoD to further develop the technology. The results of these investigations were reported in various publications [35-37]. By the year 2000, this technology advanced to the point that the formation of a spin-off company was judged to be appropriate [38-43]. In August 2000, Atmospheric Glow Technologies, Inc. www.atmosphericglow.com was formed to develop commercially the use of active species generated by the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP™) to decontaminate and sterilize fabrics and solid surfaces, as well as for other healthcare and environmental remediation purposes.

During the period prior to the formation of AGT, research in the UT Plasma Sciences Laboratory demonstrated the complete sterilization at room temperature of samples contaminated with as many as 100 million microorganisms in times ranging from 5 seconds to 5 minutes [38-43]. Samples exposed in the Mod VI Reactor of Figures 1 and 2 consisted of fabrics, solid surfaces, agar medium, and filter paper. A characteristic example of such data are shown in the *E. Coli* survival curve of Figure 8. The first 20 seconds of the exposure was the expected exponential decrease due to stress on the microorganisms by active species of the plasma. At this point, however, there was a knee in the curve after which the numbers of microorganisms dropped rapidly to zero, as the result of rupture of the cell wall, as is clearly indicated in the SEM images in Figure 9.

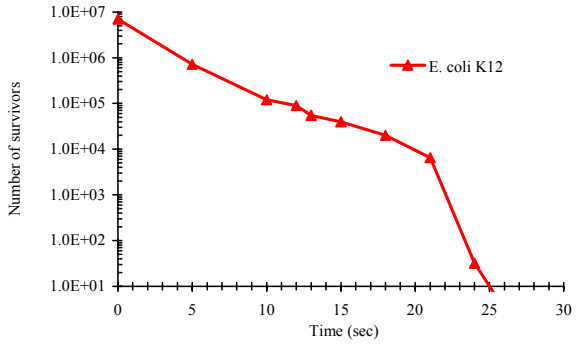
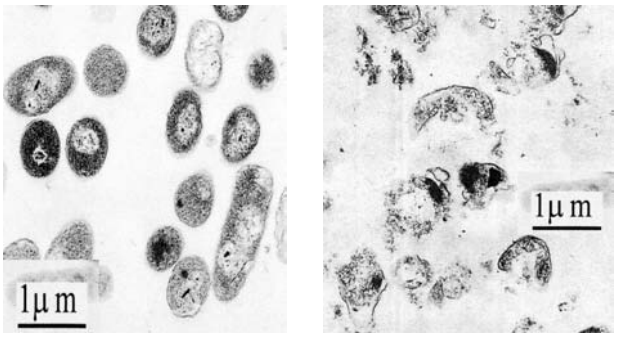


Figure 8. Characteristic survival curve for a polypropylene sample containing E. coli (6×10^6) cells exposed for the times shown to an air plasma in the OAUGDP™ Reactor for the operating conditions 10kV rms and 7 kHz.

Sterilization was observed using air as the working gas in an OAUGDP™, for direct exposure of samples to the plasma, for sealed spore and sample strips, and for samples in sealed medical sterilization bags. The killing mechanism is believed to be toxic stress due to OAUGDP™ active species during the first phase of the survival curve, followed by structural damage to the cell walls during the latter phase of exposure (shown in the SEM image). The active species most responsible for sterilization and decontamination appears to be atomic oxygen, which has a very high chemical rate constant for oxidation reactions, and one of the smallest atomic radii of any element in the Periodic Table. This latter characteristic allows atomic oxygen to diffuse rapidly and travel through small cervices to reach contaminants or microorganisms.

The UT Plasma Sciences Laboratory and later AGT has worked on several contracts that address the use of the OAUGDP™ for the decontamination and sterilization of simulants for chemical and biological warfare agents [44]. AGT later showed the effectiveness of the OAUGDP™ for the decontamination of surfaces compromised by actual CBW agents.



(a) Before exposure (b) After 30 sec. plasma exposure

Figure 9. Scanning Electron Micrograph (SEM) images of E. coli and *Staphylococcus aureus* a) before and b) after 30 seconds exposure to the OAUGDP™ operating at 10 kV rms and 7.1 kHz.

Oil of wintergreen, a chemically stable simulant of chemical warfare agents, has been oxidized and denatured by a 5-minute exposure to the OAUGDP™. Since oil of wintergreen is inert to ozone exposure, atomic oxygen is the only active species likely to produce this and other effects (i. e. sterilization). A wide range of biological warfare simulant microorganisms have been killed at room temperature and within a few tens of seconds on a variety of surfaces, including glass, metals, fabrics, and imbedded in agar.

Another decontamination technology using the OAUGDP™ is the sterilizable air filter shown in Figure 10 [45-47]. This filter utilizes a constant DC electric field across the filter material to enhance the filtration efficiency to high standards. When viruses or other microorganisms are filtered out, a OAUGDP™ plasma is generated by suitably energizing the electrodes on either side of the filter, and its active species decontaminate the filter with a few tens of seconds of exposure every 24 hours. In this way, harmful microorganisms can be filtered out of HVAC systems, and the "sick building syndrome" prevented.

VI Etching of Photoresist and Nano-Scale Etching of Surfaces with the OAUGDP™

In the work of Section IV and in Figure 7, we demonstrated the removal of adsorbed surface monolayers of

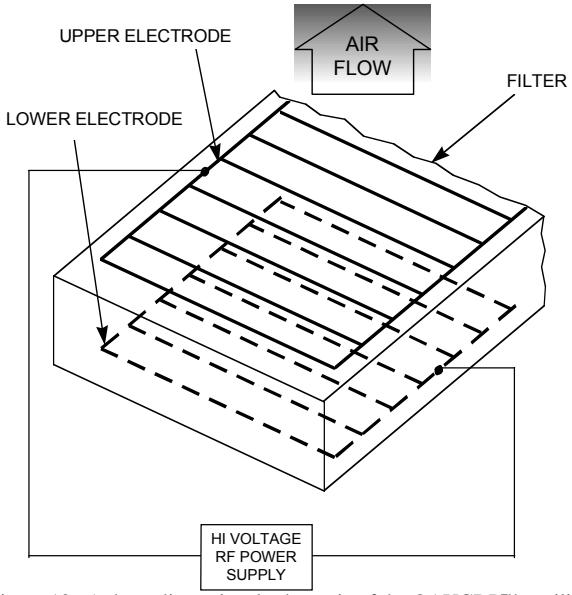


Figure 10. A three-dimensional schematic of the OAUGDP™ sterilizable air filter that recently won a 2002 R&D 100 award.

contaminants from such materials as polymers, plastics, metals, papers, fabrics, and glass [32, 48, 49]. If the surface of these materials is exposed to the OAUGDP™ for minutes rather than seconds, the active species of the OAUGDP™ etch away the surface of the material as well as remove surface contaminants [48, 49]. With the assistance of the Eaton Corporation of Rockville, MD, we determined that an air OAUGDP™ could strip photoresist at the rate of 270 nanometers/minute, under relatively low power and low density OAUGDP™ operating conditions, and do so with a uniformity of stripping rate of about 5% across the diameter of the etching plasma (15cm). These results are illustrated in the topographic profile of an OAUGDP™-etched photoresist coated silicon wafer in Figure 11. Problem areas with OAUGDP™ etching of microelectronic structures are evident in the SEM images of the wafer surface in Figure 12. These show linear structures along the gas flow direction across the wafer, and "mesas" or "spires" underneath what are presumably dust grains occasioned by the absence of clean room conditions.

Photomicrographs of polymeric materials etched with a OAUGDP™ for periods of up to five minutes have shown etching rates of approximately 500 nanometers per minute, and evidence of vertical etching in the form of spires left by etching around, but not under, small titanium dioxide inclusions which were a coloring agent in the material tested. An example of such nanoscale etching at one atmosphere is shown in the SEM image of Figure 13. These vertical spires were up to 2.5 microns high, and as small as 200 nanometers in diameter. The physical process responsible for this very anisotropic etching at one atmosphere is not understood.

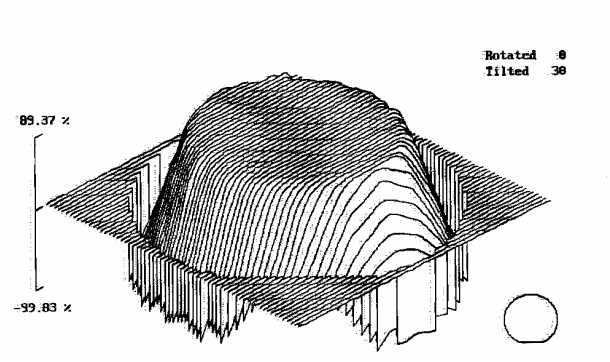
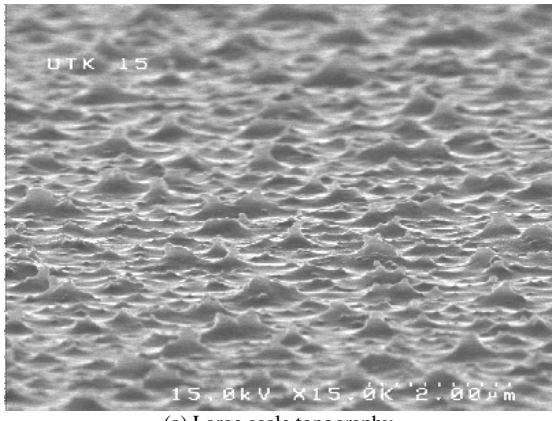
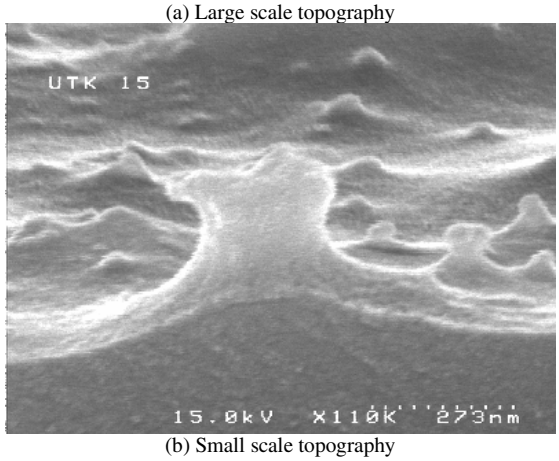


Figure 11. Isometric contour plot of etching depth across a uniform coating of photoresist on a 20 cm diameter silicon wafer (outer circle) directly exposed to an air OAUGDP™ for five minutes. Wafer and topographic analysis courtesy of Eaton Corporation.



(a) Large scale topography



(b) Small scale topography

Figure 12 Scanning Electron Micrograph images of photoresist etched for 5 minutes by OAUGDP™ air plasma. Lineations were produced by gas flow across wafer. SEM images courtesy of the Eaton Corporation.

VII MERCURY FREE FLUORESCENT LAMP

The mercury used in fluorescent lamps is a serious contaminant of landfills and groundwater. The U. S. Environmental Protection Administration (EPA) therefore has asked the lighting industry to develop lighting devices as

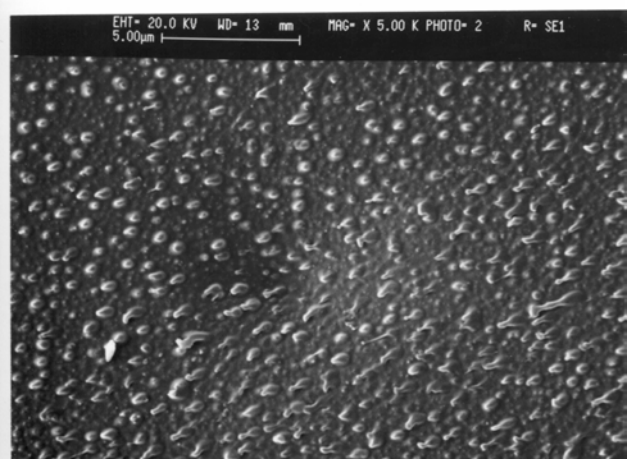


Figure 13. Scanning electron micrograph (SEM) of PET directly exposed to an air OAUGDP for five minutes, under operating conditions 5kHz and 12 kV rms. Note the sub-micron spires under titanium dioxide grains.

efficient as conventional fluorescent lamps, but without the mercury needed to provide UV radiation to excite their phosphors. At the UT Plasma Sciences Laboratory, we have developed a fluorescent lamp based on the One Atmosphere Uniform Glow Discharge Plasma, which requires neither a vacuum nor the use of mercury. This OAUGDP™ Lamp consists of a OAUGDP™ surface layer on the outside of a cylinder or on a flat panel [11, 14]. The electrodes and surface of the OAUGDP™ Lamp are covered with phosphors, which can be the same as those used in the interior of ordinary fluorescent lamps. These phosphors are excited by the ultraviolet radiation from the OAUGDP™ layer, and emit visible radiation in a manner similar to that of a normal fluorescent lamp.

VIII. Plasma Chemical Vapor Deposition (PCVD) at Atmospheric Pressure

Plasma Chemical Vapor Deposition (PCVD) can economically form thin films, and films that cannot be created in any other way. As a result, the production of PCVD coatings for microelectronics, optics, food packaging, metallurgy, and other applications has been gaining importance over the past 20 years. The OAUGDP™ has plasma and active species concentrations at least as great as the low pressure glow discharge plasmas currently used for PCVD. With the OAUGDP™, we have produced oxide coatings on metals, and SiOx coatings on polymeric films [50, 51]. Plasma chemical vapor deposition with a OAUGDP has the very significant advantages of requiring neither expensive vacuum systems, nor batch processing of fabric webs or thin films used in packaging.

IX Acknowledgements

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X. References

Hard copies of many of the references by University of Tennessee authors may be downloaded in their entirety from the Website http://plasma.ece.utk.edu by clicking on "publications" on the homepage, and then clicking on the title of the desired reference.

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