the Bell Jar
Vacuum Technique and Related Topics for the Educator & Amateur Investigator

Notes from the Vacuum Shack No. 16 March 2021

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Triode #1, a Homemade Electrical Sandwich

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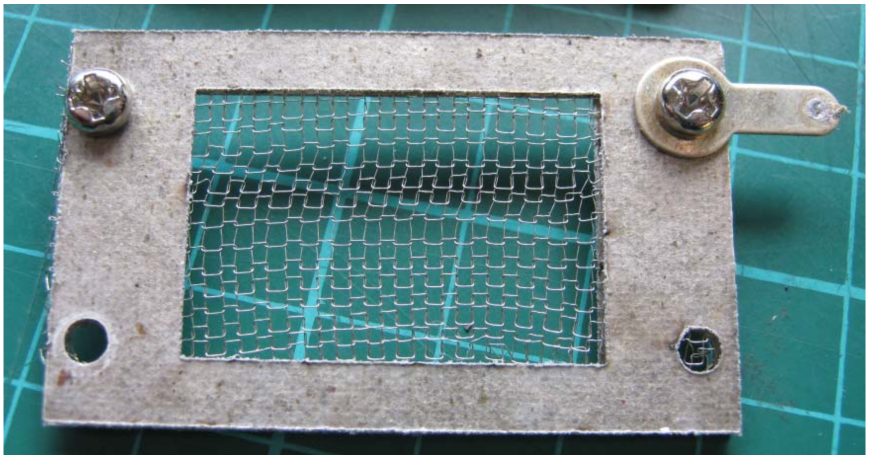
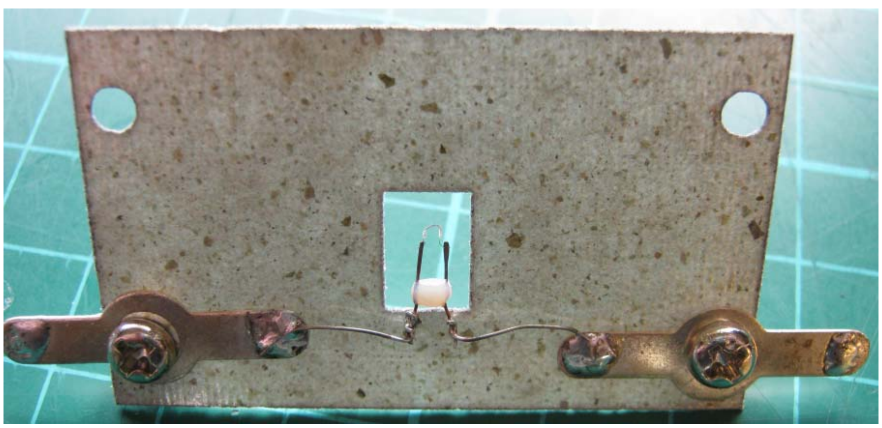
Using a 200 micron mica substrate, three elements were fabricated with the aid of a laser cutter. Each element shared a basic outline of 30 x 50 mm, with a 3 mm mounting hole in each corner.

For the anode, the centre of the element was painted with Aquadag in the shape of a square, with a connection point leading over to one of the 3 mm holes. Not easy to apply this material, it kept balling. The surface was most likely contaminated with oil and should have been cleaned using a flame (*or cold plasma – Ed.*) before proceeding. Small amount of aquadag slowly took to the surface, each was dried using a hot-air gun. A total of five layers were applied. An M3 solder tag was bolted to the assembly.

The grid was a sandwich of a fine metal mesh (possibly stainless) between a pair of mica frames, secured in two places with M3 screws. Great care was taken to remove all loose strands after the mesh was cut. The M3 bolt pushed through the hole caught on a few strands, making electrical contact. This was attached to an M3 solder tag.



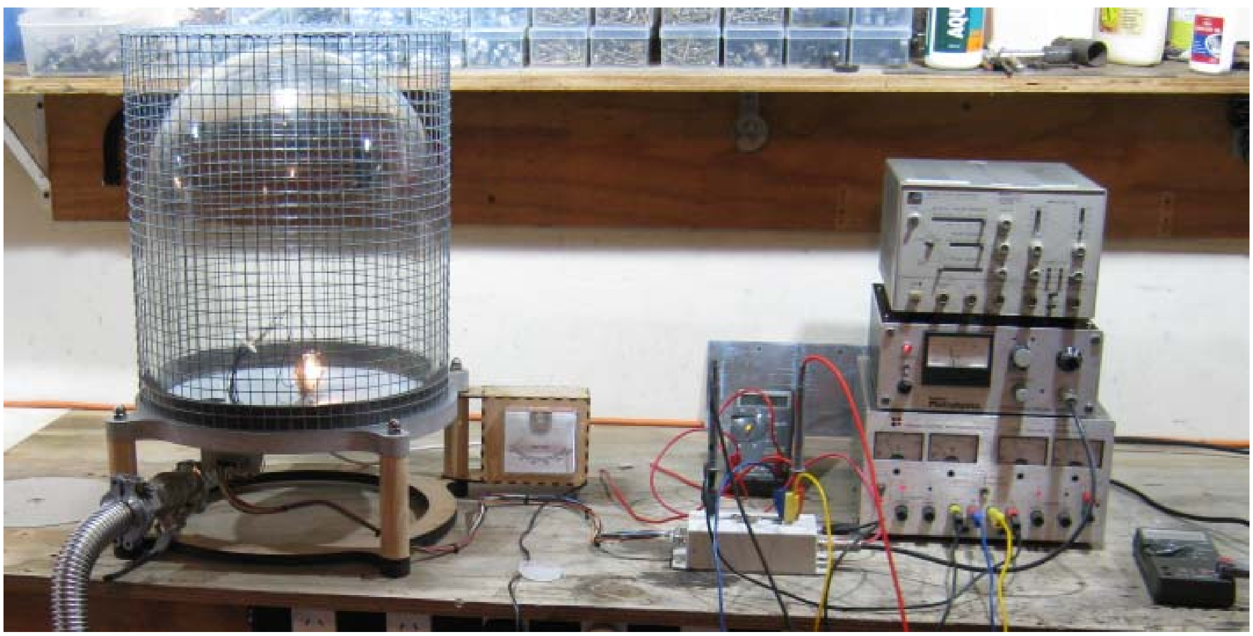
The filament element was removed from a 6 volt, 30 mA lamp, and mounted in a similar assembly as above. The filament was surprisingly robust, with a small glass bead holding the two steel wires apart, to which the tungsten filament was welded.



At the start of the build, it was unclear how the elements would be stacked. After some experimentation, two M3x20 screws were used (with nuts as spacers) to separate the elements. Terminal points on the filament and grid had to be moved to accommodate this new arrangement.

Low voltage was provided from a dual-rail powers supply; 6 volts for filament, 0 to –30 volts to the grid. High voltage was supplied from a 0 to +1000 volt adjustable supply, with a 56k series current limit resistor. A meter was in series to measure the anode current.

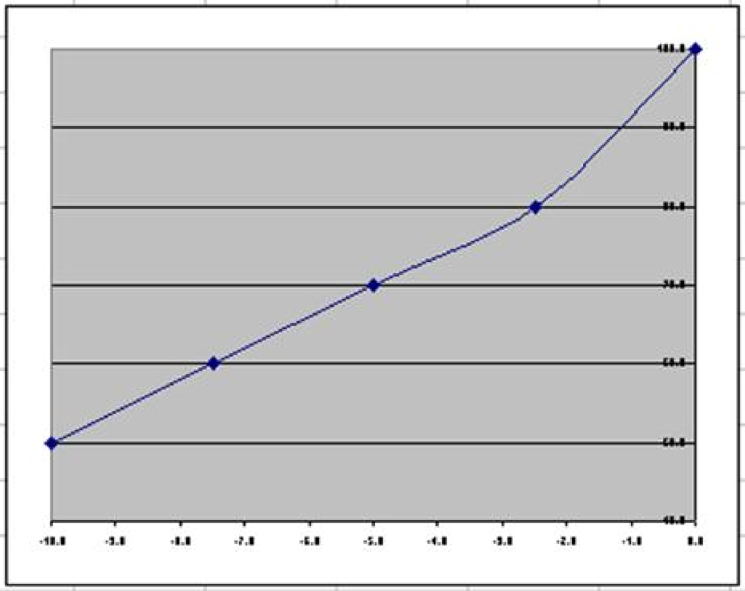
The vacuum pump had no difficulty pulling down to the normal 5 x 10^{-6} Torr range. My single largest concern was applying filament current too early, or removing it too late during the system cycling process. The photos on the next page show the stack and the unit in operation.



The data and graph on the next page show the transfer characteristics.

Va 300V, pressure 5 x 10-6 Torr

Vg	Ia (uA)
0.0	100.0
-2.5	80.0
-5.0	70.0
-7.5	60.0
-10.0	50.0



Electron Optics Kits – Then and Now

Mark Atherton's contribution reminded me of some information that was printed in the Bell Jar *in the Spring 1994 issue (Vol 3, No. 2) on the subject of electron optics kits. What follows is from that issue with some updates.*

The inquiry by Jack Sieber in the Winter 1994 issue concerning some kit form vacuum tubes that were available some time ago (1950s per Jack's recollection) has generated some interesting correspondence from the readership.

Dr. Bruce Kendall of State College, PA provided some information with regard to vacuum tube/electron optics kits that were in use in that general time-frame. Bruce writes

> Jack may be referring to the 'Harries Physikit Vacuum Tube Set' in use at MIT and possibly other universities in the late 1950s. These kits allowed construction of operating glass vacuum tubes with joints made by 'solder glass'. The kits were apparently developed by J. H. Owen Harries, a consulting engineer in Bermuda who had a background in commercial vacuum tube design.
>
> I remember seeing an example of the Physikit in Canada around 1960. At about the same time, Owen Harries published a description in "American Journal of Physics."

Unfortunately, his planned large-scale production never eventuated. His work did, however, show what could be done with relatively simple equipment.

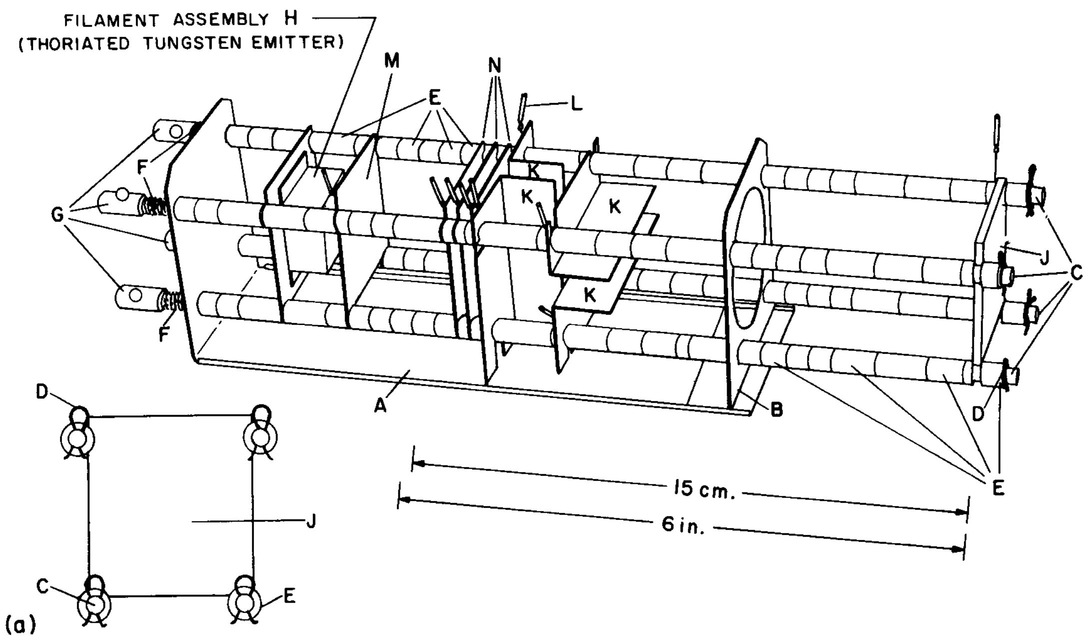
Around 1964 we tried unsuccessfully to obtain several Physikits for use in laboratory classes at The Pennsylvania State University. After reviewing various alternatives, including the later and different kit of parts also developed at MIT, I became involved in developing our own system based on reusable, planar, interchangeable electrodes. With the aid of Holger Luther (then a graduate student) and Don David (instrument technician), two kits were developed with an associated vacuum system for continuous pumping. The 'elementary' kits could be used to make working thermionic diodes, triodes, pentodes, electron guns and a cathode ray tube. The 'advanced' kit extended this to include working ion guns, electrostatic lenses of various types, ionization gauges, and electron multiplier, and a mass spectrometer.

After this equipment won an award from the American Association of Physics Teachers and was exhibited in New York in 1966, we were approached by various prospective vendors regarding commercial production. Don David left Penn State and began manufacture of the kits which were sold for several years by the Ealing Corporation, based in the Boston area. My impression is that most of them were bought by colleges in the Northeast. Some users operated them in bell jars, instead of their original housings which were based on food processing components. During this period extensive jigs and tooling were constructed in an attempt to reduce production costs.

Eventually Don's business expanded and diversified and kit production ceased. The jigs and other tooling were discarded and after a long period of storage passed into my hands so that occasional requests for spare parts could be filled.

A drawing of this kit, assembled as a CRT, is shown in the figure on the next page. Some relevant references include the following:

1. J.H.O. Harries, American Journal of Physics, **28**, 698 (1960).
2. C.K. Crawford, Review of Scientific Instruments, **36**, 844 (1965). Describes the MIT kit.
3. B.R.F. Kendall, H.M. Luther, American Journal of Physics, **34**, 580 (1966). This article, *Apparatus for Teaching and Research in Electron Physics*, discusses the design of the Penn State kit in detail.
4. B.R.F. Kendall, Journal of Vacuum Science and Technology, **5**, 45 (1968). Describes a simple pumping system for the optics kit.
5. B.R.F. Kendall, H.M. Luther, D.R. David, American Journal of Physics, **37** (1969). This article, *Apparatus for Studying the Principles of Electron Physics*, describes the integrated pumping system and electron optics kit for student use.
6. B.R.F. Kendall and H.M. Luther, *Construction and Use of a Cathode Ray Tube*, in "Experimental Vacuum Science and Technology" (Marcel Dekker, NY, 1973) This book, edited by the American Vacuum Society Education Committee, contains a wealth of useful information and experiments. This particular chapter describes a project using the Penn State kit.



Electron Optics Kit. The primary features include the main frame A, ceramic rods C, retaining clips D, ceramic spacers E, compression springs F & clamps G. Electrons are generated at H, focused by elements M, N, and are deflected by plates K. The beam is detected by phosphor plate J. (a) shows the self-aligning feature. Reprinted from *Experimental Vacuum Science and Technology*, p.196, by courtesy of Marcel Dekker, Inc.

The only contemporary equivalent of this kit that I am aware of is a set of parts which one can obtain from Kimball Physics, Inc., a manufacturer of electron and ion optical systems and what I'd call heirloom quality UHV vacuum hardware. Known as 'eV Parts', three kits of varying complexity are available as well as smaller assortments of components. All parts are UHV compatible and consist of plates, cylinders, mounting rods, insulating spacers, brackets, phosphor screens, springs, grids, etc. From these can be made ion and electron sources, Knudsen cells, electrostatic lenses, LEED equipment, Faraday cages, mass spectrometers, scanning microscopes, etc.

Prices for the kits range from nearly $1000 to over $4000. For those with deeper pockets than mine, Kimball Physics can be reached at Kimball Hill Rd., Wilton, NH 03086, https://www.kimballphysics.com/

Update on Surface Micro-Discharge Plasmas

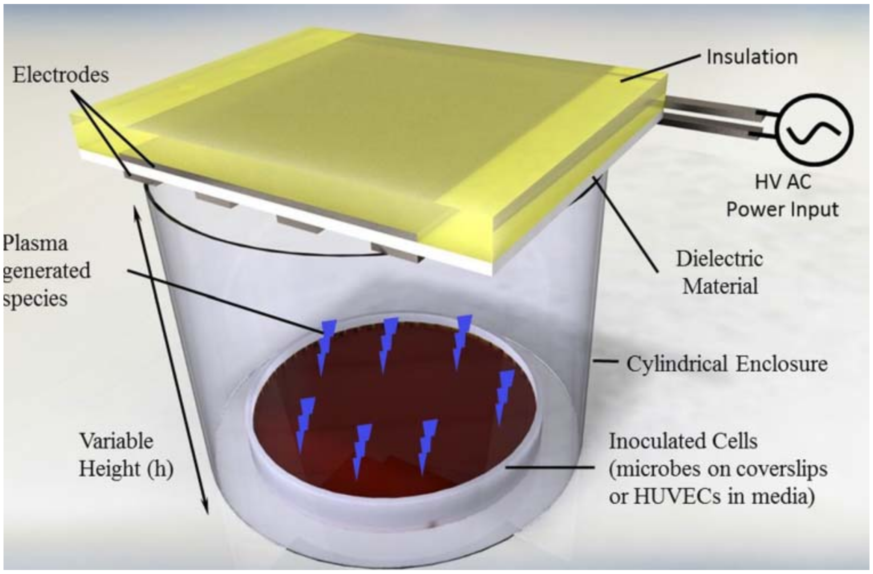
Last month I presented some initial results from my prototype SMD plasma applicator. I noted that the plasma was fairly faint when driven by my semi-homebrew high voltage source. This source uses a variable autotransformer that drives a 12 volt electronic lamp transformer which, in turn, is connected to a FLYTCL100 transformer from Information Unlimited [1]. While I mentioned that I'd look at a "beefier" supply, I decided to try a thinner sheet of the Teflon sheet which serves as the dielectric. As originally built, I used a 0.039 inch think sheet from McMaster-Carr. I had some 0.005 and 0.010 inch thick Teflon (from the same supplier) and decided to try the 0.010" sheet. This worked rather nicely. The discharge is still dim but the amount of ozone (and presumably other active species) was quite enhanced.

In the meantime, I came across a 2018 paper by Pai *et al*. [2] that describes a very simple SMD applicator using self-adhesive copper foil and, interestingly, the same 0.010" thickness of Teflon dielectric. The paper references the MINIMAX70 (NEON21) power supply from Information Unlimited. I purchased one of these (about $40 plus shipping), and, with my wire mesh grid, the performance was totally unimpressive. I did a quick substitution of 3/16 inch wide copper tape for the grounded grid and the plasma became quite visible in dim lighting. The copper tape has a more intimate contact with the dielectric.

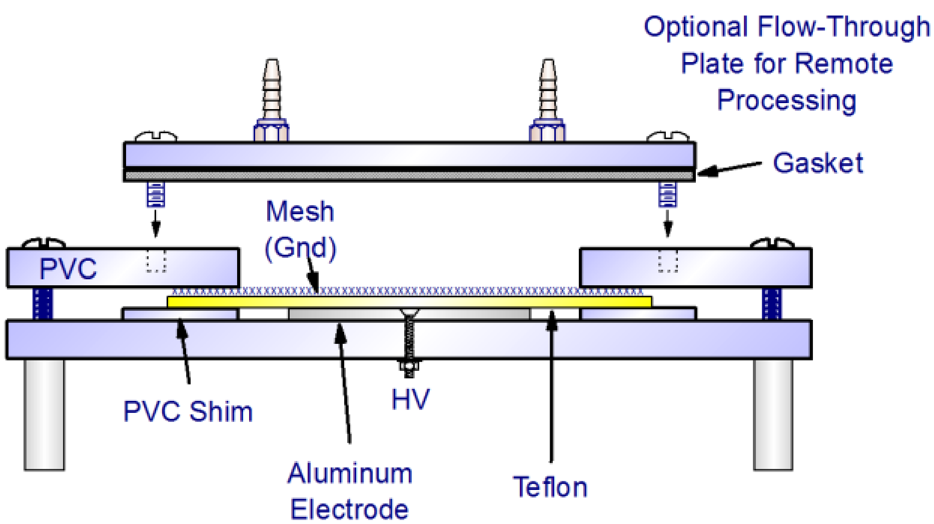
Going back to my original power supply, I tried the copper foil and it was, I'd say, similar in performance to the wire mesh but that's purely subjective based on visual observation and odor.

Getting back to the visual intensity of the plasma, in looking at some other papers, e.g.Morfill *et al*. [3], it appears quite normal. More on this paper later.

Going back to reference [2], the figure below shows the very simple experimental apparatus used by Pai's team. The plasma applicator simply sits on top of an open-top container with the test sample resting on the bottom. This was used for the plasma treatment of prokaryotic and eukaryotic cells at various distances. (The reference is open access, licensed under a Creative Commons Attribution 4.0 International License [4].)



I decided to make some changes in the construction of my prototype device. The "bumper" disks are not quite coplanar with the surface of the driven electrode. I took a piece of 1/8" PVC (same thickness as the powered electrode) and cut it to the width of the support plate. I then cut a hole in it the same size as the hole in the upper plate (4.25" diameter). Then, I acquired a piece of adhesive backed Teflon (also from McMaster-Carr) to replace the non-adhesive sheet. This bonds the dielectric securely to the electrode. The figure below shows the altered layup along with some other features.



The wire mesh is not leak tight by any means. If it is desired to minimize gas leakage from the open area out the side, a bead of silicone sealant may be applied to the perimeter of the mesh before assembly. Less messy and perhaps more forgiving to disassembly would be some combination of strips of adhesive backed copper film and a gasket made from a soft elastomer such as closed cell neoprene.

The illustration shows a gasketed plate with two gas ports that could be used to produce activated gases that would be directed to a remote chamber. Some more on this in the *Afterglow* section a bit further on.

Applications of SMD Plasmas

SMDs are finding uses in decontamination and healing. The following references describe some applications in these areas.

The previously cited paper by Morfill *et al*. [3] describes the use of a very similar structure as I've described for disinfection and decontamination in, for example, a hospital environment. As a side note, "nosocomial" in the title means hospital and community associated.

Klämpfl *et al*. [5] discuss sterilization of spores and other microorganisms using SMD.

Some of the authors of the above papers (and many others) are affiliated with Terraplasma, GmbH which specializes in SMD-type devices. Their website [6] states “Since 2011 Terraplasma GmbH, a spin-off from the internationally renowned Max Planck Society, has been offering expertise in the field of cold atmospheric plasmas. The application areas covered are medical technology, hygiene, water treatment, odour management and air purification.” Under the “About Us” tab they have listings of patents and publications.

They have some interesting products. My favorite is the *Happy Toe*, “Our solution against nail fungus.” I could have used one of those a few years ago.

The Plasma Afterglow of a SMD Cell

Rather than placing the target item in close proximity to the SMD electrode structure, the SMD can be used so activate a gas that is passed over it. The gas is then led to a chamber where the disinfection takes place. A process for the decontamination of spacecraft components is described by Müller *et al*. [7] where the inactivation efficacy of the bacterial endospore *Bacillus atrophaeus* is tested using air that is recirculated through a series connected test chamber and a SMD plasma source. The test samples are not exposed directly to the plasma but to the afterglow. Since the plasma is remote, the exposure is to the long-lifetime species. The concentration of these species is enhanced through the closed loop configuration. From the paper:

> For the indirect plasma treatment, only stable species with a long lifetime, e.g. O_3 , H_2O_2 , and NO_2 , are able to reach the target. This cocktail of long-lifetime plasma species is called “plasma afterglow” and is predominant for the treatment of targets located at a certain distance from the plasma source.

A previous study had shown that the inactivation efficacy of microorganisms increases with humidity. Therefore a water bubbler was inserted into the circuit to bring the relative humidity to 90%. The recirculation is driven by a small (3.5 l/min) membrane pump.

The SMD chamber is interesting in that the dielectric is made from quartz tubing with an inside diameter of 12 mm and length of 100 mm. Most of the tube length on the outside is covered with copper which serves as the driven electrode. Inside the tube is a stainless steel spring which makes tight contact with the tube.

With that background, I’d think that my planar SMD sandwich would serve quite well. On the other hand, I like the spring-in-tube approach. One does have to get the correct spring OD to make a good contact with the tube. That said, the key elements are the partial contact and the ability of the gas to flow through the tube. Therefore, things like BBs or a packing of stainless steel scouring pad material might be worth looking at.

Summary

As I’ve stated a number of times, it would seem that cold plasmas would be a very useful area for students and amateurs to perform some interesting studies. The equipment is relatively simple and, especially for the biology minded, the applications are very intriguing.

References

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6. https://www.terraplasma.com/en/
7. Meike Müller, Tetsuji Shimizu, Sylvia Binder, Petra Rettberg, Julia L. Zimmermann, Gregor E. Morfill, and Hubertus Thomas, *Plasma afterglow circulation apparatus for decontamination of spacecraft equipment*, AIP Advances 8, 105013 (2018). https://www.researchgate.net/publication/328208243_Plasma_afterglow_circulation_apparatus_for_decontamination_of_spacecraft_equipment

Just a Little Bit on Aerosol Dispensers for Atmospheric Pressure Plasma CVD

In the September 2020 issue I presented some information on thin film chemical vapor deposition using essential oil precursors. I showed a tentative representation of a plasma jet with a simple applicator for a precursor in aerosol or vapor form. The applicator could also be used with gases.

As I had mentioned, the use of essential oils as a plasma enhanced CVD (PECVD) precursor would seem to be a logical choice for amateur experimenters as they have practical uses in the formation of anti-microbial films as well as in other applications. Unlike the more semiconductor oriented precursors, they are also more readily available and are considerably less toxic. Essential oils that are frequently referenced in the literature include 1,8-cineole, linalool, eugenol, carvacrol and others. I've built up a small inventory and, I have to admit, my home lab smells really nice at times. In that article I also noted that most of the literature that I've come across involving essential oils refer to parallel plate DBD reactors as opposed to the APPJ configuration.

The usual method of injection for APPJ devices is into the plume at the exit of the jet. For the parallel plate DBD, the precursor can be deposited on a substrate immediately before being exposed to the plasma or be injected through the grounded electrode that is adjacent to the surface to be coated.

A very useful and recent (2020) paper is a review of aerosol-assisted atmospheric pressure plasma deposition by Palumbo, *et al*. [1]. This includes many practical examples and a lengthy list of references. One study that is referenced in the paper [2] deals with wound healing tests using a medical APPJ device (J-Plasma by Apyx Medical) with a nebulizer normally intended for the injection of samples in chromatography (Burgenor Reseach). The nebulizer and plasma jet are parallel to each other and inject into a 19 mm ID acrylic tube. A production version of this is represented by the BioDep equipment and process by TheraDep [3]. This is intended for the deposition of coatings on medical devices such as high density microplates that are used for diagnostic tests. A video on the web page shows the process in action. The production device replaces the plasma jet with two corona points that are placed on either side of the nebulizer nozzle. An experimental version can be used for wound healing where the device simultaneously performs the functions of sterilization, coagulation and applying a biologically protective coating.

Getting liquids turned into aerosols or vapors is an area of increasing interest based on the seemingly infinite number of liquid (and solid) precursors that are being used in industry. For liquids, the methods generally involve bubblers, direct vaporization and various types of aerosol generator including gas driven atomizers as well as ultrasonic and electrostatic methods.

First off, I have to say that doing web searches for information on vaporizers turned up a lot of references to various e-cigarettes, bongs and so forth. More mundane applications include inhalers and perfume atomizers. Very nice nebulizers are commercially available for chromatography but they are also quite expensive.

In looking at the options, I figured I'd concentrate on two approaches. The first would be more quantitative method (exactly known mass per unit time) and, second, a less quantitative but reproducible method. For the former, I'm looking at feeding an atomizer/vaporizor with liquid from a syringe pump that I have in my stack of stuff. For the latter I've begun looking at adapting a commercial gas driven nebulizer for essential oils. I'll start with the second one since it's progressed beyond the thinking about stage.

There are, as far as I know, two types of commercial device for atomizing essential oils. In one, the oil is mixed with water and the nebulizer works with an ultrasonic transducer, pretty much like an ultrasonic humidifier. This is not what we want. The second type draws the oil from a small bottle with a suction tube. Air, from a small compressor, blows through an orifice across the top of the draw tube, atomizing the liquid. This is like the action of a perfume atomizer.

In the devices I've seen, the spray is directed toward an impact barrier. This creates a nice fog at the outlet without any apparent large droplets. There is a residual that forms on the barrier. This drains back into the bottle.

No frills essential oils nebulizers are pretty inexpensive. I'm looking at two. The first one, in hand, is shown in the figure to the left. The wooden box contains a small air pump and some electronics to control start/stop and charging the self-contained battery. The important part is the glass piece. This has a tubulation (not visible) that connects to the pump through the box.

Inside this piece is the nebulizer. At the top is the outlet. The metal cylinder joins the nebulizer to the supply bottle. The draw tube dips to the bottom of the bottle. I measured the pump displacement and it's about 1.5 liters/minute.

The device emits a nice stream of aromatic fog. I connected 6 inch a piece of 3/16" ID silicone tubing to the outlet and the fog made it through the tube quite well with no readily apparent condensation. Of course, it may be beneficial to add a heated vaporizer to the line.

Since the carrier gas should be inert (I use argon), the pump was superfluous. Based on some consumer literature, the consumption rate, with the stock pump, should be on the order of 1cc/hr.

The next steps will be to set up a low flow rotameter to my argon supply and connect the device to my APPJ. More on that next time.

References

1. Fabio Palumbo1, Chiara Lo Porto, Francesco Fracassiand Pietro Favia, *Recent Advancements in the Use of Aerosol-Assisted Atmospheric Pressure Plasma Deposition*, Coatings 2020,10, 440; doi:10.3390/coatings10050440. https://www.researchgate.net/publication/341035075_Recent_Advancements_in_the_Use_of_Aerosol-Assisted_Atmospheric_Pressure_Plasma_Deposition
2. L. O'Neill, D. O'Sullivan, M. Fourkas and J. Tartaglia, *Evaluation of the J-Plasma Electrosurgical Device Combined with Nebulized Collagen for Burn Healing in Rodents.* Plasma Med.2018, 8, 365–377. http://www.dl.begellhouse.com/download/article/1a26f4d57b516d57/(5)PMED-28625.pdf
3. https://www.theradep.com/

Articles of Possible Interest in *Vacuum Technology & Coating* Magazine

February through May 2021
Difficult Deliveries
This four part series deals with the delivery of solids and liquids into process systems. The February column series looked into the issues and solutions to the delivery of solid (and to some extent liquid) precursors from temperature controlled ampoules using a carrier gas to drive the vapor into the process chamber. The March column covered the use of optical and acoustic transducers for monitoring and controlling the partial pressures of the precursor vapors in the carrier gas. The April column covers various types of vaporizer. Finally, the upcoming May column will deal with mass flow meters and controllers for liquids. These include pumps, specialized thermal MFCs and MFCs based on the Coriolis effect.

Articles may be accessed at http://vtcmag.com/. Scroll to the bottom of the page to the back issue selection box. Look for my columns and you can probably find other articles of interest in each issue.

End Notes

I haven't forgotten about the pseudospark and saddle field sources (the stuff that involves vacuum) but I've just been having a lot of fun with the atmospheric pressure plasma stuff. Next month's issue will have more vacuum stuff.

As usual, comments and contributions are welcomed.

Steve