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

Notes from the Vacuum Shack No. 5 April 2020

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Look Squirrel!
Atmospheric Pressure Dielectric Barrier Discharges

Actually I don't tend to get distracted by shiny objects but atmospheric pressure plasmas have recently gotten my attention. Gotten my attention *again* is probably more accurate. In 1965 an article entitled *Corona Chemistry* appeared in "Scientific American" [1]. While the term "corona" was used, what the authors were describing was really a dielectric barrier discharge or DBD. The article describes how the plasma can be used as a chemical catalyst. The link at the reference leads to the full article. For those without access rights to the full article, a picture of the reactor is shown. This shows vegetable oil that is being hydrogenated in a hydrogen plasma. The article interested me enough that I clipped it from the magazine to save.

Other than this example, I had no idea of the breadth of the application space for the DBD until Prof. Sing Lee introduced me to the book "Plasma Technology in Emerging Economies" [2]. This book was briefly reviewed in the January 2020 issue of this newsletter. While my attention was first drawn to the chapters on Dense Plasma Focus (DPF) devices which dominate the first half of the book, the latter chapters have a substantial focus on the dielectric barrier discharge and atmospheric pressure plasma jets (APPJ). The discussions range from very simple set ups to applications in materials modification, thin film deposition and medicine.

One result of this was my current series on plasmas in *Vacuum Technology & Coating*. The articles in the February and March 2020 issues dealt with the basics and process configurations of low pressure plasmas. The April article covers the characteristics of non-equilibrium, low-temperature atmospheric plasmas and the characteristics and uses for corona discharges. The

May article will deal with DBD and June will cover APPJ.

This all led me to begin putting together some simple apparatus for DBD and APPJ. In addition to learning some more about the characteristics of these animals, I also figure it would be a nice way to get familiar with some voltage, current and optical emission diagnostics without the added complexities of kiloamp/megawatt impulse discharges as with DPF and IPD plasma guns.

The next section provides a brief overview of low-temperature plasmas and DBD.

Low-Temperature Atmospheric Pressure Plasmas and DBD

Starting with Siemens' discovery of the dielectric barrier discharge in 1857, this mode of plasma generation was primarily used for ozone production. Daphne Pappas, in her 2011 review [3] noted the rise of papers over the past few decades. Prior to the late 1990s, academic papers on atmospheric pressure plasmas were sporadic and in the single digits each year. In 1997 the figure rose to 68 and then, by 2009, there was a further 10-fold increase. This is because the DBD atmospheric pressure plasma was found to have myriad applications in materials treatment, film deposition, pollution control and for medical and biological purposes.

In a 2007 article, J. Reece Roth [4] stated "Any plasma processing task possible with a glow discharge in vacuum can also be performed by a glow discharge at one atmosphere, provided that long mean free paths are not required." Pappas added:

> Industrial plasma engineering utilizing atmospheric equipment has a promising future in materials functionalization, deposition of organic and inorganic coatings, and sterilization of biomaterials and biocidal materials for the following reasons:
>
> - Unique chemical environments can be obtained through the formation of free electrons, radical, and charged and excited species;
> - Provided a plasma system is available, the process cost and equipment maintenance is very low; and
> - Plasma processes performed under atmospheric pressure conditions are ecofriendly as the formation of by-products is minimal, while the produced waste is insignificant.

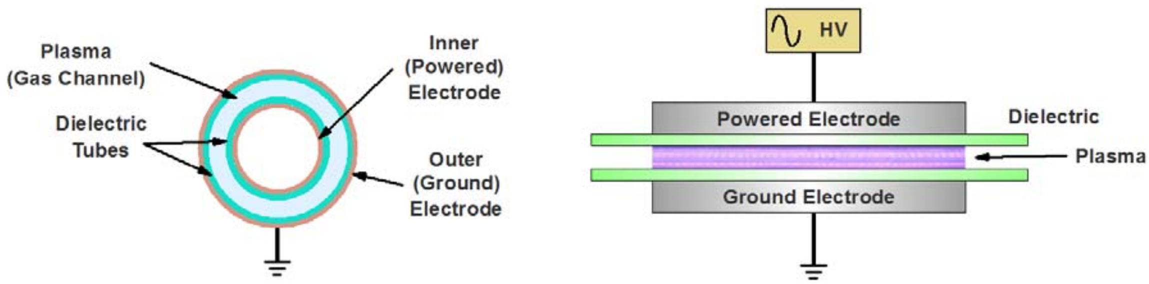
The common low-pressure parallel plate electrode glow discharges are marked by having an electron temperature (Te) that is higher than the molecule or ion gas temperatures (Tg) which are close to room temperature. As the pressure increases, the electron and gas temperatures merge as the mean free paths decrease. This will normally result in a thermal (hot) plasma or arc where the electron and gas temperatures are in equilibrium. At atmospheric pressure, temperatures can approach 10,000 K.

The intent of the DBD is to produce a uniform plasma discharge while avoiding the conditions for an arc breakdown. The function of the dielectric is to permit the build-up of charges on the surface to create a discharge but then to cause the discharge to extinguish as the voltage between the electrodes falls. During the process, the electron temperatures will be high but the gas temperature will remain low, essentially at room temperature. In these conditions we have a non-equilibrium plasma.

The potential that is applied to the electrode may be in the form of pulses or alternating (sinusoidal) current. The frequencies may range from the line frequency (50 or 60 Hz) up through the high frequency (e.g. 13.56 MHz) range. kHz range power supplies are very common.

Other strategies may be used to produce a similar effect. One is the use of very fast rise/fall time pulses (nanosecond range). Here the quickness of the pulse prevents the transition to an arc, even without a dielectric covering.

The figure below shows the general configuration of coaxial and planar DBD electrode structures. The figure on the left represents Siemens' ozone generator. The configuration is still widely used for ozone generation and for pollution mitigation. On the right is the planar configuration. Here, both electrodes have dielectric coverings. The dielectrics may be glass, quartz, mica, ceramic or even polymers such as PTFE. Generally, the dielectric layers are 1-2 mm in thickness. In alternate configurations, one of the electrodes may be uncovered or the dielectric may be placed at a midpoint position.



In the usual situation, the body of the plasma is not homogenous but generally consists of filaments (also called micro-discharges) that are distributed across the gap. This form of plasma is often referred to as filamentary DBD (FDBD). These filaments are due to the accumulation of the electrons across the dielectric and typically have a diameter of about 100µm and a lifetime of 100-200 nanoseconds. The filamentary structure is depicted in the electrode gap. By adjusting the parameters (gap, gas, voltage, frequency, etc.) it is possible to produce a uniform glow.

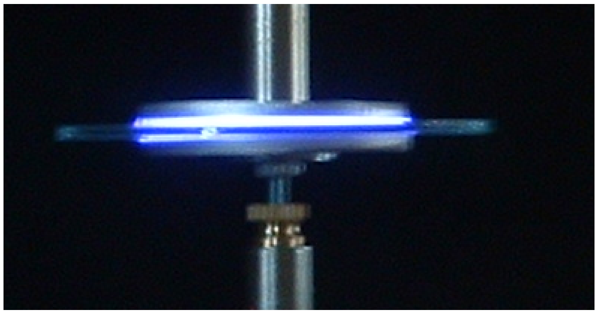
A Simple DBD Experiment Platform

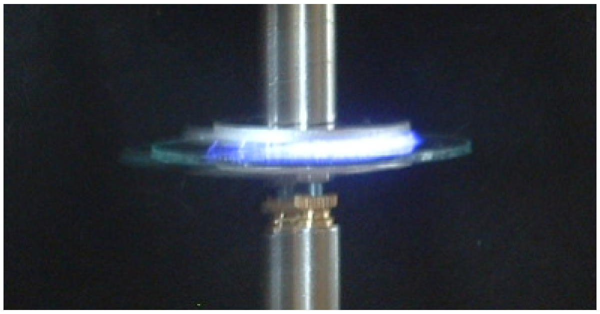
The picture on the next page shows my simple device for experimenting with DBDs. Most of it is assembled from materials I had hanging around. The wood base is a bamboo cutting board (the same type I used for the BVES vacuum trainer). This is about 6 x 13 inches. I used four 4.25" long threaded standoffs to hold the 3/8" thick PVC platform that supports the upper electrode. This platform is 5 x 9 inches. I have several sizes of 1/8" thick aluminum disks for the electrodes. The ones shown are 2" in diameter. The upper electrode is fixed to the metal standoff and the connection is to the binding post at the top. The lower electrode's height can be adjusted by means of a screw and locking knurled nut. The lower electrode is grounded and the connection is brought out to the binding post to the left. In the picture I have one 1.5 mm x 3-1/4" glass disk as the barrier. The upper electrode is bare but a second can be added if bonded to the electrode.

The brass compression fitting serves as an inlet port if I want to work with a gas other than air. Usually this is helium or argon. This is used in conjunction with a 4” diameter clear acrylic tube that goes between the base and the PVC plate.

I just finished this the other day but initial results are encouraging. For power, I am using the Amax 150 watt HD150-120 lamp transformer (20 kHz at 12 volts output) that was mentioned last month. This drives a TR-301H flyback transformer (1:286 turns ratio) from Information Unlimited (https://www.amazing1.com/transformers-high-voltage-high-frequency.html). I have not yet tried 60 Hz (e.g. oil burner transformer with Variac) but will shortly. I also have a variable pulse width, variable frequency, variable voltage power supply in the works.

The pictures below show the discharge in two configurations. On the left is a fairly uniform plasma (the filaments can’t be discerned with the naked eye) with the gap set at about 1 mm. In the photograph to the right I tilted the lower electrode a bit from about 1 mm to 2+ mm across the gap. As the gap increases from right to left you can see the change in density of the filaments.





Planned diagnostics include a Pearson 2877 wideband current transformer (placed around the ground lead) and a low inductance high voltage probe. A low value resistor in the ground leg may also be used to monitor the current when used with a voltage probe.

Summary

Well, this isn't vacuum but to repeat the quote by Roth "Any plasma processing task possible with a glow discharge in vacuum can also be performed by a glow discharge at one atmosphere, provided that long mean free paths are not required." Given this, there are ample opportunities to experiment with atmospheric pressure plasmas for materials treatment (e.g. enhancing wetting), coating (plasma enhanced CVD) and biological studies. There are also significant complexities in the discharge itself (filamentary vs. the various forms of homogenous discharge) to keep one occupied. This is an area where an amateur might actually be able to produce some original research using a relatively minimal set of esoteric tools. Finally, perhaps the DBD could find a place in the plasma-oriented STEM programs that are in place at educational institutions.

Next month I'll report on any additional progress here and will also cover one or two simple APPJ devices. Unless another shiny object appears.

References

1. John A. Coffman and William R. Browne, *Corona Chemstry*, Scientific American, Vol. 212, No. 6 (June 1965), pp. 90-99 https://www.jstor.org/stable/24931911?seq=1
2. Rajdeep Singh Rawat (Ed.), *Plasma Science and Technology for Emerging Economies – An AAAPT Experience*, Springer Nature, 2017.
3. Dapne Pappas, Status and potential of atmospheric plasma processing of materials, J. Vac. Sci. Technol. A 29(2), Mar/Apr 2011.
4. J. R. Roth, S. Nourgostar, and T. A. Bonds, *The One Atmosphere Uniform Glow Discharge Plasma (OAUGDP)—A Platform Technology for the 21st Century*, IEEE Trans. Plasma Sci. 35, 233 2007.

New Book on Nikola Tesla: *Nikola Tesla and the Electrical Future*

The March 2020 issue of "Physics Today" contained a review of Iwan Rhys Morus's 2019 biography of Nikola Tesla. The review, by Richard Bradley, notes that this biography comes at Tesla from a different perspective from previous treatments. He writes:

> In *Nikola Tesla and the Electrical Future*, historian of science Iwan Rhys Morus examines this "man of apparent contradictions" in the context of the cultural and technological revolutions of the late 19th century. The book includes an extensive bibliography and endnotes; the author clearly is an authority on the history of electrical technology. Morus convincingly dispels the image of Tesla as a man out of time and replaces it with a more realistic view of a brilliant inventor who was nevertheless the product of his era, his interests and activities shaped by the world in which he lived.

Check out the full review at https://physicstoday.scitation.org/doi/full/10.1063/PT.3.4432
The publisher is Icon Books. My copy is on order.

Richard Hull’s Fusor V Project

Building on his learnings from Fusor IV, Richard has begun construction of Fusor V. A carry over is the Tylan General capacitance diaphragm gauge (CDG). This is a 0.1 Torr full scale temperature controlled unit. The diffusion pump is being replaced by a Pfeiffer TCP40 turbo-molecular pump that he purchased from Bill Connery in 2018. (Bill passed away in 2019.) The pump had 2946 hours on it and had set idle for more than 2 years. It was due for lubrication of the bearings. The bearing oil (100 ml) was purchased from Duniway Stockroom. It only needed 10 cc.

Richard was delighted at the ease with which the new pump evacuated his chamber and it was easy to reach the proper zeroing pressure for the gauge. Richard had a question regarding the gauge: “Can the capacitive diaphragm be damaged or knocked out of calibration i.e. keeping the gauge at deep vacuum for protracted periods?”

My answer: The reference pressure behind the diaphragm is on the order 10^{-8} Torr, sustained by a chemical getter. At low pressures, a decade or so below the resolution, the diaphragm is flat. There is absolutely no harm in keeping the gauge under high vacuum. On vacuum pressure test stands and tools, a standard procedure is to always keep the gauges (if possible) under high vacuum when not being used. The separation between the diaphragm and the ceramic substrate that has the electrodes is very small. When subjected to high pressures the diaphragm will actually lay flat on the substrate. In the case of MKS Baratrons®, the overpressure limit is 45 psig. Above that pressure there's the possibility of cracking the ceramic. When that goes, so does the diaphragm. When subjected to high pressures (>1 atm but below the break point) it is good practice to rezero. Zeroing should be done at a pressure a couple of decades below the gauge’s resolution.

Follow Richard’s project at https://www.fusor.net/board/viewtopic.php?f=6&t=13176

Simple Mini System for Evaporation

Bill Connery

Bill was one of the early contributors to the Bell Jar. This article was originally presented in Volume 1, Number 3 (Autumn 1992). As noted above, Bill passed away in 2019.

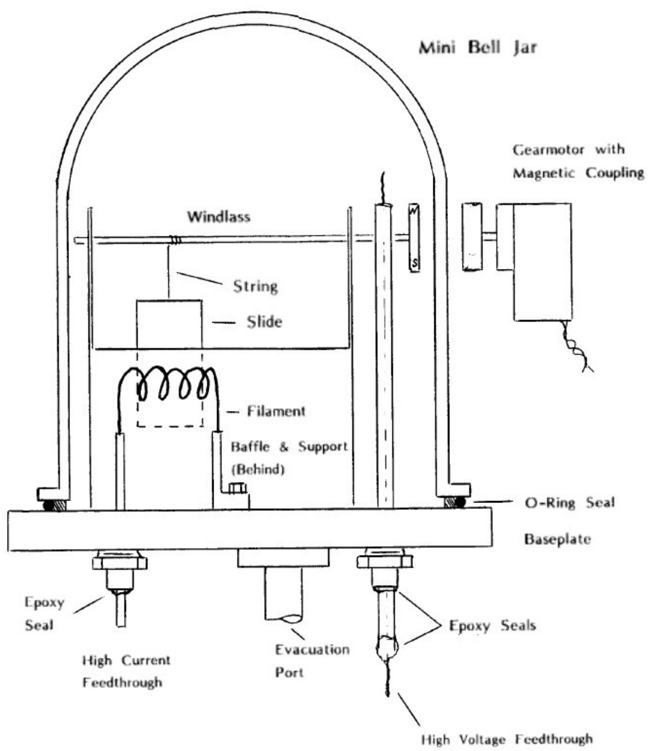
The following is a description of a simple evaporation chamber that was developed to make small optical components. This system has proven adequate for the coating of small mirrors, depositing antireflection coatings, and - with a simple magnetically coupled motion feedthrough - fabricating graded neutral density filters.

The incentive for building this system came from the need for a linearly graded optical filter. Commercially manufactured filters were too expensive, but given the availability of a small vacuum system, making the required fixturing and a small chamber seemed to be a reasonable and affordable solution.

The vacuum system itself consists of a 2 inch CVC MCF61 diffusion pump. Backing this is a small (2 cfm) Leybold mechanical pump. An Edwards thermoelectric cold trap is used to prevent backstreaming of the oil, Inland perfluoropolyether. The thermoelectric element maintains the trap at about 0° C.

For the evaporation chamber, a small bell jar was improvised from a glass cover which came from a surplus aircraft gyro. The jar is about four inches in diameter and six inches high; walls are a quarter inch thick. The only marking on the jar is the manufacturer's name, Kearfott. The base plate was fabricated from half inch aluminum plate.

An O-ring, mounted in an aluminum split ring, seals the jar to the base plate. An alternative to the gyro cover could be the sort of covers used for electric meters or explosion proof lamps. As these vessels are not designed for vacuum use, care should be exercised. A shield (e.g. fabricated from Plexiglas) should always be used with glass chambers, even ones designed for vacuum. The figure below shows the chamber with its associated fixturing.



There are two feedthroughs. One is for a high voltage electrode which is used for outgassing during the pumpdown cycle. The other is a high current feedthrough for the "hot" lead to the source filament. Both of the feedthroughs are fabricated from standard stainless steel Cajon brand fittings: 1/4 inch male pipe thread to welded tube. For the high voltage feedthrough, a piece of ¼ inch od glass capillary tubing is epoxied into the fitting. A piece of bare nickel wire is passed through the bore of the tube and is likewise sealed with epoxy. The high current feedthrough is made in a similar manner: a piece of 1/8 inch square copper bar is first coated with a thin coating of epoxy (where it will pass through the fitting); once the glue has set, the bar is inserted into the fitting and more epoxy is applied. An important point to remember in the fabrication of the feedthroughs is to confine the epoxy to the area above the threads: epoxy in the threaded area, which is subject to strain when the fitting is tightened, has a tendency to crack.

For providing uniform coatings, the item to be coated is suspended a short distance from the evaporation source, a coil of tungsten wire. The means for producing the variable graded filters is also shown in the illustration. Here the substrate (typically a microscope slide) is held by a string below sort of a windlass. At the end of the shaft is a magnet which provides coupling to a rotating steel bar (driven by a small gear motor) outside the chamber. A baffle is placed in front of the substrate. Initially the substrate is in the low position, behind the baffle. Once the evaporation cycle has begun, the motor is activated and the substrate rises from behind the baffle. Thus the deposit will be thicker (more opaque) at the top and thinner at the bottom.

While a thermocouple gauge is incorporated in the system, its use has not been required as the high voltage outgassing discharge gives a good idea of what is going on in the chamber. During the pump down cycle the heater is also degassed. Once blackout of the discharge occurs the evaporation cycle may be started. It is important to quickly progress from the outgas phase to the start of the evaporation cycle.

Research Papers On Line

Between December of last year and January I checked out a few sources for research papers. Among them were ResearchGate (https://www.researchgate.net/) and Academia.edu (https://www.academia.edu/). The former has no fee but, for most papers, you have to make a request to the author for a copy. That has had mixed success. The latter does have a nominal subscription fee to take advantage of all of the features. I've been quite happy with the service and have found a lot of material in my areas of interest.

There are other features. For example, based on my searches, I get email notifications of papers that may be of interest. Also, they send out notices if my name shows up in a paper. What they look for is "Stephen" and "Hansen" adjacent but in any order, with or without a comma. Using this, I get a lot of references but they are almost always someone else and most of the papers are in fields having nothing to do with physics. Some are stomach turners. So far, I did find one paper, *Synthesis and Characterization of Nano Aluminum Particles*, that was published by a team at the Mar Athanasius College of Engineering, Kothamangalam, India. The reference was to my booklet on exploding wires. That was kind of interesting.

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

March 2017
Leak Free? It Depends. Norman Milleron stated "One man's vacuum is another man's sewer." The degree of needed vacuum quality is dependent upon the application. Best practice in one application might be a sin in another.

April 2017
Leak Free? Some Troubleshooting Dos and Don'ts. This article covers some hints for avoiding problems where possible and identifying them if and when they occur.

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

Vacuum System for Sale

This vacuum system that was used only one time to de-bubble potting compound. Since then it has been in storage. It consists of the following items:

1. Hyvac Model 91138-001 Mechanical pump
2. 12" dia. by 12" high Pyrex bell jar and hand cut gasket
3. Steel bell jar base and stand with hose coupling and a bleed-off valve and
4. mechanical gauge (gauge needs oil refill, half empty but working)
5. Implosion cage for the bell jar
6. Rubber hose to couple pump to bell jar stand with hose clamps
7. Almost a full quart of Hyvac mechanical pump oil

These can be seen in the picture (more pictures can be provided). The system is completely operational and pumps the gauge down to its lowest reading. Asking $1200 plus actual shipping charges. Crating is included. Reasonable offers will be considered.
Please email, text or call Joe Malek if you have interest.
joe.malek@eleksys.com Cell/Text: (727) 871 4751

Your Projects

The 10 volumes of the printed *Bell Jar* had a high proportion of reader projects. If you are working on something that is vacuum related that may be of interest to others, please contact me.

That's it for this month. Stay healthy!

Steve