the Bell Jar

Vacuum Technique and Related Topics for the Educator & Amateur Investigator

Notes from the Vacuum Shack **No. 18 May 2021**

In this issue:

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Vacuum Microwave Drying and Woodturnings

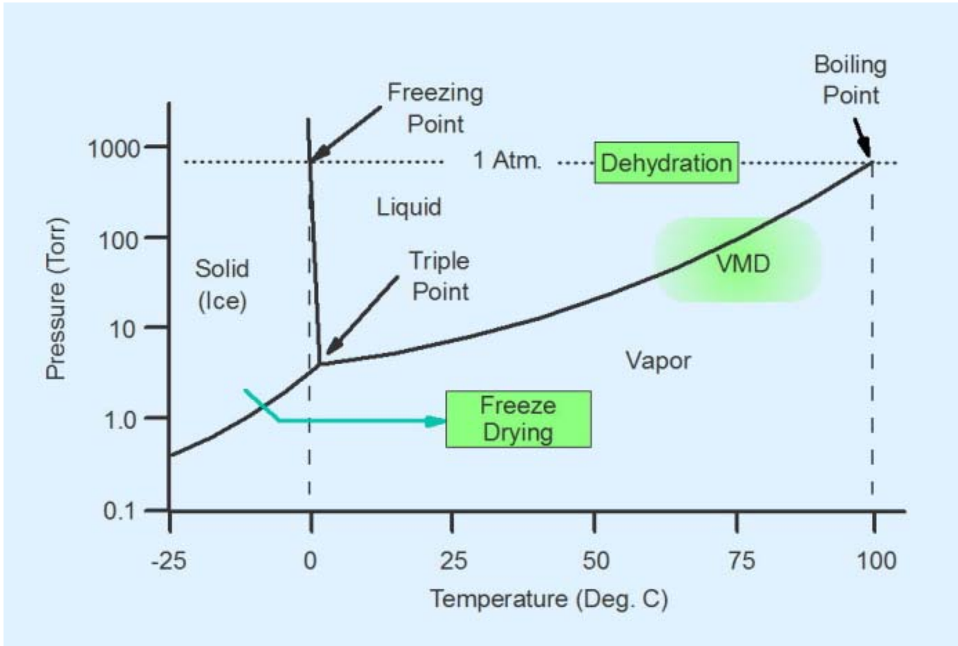
This article has two parts. The first provides an overview of vacuum microwave drying (VMD) and where that process fits relative to freeze drying and atmospheric pressure drying. The second part covers the work of Earl Rich who, in the Spring 1998 issue of *the Bell Jar* reported on his work with the vacuum microwave drying of wood turnings.

Vacuum Microwave Drying

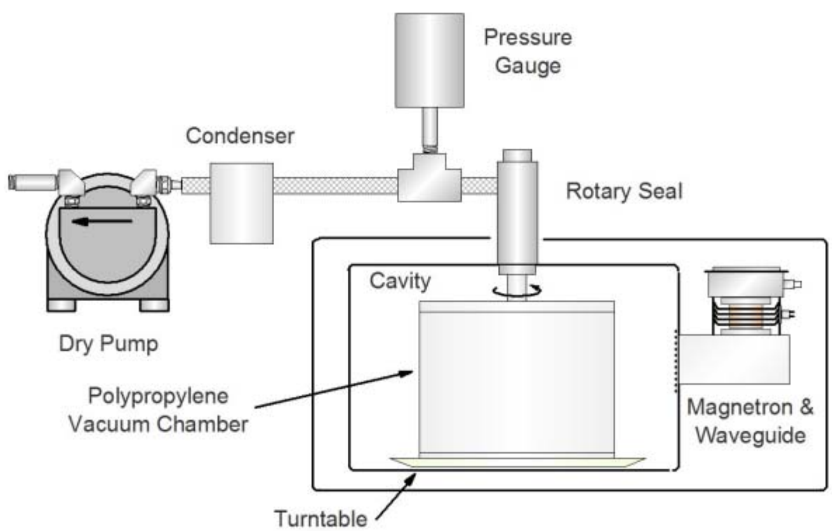
The following is adapted from my article in Vacuum Technology & Coating, February 2019.

The figure on the next page depicts the phase diagram for water. Three drying processes are noted. Common atmospheric pressure dehydration is, as the name implies, performed at atmospheric pressure. The primary variable is the drying temperature. Freeze drying relies on the sublimation of water from the solid state. This dictates a certain pressure upper limit for proper operation i.e. the process must be run below the triple point of water. Typical freeze drying processes operate in the range of 0.1 to 0.5 Torr and the drying temperature may be elevated to around 40 °C or so – warm enough the accelerate the drying but not hot enough to damage the product.

In the case of VMD, we are using microwaves and moderately low pressures to quicken the drying process. I've shown the boundary around the VMD label as being rather fuzzy as the vacuum and temperature parameters can vary substantially per the requirements of any specific application.



VMD has been applied in laboratory applications as well as in full production. An example of the latter would be the VMD drying of lumber. On a smaller scale, and related to Earl Rich's effort, would be the 2015 paper by Monteiro *et al.* [1]. They described a VMD system based on a domestic microwave oven. The motivation was to have a lab-scale apparatus that would be simple and economical. The figure below is a simplified diagram of the apparatus, based on the authors' description.



The attributes are much the same as that of Earl Rich's wood drying apparatus. The most significant change is the use of the oven's turntable to rotate the vacuum vessel containing the product. This required the addition of a rotating vacuum seal. The authors also optimized the waveguide.

For more information on VMD, check out the *VT&C* article noted above.

Reference

1. Ricardo Lemos Monteiro, Bruno Augusto Mattar Carciofi, Antonio Marsaioli Jr., João Borges Laurindo, *How to make a microwave vacuum dryer with turntable*, Journal of Food Engineering, Elsevier Ltd., 2015. Available on line at http://dx.doi.org/10.1016/j.jfoodeng.2015.06.029

Quick Drying of Woodturnings

Earl Rich, Mont Vernon, NH
From *the Bell Jar*, Spring 1998

In June of 1998 I received an email from Earl describing his work and I promptly drove over to see him and his shop. The following describes his foray into mixing vacuum, microwaves and the craft of woodturning. - Ed.

For the past 6 months, I've been attempting the craft of woodturning, specifically, making wooden bowls from green wood. After monitoring the internet newsgroup *rec.crafts.woodturning* I found that the usual recommended procedure is to rough turn the bowl, leaving 1/2 inch or so wall thickness, and then set it aside for a few months in a paper or plastic bag to slowly dry. The goal is to dry it so that it doesn't crack or distort too much. Slow drying inside the bag minimizes differential drying where the edges or base shrink more than other parts.

Some woodturners have been experimenting with microwave oven drying, but the trick is to not overdo it and there is still a problem with differential drying. One turner out on the west coast was trying to dry in a vacuum, but wasn't having too much success. Since I have access to lots of surplus vacuum equipment, I decided to give it a try.

My first chamber was a 14" diameter by 24" high by 3/8" wall Pyrex tube. I made end pieces out of ¾" Plexiglas and for good measure added a load cell to weigh the bowl during drying. I also incorporated a temperature monitor. The vacuum pump was a Welch 2-stage rotary oil pump salvaged from a helium leak detector. It worked, but the pump didn't handle the water vapor very well as the oil became contaminated rather quickly. It was obvious I didn't know much about vacuum systems.

The temperature sensor showed that the bowl temperature dropped quickly as a result of evaporative cooling. As a result, the rate of moisture evaporation also decreased. To counter the evaporative cooling effect I added an infrared lamp inside the chamber. Now the problem was that, since the surface of the bowl became dry before the inside, the dry wood was now an insulator and kept the heat from reaching the inside of the wood. There was still a problem with uneven drying and distortion.

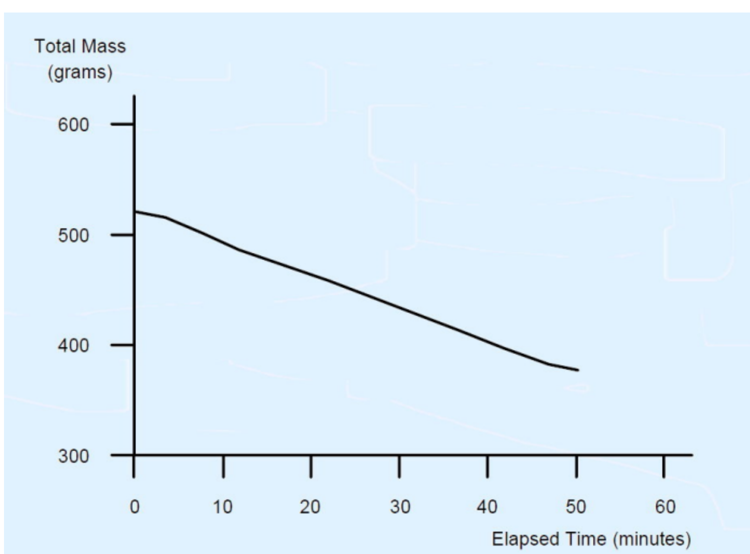
Well, if nothing else, I am not a quitter. My next experiments involved removing the bowl periodically, microwaving it to heat it up, and subjecting it to vacuum again. At this time I also added a simple cold trap to try to intercept the water vapor before it got to the pump. That helped, but was quite a hassle and wasn't very convenient.

My next thought was, "why not put a vacuum chamber inside the microwave oven?" Mentioning this to the newsgroup got a few bricks thrown my way, but I didn't let that slow me down. My first chamber made with flat Plexiglas was a partial success, but 3/8-inch plastic was not nearly stiff enough to keep joints and door gaskets vacuum tight. The next chamber was made of 5/8 and 3/4-inch Plexiglas with mitered joints. I also used stainless bolts to further secure the edges and fresh RTV sealer before assembly. The microwave oven was a town dump salvaged unit, so I had no misgivings with punching holes in it. The vacuum connection was a bolted on bulkhead fitting (brass) with a copper nipple going to a tee connector for ball valves.

The pump I am using now is from a local surplus seller. It is a double cylinder diaphragm pump made by Gast. Being an oil-free (dry) pump, it tolerates moisture better than the Welch. Even with small leaks in the system, I have no problem getting down to 25+ inHg, a level that seems completely adequate for this task since the microwave energy easily gets the wood to temperatures in the 150 °F range.

With this success, I next set about to improve the cold trap. My best one is a 6" diameter stainless bellows clamped between two plates of high density polyethylene. Below that is a Plexiglas tube 5 inches in diameter and 6 inches long that accumulates the water. The whole cold trap is immersed in a 5 gallon bucket of cold water. Since I'm trying to make something anyone can duplicate, I also made one out of an apple juice bottle. This works well too.

Now I have something that really works. It's amazing the amount of water that can come out of a bowl. Typically about 1/3 to 1/2 the weight is water. The best thing is that I can turn a green bowl (maple, birch, beech, etc.) and have it ready for finishing in less than 1 hour. Other turners usually take 3 to 6 months to get to this point. The plot below shows the water loss for a 6-inch diameter maple salad bowl with 22 inHg vacuum, 1175 watts with 50% duty cycle (approx. 17 sec. on/17 sec. off) and the cold trap at 60 °F.



I have gotten a lot of encouragement from the newsgroup since I make regular postings on my progress. I've also gotten lots of good advice, particularly on the need for a cold trap. I recently subscribed to *the Bell Jar*, and the first issue I read had very pertinent articles on microwave ovens, freeze drying, and a good explanation of the triple point of water (Volume 6, Number 1).

Since my process takes so much time out of the woodturning experience, I have been advised to "patent it as soon as possible," publish in wood working magazines, sell plans, sell equipment, etc. Since I don't believe in the patent system for individuals, that leaves the last few options.

First, I am not quitting my day job. I'm not going to get rich out of this. I'm having fun and some recognition, and that is mostly enough. I plan on writing a manual so people can duplicate this work, and since my sister is a professional videographer specializing in special interest videos, I will also go that route. This has turned out to be a really a fun project, taken on for the sheer enjoyment of applying a scientific approach to an old craft. The surprise was that it has solved a real problem. Being new to woodturning, I didn't realize that it couldn't be done because it never had been done. Some advantages to being ignorant!

Afternote: I tried to contact Earl to no avail. From what I can tell, he's still in Mont Vernon. The rec.crafts.woodturning newsgroup no longer is active but the posts are archived. - Ed.

Home-Made Plasma Experiments

Mark Atherton, New Zealand markaren1@xtra.co.nz

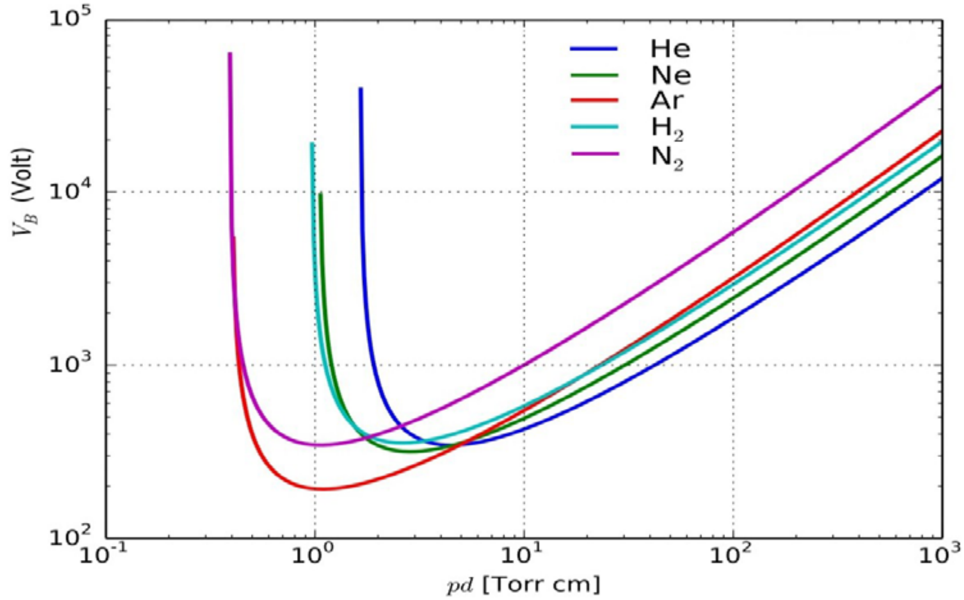
Background

Having an Electrical-Engineering background, the whole vacuum arena was initially quite a fascinating puzzle. As well as issues relating to out-gassing, the magic 'glow region' initially caused some puzzlement, as well as destruction of some early attempts to get electricity into a vacuum environment.

Glow Region

At normal atmospheric pressure, air has a breakdown voltage in the order of 3 kV per mm. Drop that pressure to something around 800 millitorr and the breakdown voltage plummets (the Paschen curve) causing all manner of mischief and issues to the unwary. Characteristics for various gases are shown in the illustration on the following page.

Having discovered this phenomenon, it seemed that some low cost experiments might be possible using air (the majority of which is nitrogen), as well as argon (available is relatively small quantities from the local welding supply company).



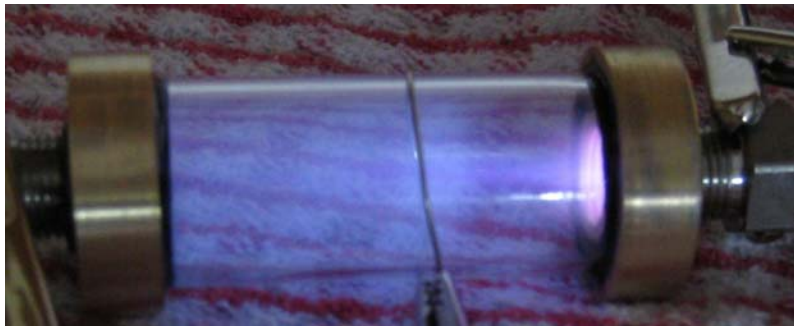
The Paschen curve

Plasma Chamber

A small plasma chamber was constructed from turned brass stock, glass tubing, a couple of stainless Swagelok couplers and the whole mess being held together using jewelers wax. The brass ends are turned from 25 mm round bar, drilled and tapped 1/8" NPT. The gas connection is via Swagelok to NPT stainless 316 adaptors. NPT threads are sealed using JB weld epoxy (which exhibits very low outgassing under vacuum).

Swagelok fitting, brass end, and plasma tube

Cutting the 20 mm OD glass tube was done using a lathe. The tube was held in the chuck with a suitable soft-jaw and rotated slowly. The cutting was done using a Dremel with a diamond tipped cutting disk. First attempt to tidy the ends was to fire-polish the ends using an LPG flame. A much more satisfactory solution was to use a flat abrasive sheet, and lots of rubbing.



Roughly 800 millitorr and 1 kV at a few milliamps

The first attempt at sealing the brass and glass was to use a high vacuum compound (Apiezon Q), but this was less than satisfactory. My final solution was to use some black jewellers wax, and a hot air gun. This sticky stuff along with well-degreased components worked very well, and has not shown any problems with outgassing (at least at the modest vacuums required by the plasma).

Swagelok Fittings

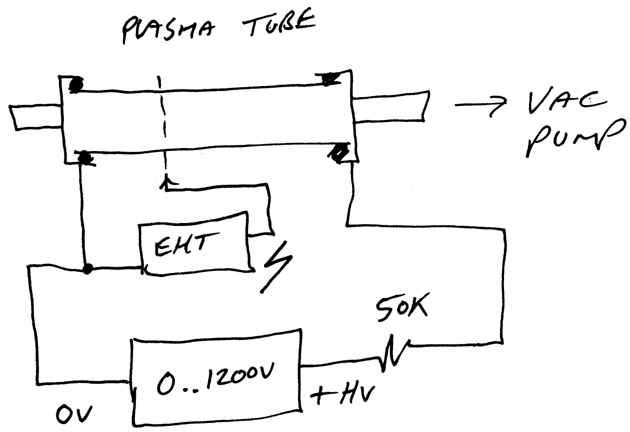
The Swagelok connectors had been removed from some old environmental gas monitoring equipment; this was the first time I had put any into service.

Swagelok tube fittings

Swagelok manufacture a whole slew of fittings aimed at industry, as well as the scientific community. Personally, the main point of interest is the ready availability of replacement ferrules, allowing the re-use of the connectors and adapters from salvaged equipment.

Setup for Continuous Plasma Generation

The simplest initial setup was to place a current limit resistor (50k, 10 W) in series with a variable 0-1200 VDC power supply, and connect either side of the plasma chamber to this excitation voltage. Plastic tubing was used to connect the plasma chamber to the vacuum pump ensuring a fully isolated test environment. The variables for the experiment were the adjustment of high voltage and vacuum.

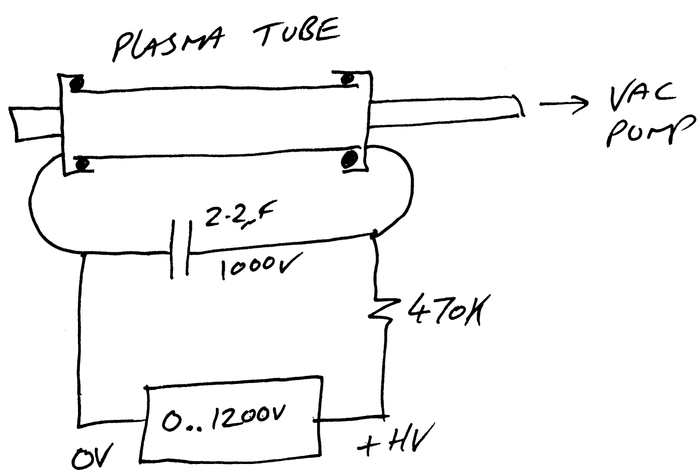


Steady-state plasma generation

To investigate the trigger characteristics of the tube, an extra-high-tension (approx. 20 kV) BBQ igniter was connected between an initiation-wire strapped around the centre of the tube and ground. By dropping the variable HV supply below the trigger point of the tube it was possible to start the plasma using a single pulse from the EHT BBQ igniter.

Setup for Pulsed Plasma Generation

For this experiment, the 50k current limit resistor was increased to 470k, and a 2.2 µf, 1 kV capacitor was connected directly across the brass ends of the plasma tube. The HV supply was set at around 1 kV, and the tube flashed every few hundred milliseconds once the vacuum was set to the correct pressure.



Flashing plasma generation

For the tube to repeatedly flash, the charging current through the resistor slowly increases the voltage across the capacitor until the strike-voltage is reached. Upon striking, the bulk of the charge stored in the capacitor is converted into plasma, dropping the capacitor below the strike potential. The cycle then repeats. I did somehow manage to resist the temptation to replace the small capacitor with a 100 µF 2.2kV component, but I could just see the resulting flying glass with my minds-eye after the first explosion.

Vacuum Pump Setup

The standard setup for the pumping station is a mechanical roughing pump followed by a Pfeiffer turbo-pump. In the initial build, the turbo-controller was also responsible for managing the roughing pump as well as the associated vent solenoid. These three components have now been separated and are controlled by individual switches.

One of my main initial concerns was if the glass tube of the plasma setup imploded, and the resulting fragments made their way into a fully running turbo-pump assembly. Several steps were taken to reduce the possibility of this kind of damage; all vented ports of the turbo-pump chamber were fitted with KF25 sieves. As noted above, the system was rewired so that the turbo-pump didn't need to be running since the required vacuum could be attained using just the roughing pump. Finally, the vacuum system and the plasma chamber are connected using ¼" industrial pneumatic tube; this is itself has a small cross section reducing the severity of damage should the glass-hit-the-fan.

Vacuum Adjustment

The single most difficult part of these experiments has been trying to set the vacuum to a specific level. This has been attempted by running the roughing pump for small amounts of time with the hope that the settled vacuum was a required. This is far from satisfactory.

The next generation setup will include a 100 cc per minute mass flow controller, allowing the controlled injection of air (nitrogen) from a vented port, or argon, from a small welding cylinder via a 5 psi regulator. This will allow the roughing pump to be run continuously, and the gas injection rates (and associated vacuum) to be adjusted easily.

Summary

Initially, this whole project was almost dismissed on ground of simplicity, but in hindsight (and as is quite usual) masses of things have been learned so far and a small number of very useful items acquired for reuse in later ventures.

Thanks go out to Ecotech Services Ltd in Christchurch, NZ for their continued support and supply of interesting components and systems for various projects.

Mark Atherton
New Zealand
27 May 2021

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Plasma Activated Water (PAW)

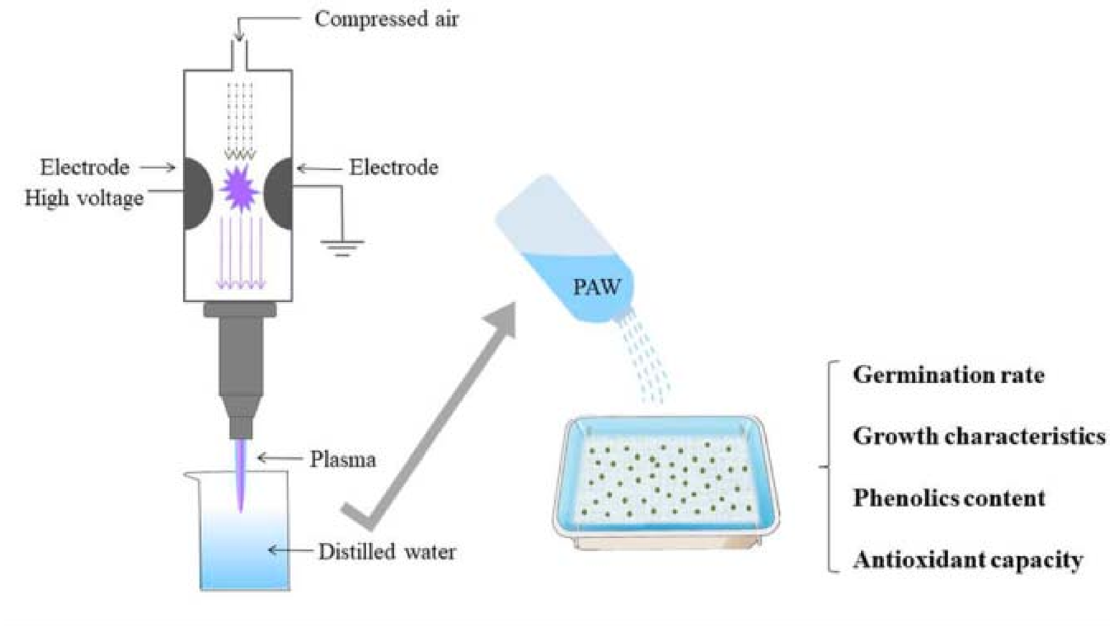
Plasma activated water represents another emerging application of atmospheric pressure cold plasmas. The principle is simple: you expose the water to almost any kind of atmospheric pressure plasma. This could be a plasma jet (APPJ), gliding arc discharge (GAD), dielectric barrier discharge (DBD), corona, etc. Some researchers have run a blown discharge under the surface of the water. The discharge then produces various active species in the water. The activated water can then be used in a variety of applications.

A 2018 paper by Thirumdas *et al*. [1] provides an excellent overview of PAW. The following is from the abstract:

> Background: Cold plasma is an emerging non-thermal disinfection and surface modification technology which is chemical free, and eco-friendly. Plasma treatment of water, termed as plasma activated water (PAW), creates an acidic environment which results in changes of the redox potential, conductivity and in the formation of reactive oxygen (ROS) and nitrogen species (RNS). As a result, PAW has different chemical composition than water and can serve as an alternative method for microbial disinfection.
>
> Scope and approach: This paper reviews the different plasma sources employed for PAW generation, its physico-chemical properties and potential areas of PAW applications. More specifically, the physical and chemical properties of PAW are outlined in relation to the acidity, conductivity, redox potential, and concentration of ROS, RNS in the treated water. All these effects are in microbial nature, so the applications of PAW for microbial disinfection are also summarized in this review. Finally, the role of PAW in improving the agricultural practices, for example, promoting seed germination and plant growth, is also presented.
>
> Key findings and conclusions: PAW appears to have a synergistic effect on the disinfection of food while it can also promote seedling growth of seeds. The increase in the nitrate and nitrite ions in the PAW could be the main reason for the increase in plant growth. Soaking seeds in PAW not only serves as an anti-bacterial but also enhances the seed germination and plant growth. PAW could potentially be used to increase crop yield and to fight against the drought stress environmental conditions.

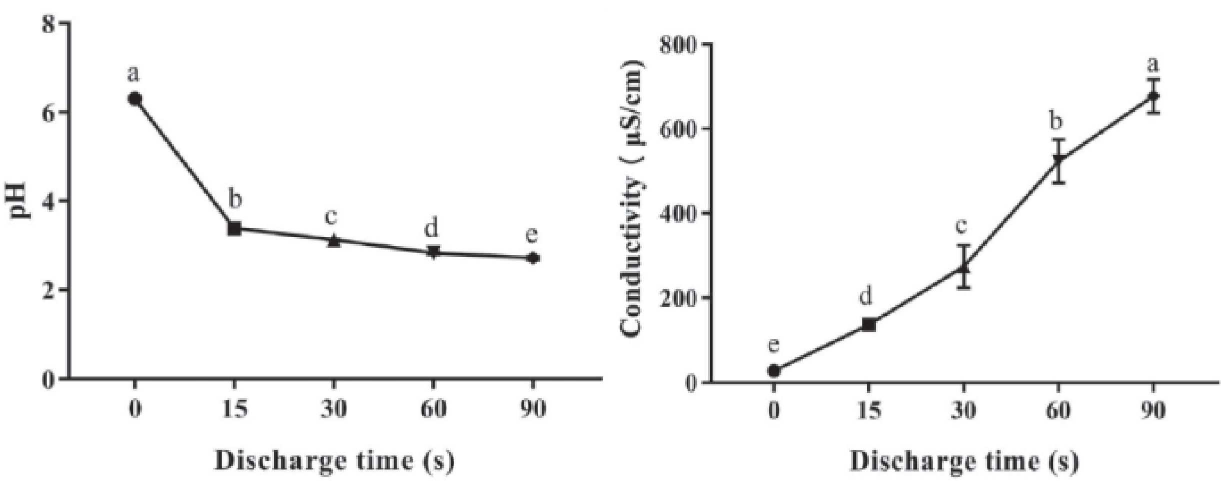
The illustration on the next page from Fan *et al*. [2] shows on approach to using PAW for germination studies. The paper describes a cold plasma jet source operating at 5 kV, 40 kHz and 750 watts. 200 ml of treated water was produced in exposures of 15, 30, 60 and 90 seconds. The high power level suggests something like a blown arc device. I made an inquiry to the corresponding author for some more details but have not yet received a reply.

The paper describes the results of the exposure over the various intervals for parameters including pH, electrical conductivity, NO_2^-, NO_3^-, H_2O_2 and ORP (oxidation-reduction potential).



PAW Experimental Set Up. From Fan *et al*. [2], *Effects of plasma-activated water treatment on seed germination and growth of mung bean sprouts*. Open source document.

The plots below show the results for pH and conductivity.

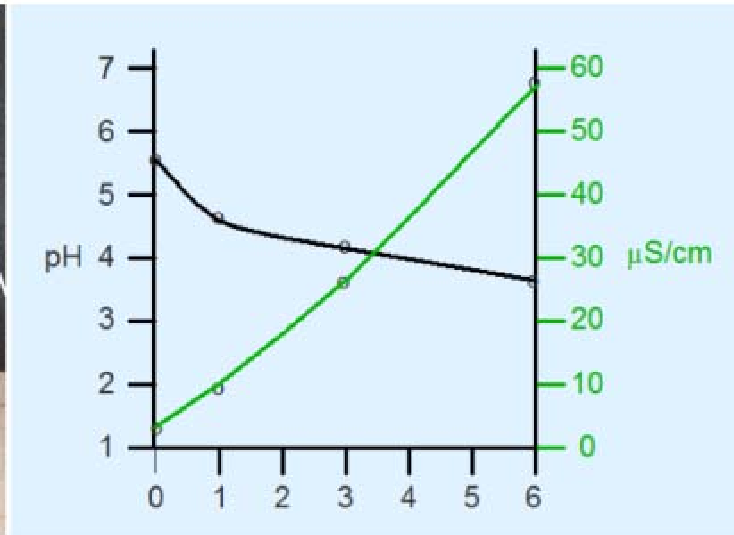


Plots for pH and electrical conductivity. From Fan *et al*. [2]. Open source document.

An Experiment

I made a small (250 ml) batch of PAW, starting with distilled water. I placed a 4" OD x 6" tall acrylic cylinder on a magnetic stirrer and then placed my inverted SMD device (described in the February 2021 issue) on top of the cylinder. The SMD was driven by my Information Unlimited PVM500 power supply. For all I know, I'm possibly the first person in Owl's Head to deliberately make PAW.

I used a handheld pH/conductivity meter to measure changes over the test period. pH should decline and conductivity should increase as the reactive species combined with the water. My set up and results are shown below.



The experiment self terminated at six minutes when the thin (0.010 inch) Teflon dielectric failed at a single point at the perimeter of the powered electrode. The failure was determined to have been caused by the weight of the inverted SMD apparatus which applied stress at the perimeter of the powered electrode when the mesh screen deformed. During the run, the apparatus did get fairly warm, input power to the power supply was 0.7 amps at 80 volts. Because of this, I'm in the process of replacing the screen with a stiffer piece of perforated stainless steel plate.

The plots are very similar in form to those in the paper. The big difference is in the power levels, my SMD at a few 10s of watts and the arc in the reference at 750 watts. For the amateur, the blown arc type device is also much simpler to implement. That's coming next.

This approach can be applied to substances other than water. One commercial example is the STERAMIST by Tomi Environmental Solutions (https://tomimist.com). The device has a reservoir that contains 7.8% hydrogen peroxide. An ultrasonic mister nebulizes the solution which is then blown past a pair of electrodes. The arc plasma creates a sterilizing mist that leaves no residue.

PAW and Waste Treatment

A friend of mine manages a municipal waste treatment facility. I asked him about PAW in waste management. He had never heard of it but he did a bit of searching on the web and had this to say:

> The holy grail of wastewater treatment in the upcoming five years or so is going to be dealing with PFAS (*see note at end*). But prior to it taking center stage there was a

laundry list of similar so-called "contaminants of emerging concern" that were already on the radar: waste pharmaceutical products, endocrine disruptors, and the like. Most current wastewater treatment processes are either physical or biological and are not capable of dealing with any of those. Plasma seems to have good efficacy against all of them. The rub will be to see if it can scale to deal with flows in the million gallons per day range economically.

He offered up the following links:

- https://news.engin.umich.edu/2020/08/treating-pfas-water-contamination-with-cold-plasma/?utm_source=newsletter&utm_medium=email&utm_campaign=September_2020
- https://www.sciencedirect.com/science/article/abs/pii/S2213343718304263
- https://flowrox.com/article/plasma-water-treatment/

Note. From the US EPA web site: "Per- and polyfluoroalkyl substances (PFAS) are a group of man-made chemicals that includes PFOA, PFOS, GenX, and many other chemicals. PFAS have been manufactured and used in a variety of industries around the globe, including in the United States since the 1940s. PFOA and PFOS have been the most extensively produced and studied of these chemicals. Both chemicals are very persistent in the environment and in the human body – meaning they don't break down and they can accumulate over time. There is evidence that exposure to PFAS can lead to adverse human health effects."

References

1. Rohit Thirumdas, Anjinelyulu Kothakota, Uday Annapure, Kaliramesh Siliveru, Renald Blundell, Ruben Gatt and Vasilis P. Valdramidis, *Plasma activated water (PAW): Chemistry, physico-chemical properties, applications in food and agriculture*, Trends in Food Science & Technology 77 (2018) 21–31 .https://www.researchgate.net/publication/324955915_Plasma_activated_water_PAW_Chemistry_physico-chemical_properties_applications_in_food_and_agriculture
2. Liumin Fan , Xiufang Liu , Yunfang Ma and Qisen Xiang, *Effects of plasma-activated water treatment on seed germination and growth of mung bean sprouts*, Journal of Taibah University for Science 2020, Vol. 14, No. 1, 823-830. https://doi.org/10.1080/16583655.2020.1778326 Open access: https://www.tandfonline.com/doi/full/10.1080/16583655.2020.1778326

Update on My Saddle Field Fast Atom/Ion Source

In the January 2021 issue I described my saddle field source that I built some number of years ago. I noted that the anode electrodes were a bit of overkill and not of the best geometry. The two rods were 3/16" in diameter with a huge interelectrode gap of about 22 mm. I just finished another set, also using a KF50 unbored stub. I used an aluminum stub for ease of machining and cost (currently $13 from LDS Vacuum). For the feedthoughs I used ⅛" ID x ¼" OD alumina ceramic tube from McMaster-Carr. For the rods I used 3 mm stainless rod, also from McMaster-Carr. The 3 mm rods slide easily into the ceramic tubing with very little play.

I bored two 17/64” holes in the stub (¼” was too tight) on 3/8” centers. The ceramic tubes were cut to about 2- ¾” using a diamond tile cutting wheel. The rods were cut to about 6-¼”. The rods and insulators were then assembled to the stub using JB Weld epoxy, keeping the seals on the atmosphere side. The resulting electrode structure has a gap of about 6 mm, right where I wanted it. The photo below shows the assembly.

In the coming weeks I’ll test it out under medium and high vacuum. I have a simple design for an ion extractor that will also be tried out.

Some interesting YouTube channels
Mark Atherton New Zealand

I have recently found myself watching a couple of YouTube channels with interesting vacuum and physics related content so thought it might be worth passing on details.

‘Huygensoptics’ (https://www.youtube.com/user/huygensoptics) is run by a Dutch chap called Jeroen Vleggaar, and is an extension of his optical consultancy. His videos cover a multitude of topics from a ‘Maskless Wafer Stepper’ project to ‘Fourier Optics used for Optical Pattern Recognition’. A recent vacuum-related video was ‘Vapor Deposition using Thermal Evaporation’ which also included a section on an amazing turbo-pump crash where the pump appeared to survive.

On an unrelated subject, ‘Dalibor Farný’ (https://www.daliborfarny.com/) has been building a business in the Czech Republic dedicated to manufacture and sales of modern day Nixie tubes. The man has an extraordinary breadth of skills, energy and organization which he directs at his business. His video production style is very open; he talks about ongoing trials an tribulations about a broad range subjects, along with new projects and even includes details of suppliers of some of the more interesting materials. A large amount of his small factory is custom built (including a high-output oxygen generator) which he covers in detail.

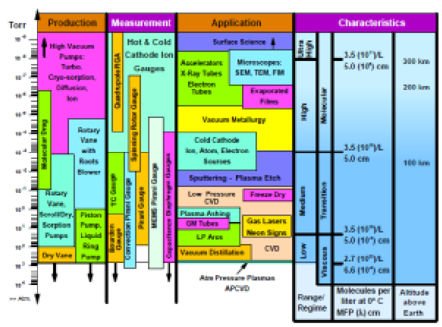
Articles of Possible Interest in *Vacuum Technology & Coating* Magazine

July 2012
Valve Conductance - What's behind the specifications
The article looks at the various conductance characteristics and specifications for isolation and control valves. This includes an explanation of the term C_v, the valve flow coefficient.

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.

Directly Linked from My Site

Creating, Measuring and Using Vacuum: How They All Fit Together



In June and July of 2015 I wrote a two-part series of articles in *Vacuum Coating & Technology* that were based on the still current version of the chart that I developed for *the Bell Jar* in its early days. The chart shows the pressure range from atmosphere to 10^{-9} Torr. The columns show the types of pump and gauge that are applicable along with a number of representative applications. Also shown are the designated ranges, flow regimes, gas density, mean free path and height above earth.

A link to the pdf of the chart is now listed on my *Articles and Publications* page, just below the index of "new" *Bell Jar* issues. There are also direct links to the articles at the *Vacuum Technology & Coating* site.

End Notes

Summer is having a hard time arriving in New England. On the plus side, this keeps me from wishing to be outdoors instead of in the house playing with vacuum equipment and radios.

As usual, contributions of any complexity are welcome.