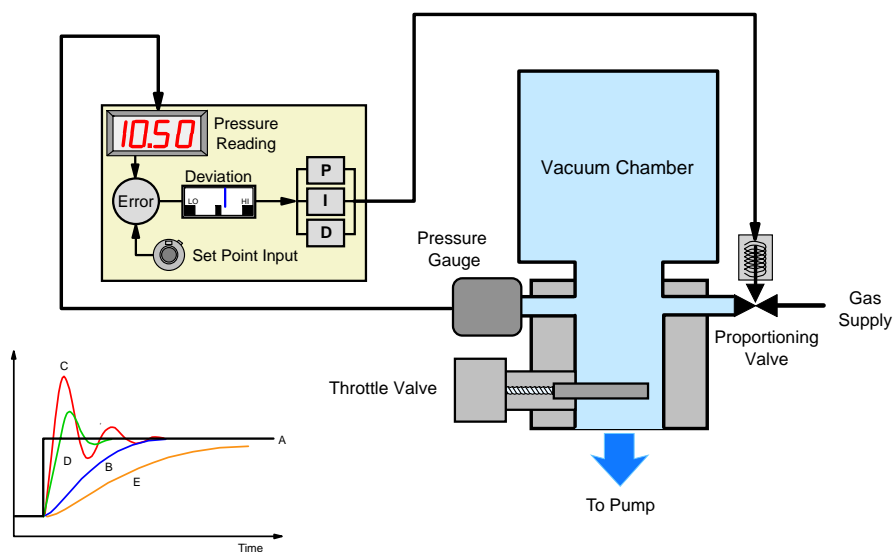


the Bell Jar

vacuum technique and related topics for the amateur investigator

VOLUME 10 NUMBER 3/4

SUMMER/FALL 2001



From Cartesian Divers to Closed Loop Pressure Control

*Also In This Issue: Synthesizing Buckyballs, Modified
Hardware Store Inverters for High Voltage Power
Supplies, a Microwave Oven Plasma Reactor, Taking
the Bubbles out of Gel Candles*

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The mission of *the Bell Jar* is to provide information of value to the serious amateur experimenter in the area of vacuum technique. Along with this, the scope of the journal includes projects in the physical sciences which require the use of vacuum apparatus of varying complexity. *the Bell Jar* also serves as a forum for information exchange. Contributions in the form of correspondence and articles are invited.

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What Next? This issue (which is actually being released at the end of 2007) represents the end of the original series of this publication. Beginning in 2008 with Volume 11, *the Bell Jar* will become an on line journal with printed copies only being made available for the complete year.

Coming Up: Preparing pumps for oxygen service, Kurt J. Lesker's amazing MicroMaze, using orifices for precise low pressure control, plasma ashing and lots more.

How to Make More Buckyballs

Greg Konesky

Abstract: Improvements in reactor design and operation are described which result in an over five-fold increase in yield of the soccer ball shaped carbon molecule C_{60} also known as Buckminsterfullerene or Buckyballs.

I. INTRODUCTION

A previous article, *How to Make Buckyballs*, appeared in *BJ* Vol. 2, No. 2, Spring 1993, pgs 7-10. This article is included in the compendium *The First Five Years*. The article described a basic system for synthesizing these hollow, cage-like carbon molecules. While the apparatus was simple, yields were poor, typically 1% or less, and highly variable from run to run. In the years since that article was published considerable improvement has occurred in understanding how Buckminsterfullerenes (simply referred to as fullerenes) form, preferred reactor operating conditions and enhanced extraction techniques.

Among the more interesting properties of fullerenes is that they are held together in a solid by weak van der Waals forces. As a result, they can be sublimated in a vacuum with relatively moderate temperatures of 350 degrees C and higher. Fullerene vapor has been used as a source of pure volatile carbon in synthesizing nanocrystalline diamond thin films using a microwave discharge. In this process, the rare carbon dimer C_2 is formed - a process unique to the decomposition of fullerenes. Thin films of fullerene can also be deposited by Physical Vapor Deposition (PVD). These films have interesting optical and electro-optical properties. Both the PVD of fullerene thin films and the electrochromic effect in these films will be the subject of future articles.

The electric arc synthesis technique is the simplest route to generating fullerenes. Two graphite rods are brought together in an inert atmosphere, typically helium, at reduced pressure. An electric current of 100 to 150 amps is passed through the contact area of the two graphite rods (each of which is $\frac{1}{4}$ inch in diameter) establishing an electric arc. Typically, 15 to 20 volts are required, either ac or dc, to maintain the arc. Fine soot is produced, most of which is unreacted graphite. Extraction of the soot with a hydrocarbon solvent, such as toluene or xylene, dissolves the fullerenes. The solution consists primarily of dissolved C_{60} and C_{70} in a ratio of 3 to 1, as well as trace amounts of smaller fullerenes down to C_{28} . The insoluble portion of the soot remaining, in addition to unreacted graphite, contains higher

fullerenes, such as C_{120} and C_{24} , carbon nanotubes, plus other interesting carbon morphologies.

II. PROBLEMS WITH EARLIER DESIGNS

In the first design described in the previous article, a compressed grommet was used to form a rotary feedthrough, permitting one graphite rod to be rotated into contact with the other. The vacuum integrity of this approach left much to be desired, as evidenced by the pressure in the bell jar being higher at the end of a run than at the beginning. Fullerenes are easily destroyed by exposure to atmospheric oxygen at the temperatures inside the bell jar during arc synthesis. Yields were typically less than 1%, as calculated by the mass of extracted solids from the soot when the solvent was evaporated, divided by the mass of graphite rod consumed by the arc. Another indication of atmospheric oxygen contamination was that the total mass of soot and other residual solids was less than what was lost by the graphite rods. In other words, some carbon had been lost as carbon dioxide.

A refrigeration compressor, used as a vacuum pump, initially pumped the bell jar down. The base pressure was only about 8 to 10 Torr, so three or more pumpdown-backfill sequences were used to minimize residual atmospheric contamination. Welding grade helium was used. Its stated purity was 99.9%, but subsequent analysis by a Residual Gas Analyzer showed it to be closer to 99.99%. The last backfill was brought up to between 100 and 300 Torr as the operating pressure for the arc synthesis. Initially, the actual pressure selected did not seem to impact yield, although it became clear later that there were other yield-limiting factors at work.

During the arc synthesis procedure, a kilowatt or more of electrical energy is dissipated in the bell jar. This heats the helium and raises its pressure. A maximum of 300 Torr initial pressure was used so that at the end of a run, which may only last for 30 seconds, the pressure did not rise to the point of unsealing the bell jar from the baseplate.

A significant improvement resulted from the use of a linear motion feedthrough intended for vacuum systems. Instead of the two carbon rods being rotated

into each other horizontally, they were now pushed linearly into each other vertically. This allowed much more graphite rod length to be consumed in a typical run (1/2 inch total graphite rod consumption for the rotary approach, versus over 3 inches for the linear approach). Air leakage during the synthesis was now minimal, as the starting and final pressures were the same, and the mass lost by the consumption of the graphite rods balanced with the total mass of the end products.

However, new problems arose with the longer duration of a run. Heating and pressure rise required the process to be broken into short arc synthesis runs followed by a cool-down of several minutes, after which the arc synthesis would resume. While the yields rose to about 3.5 %, the frequent rapid thermal cycling resulted in the failure of a bell jar, as shown in Figure 1.

Another problem resulted from both graphite rods being supported by cantilevered copper clamps. The linear feedthrough also permitted rotary motion, so both clamps had to be aligned along a common axis before they could be pushed into each other. This was easy at the start of a run, when the bell jar was clean and visual alignment was possible. But as the interior of the bell jar became coated with soot, it would be easy to misalign the two rods so that they missed each other and contacted the other rods' copper clamp. This resulted in a lot of melted copper.

The use of a linear motion feedthrough significantly improved yields. This also generated much more soot so that the total production mass of fullerene per run increased dramatically. However, the increased maintenance in frequently making new copper clamps, and loss of a bell jar, made it a mixed blessing. Reducing the duration of the arc synthesis segment and allowing more cooling time between segments together reduced thermal stress cycles and the magnitude of the pressure



Figure 1 - Fractured Bell Jar

swings. This also improved yield, and started to suggest an optimum pressure range of around 150 Torr

III. MORE IMPROVEMENTS

Elimination of the cantilevered copper clamp on the linear feedthrough became an obvious improvement. This graphite rod was now mounted coaxial with the linear feedthrough rod, so only linear motion was needed. Sometimes, if the two graphite rods were pushed into each other too forcefully, they would spot weld. A slight rotation of the linear feedthrough would easily break this weld, and the procedure could continue. The mounting of the lower graphite rod is shown in the center of Figure 2. To the left is an electrically insulated copper feedthrough (1/4 inch diameter) used to mount the upper graphite electrode.

A collar is placed on the linear feedthrough shaft outside the bell jar. It is locked in place to act as a stop so that when most of the graphite rods are consumed by the arc, you cannot accidentally go too far and touch the copper clamps together, again, producing a lot of melted copper.

A cooling coil was then added to remove excess heat and stabilize the pressure swings. This consisted of a section of copper tubing with 3" id and 4" long, outside of which was wound with four turns of 1/4 inch copper tubing, soldered in place. Swagelock bulkhead connectors were used to connect the cooling coil to the baseplate.

Normally, tap water was used as a coolant, flowing at approximately 1/2 gallon per minute. Maximum pressure variations were now of the order of only a few Torr. In one experiment, liquid nitrogen was used as a coolant. Curiously, it had no impact on yield. Perhaps the temperature differential from arc plasma temperatures (a few thousand Kelvins) to a water cooled surface (at about 285 K) versus to a LN₂ cooled surface (77 K) is not too different. Figure 3 shows the upper graphite electrode and its holder in place, as well as the cooling coil.

A refrigeration service vacuum pump, with a base pressure of 25 milliTorr, did not significantly increase yields when used for the pumpdown-backfill preparation sequence, but using 5N9 helium (99.999%) did show a slight improvement in yield. A prepped system ready to initiate the arc is shown in Figure 4, with the safety shield removed.

At the end of a run, the system was allowed to cool to room temperature, and then vented to atmospheric pressure. When removing the bell jar, a vent hose was used to collect any soot that may have been dislodged. Soot can be seen covering just about everything in Figure 5. The silvery material around the ends of the rods is sometimes called "popcorn" and is rich in carbon

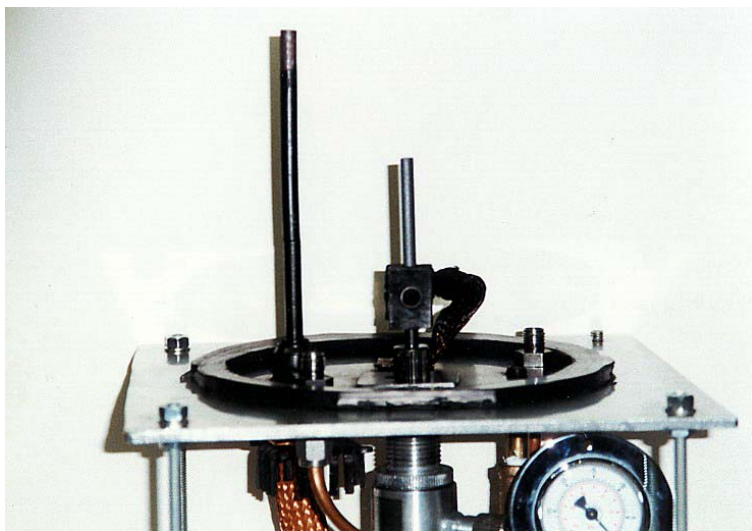


Figure 2 - Linear motion feedthrough (center) for lower electrode. Copper feedthrough for the upper electrode at left.

nanotubes. Additional fragments of popcorn are usually scattered around the baseplate at the end of a run.

IV. COLLECTING SOOT

The soot is gently swept into a large beaker, while trying to minimize the generation of any airborne particles. The popcorn is placed in a separate container as it contains little or no extractable fullerenes. Disassembling the system makes it easier to collect the soot as well as facilitate loading new graphite rods for the next run. Be sure to blow-through any cooling water that may remain in the cooling coils before loosening the Swagelok connectors. Otherwise, it will flood the baseplate when removing the cooling coil.

I tried various collection innovations, such as a tube filled with packed glass wool, connected to an aspirator.

Soot loosened with a small acid brush would be swept into this tube, which acted like a vacuum cleaner. The glass wool would then be extracted directly with solvents to remove the fullerenes from the soot trapped in it. Ultimately, it proved easier just to sweep the soot into a beaker. It's a good idea to both wear a dust mask, and provide local ventilation to remove any airborne soot. The fullerenes should be extracted from the soot as soon as possible.

V. ENHANCED EXTRACTION TECHNIQUES

Previously, the soot was simply mixed with solvent and then filtered. This process was repeated a number of times until the solvent no longer showed the characteristic fullerene red-wine coloring. This was tedious at best.



Figure 3 - Upper graphite rod and cooling coil in place.



Figure 4 - Assembled reactor without the safety shield.

A vastly improved approach is to use a Soxhlet Extractor. It operates in a fashion similar to the old percolator coffee makers, with freshly distilled solvent passing through the soot. The basic setup has a boiling flask, containing solvent, fitted with the extractor tube. A porous “thimble” is filled with soot and then placed into the extractor tube. A vertical condenser is then fitted on top of the extractor. In operation, boiling solvent vapors from the boiling flask rise up through the extractor where they are condensed by the vertical condenser. This freshly distilled solvent then falls back into the soot-loaded thimble in the extractor tube, dissolving any fullerenes. This solution eventually siphons into the boiling flask, where the process repeats.

Over time, the solution in the boiling flask will darken as it accumulates more and more dissolved fullerenes. Many researchers in the field let the extraction proceed for 24 hours, but I’ve found that 99 percent of the extractable fullerenes have been removed after about 4 hours. The volume of this solution is reduced by distillation, and then evaporated to recover the fullerenes.

Some precautions should be observed. Never use an open flame to heat the boiling flask; an electrical immersion heater in an oil bath is ideal. Use plenty of ventilation as the solvents are flammable and toxic. Never use benzene as a solvent. Some early research papers specify benzene, but we now know that it is very toxic. Toluene and xylene are alternatives that are much less

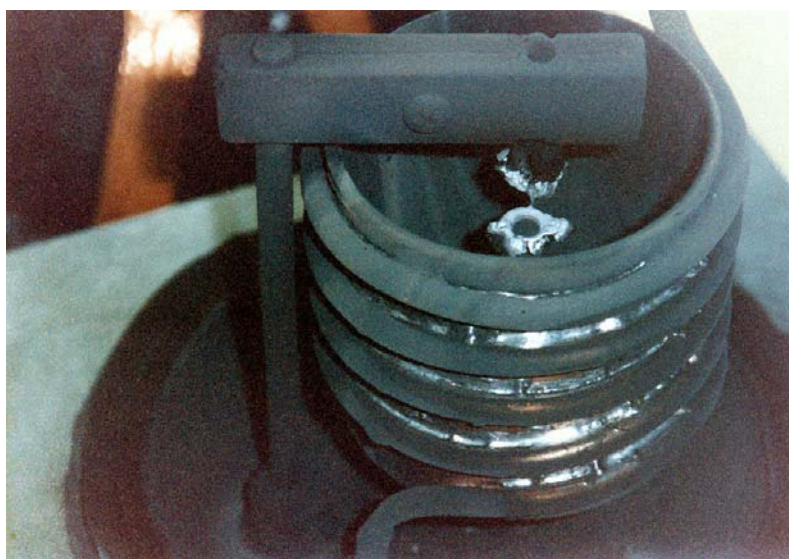


Figure 5 - Reactor interior after a run showing soot deposits.



Figure 6 - Various cooling coils. At the left is the original 3" id coil.

toxic. Distill away as much of the collected solution at the end of an extraction as possible. This solvent can then be used again. You can find inexpensive Soxhlet Extractors and associated glassware on eBay, but get new and unused ones. The siphon tube on the extractor is difficult to impossible to clean effectively.

VI. HOW TO MAKE FEWER BUCKYBALLS

The basic formation mechanism for fullerenes involves vaporization of carbon by the arc, followed by rapid quenching. It is important to then remove the newly formed fullerenes from the arc environment, otherwise they could just as easily be destroyed by it. The key is the formation of pentagons within a group of carbon

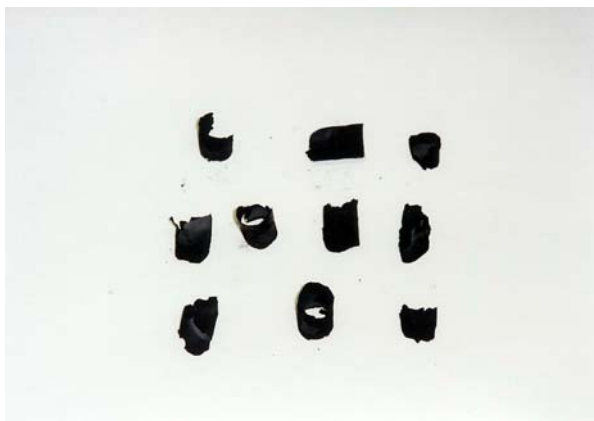


Figure 7 - Recondensed graphite from the interior of smaller cooling coils.

hexagons. This provides the curvature that eventually closes the Buckyball, and without which, planar graphite hexagons would simply form.

Given the success of the cooling coil, which consistently boosted yields to about 5.7 percent, a series of experiments was run with successively smaller and smaller cooling coils. The idea was to enhance the quenching process, and perhaps boost yields. These cooling coils are shown in Figure 6.

It is said that, experimentally, for every thing you find that works, you find ten that don't. This is the case with the smaller diameter cooling coils, where the yield drops off with smaller coils, and is effectively zero with the last two (3/4" and 1/2" id respectively).

Apparently, the quenching is so rapid that pentagons cannot form, and the interior of these cooling coils is coated with a graphite cylinder, as seen in Figure 7.

There are also curious white crystallites that form within these graphite cylinders. Some research suggests that these are yet another allotrope of carbon, sometimes called "carbolites" that are transparent, yet not diamond, that form under extremely high temperature gradients. Their physical and electrical properties are still largely unknown.

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A Happenstance High Voltage Power Supply (and having fun with DC/AC Inverters)

Steve Hansen

I. INTRODUCTION

A few years ago (Winter of 2000 I believe) I picked up a number of high voltage multiplier assemblies and some related parts from a local surplus dealer (Electronic Surplus Services of Manchester, NH). ESS was in the process of moving out of their delightful but dark and dingy 19th century factory building for newer (smaller, pricier) digs and they were in a mood to let things go for cheap.

These power supplies came in two major pieces: the stack of multiplier disks coupled with the hv step up transformer (20 kV, center tapped) and the rack mount 6 kW 220 volt/3-phase inverter. The inverter puts out about 800 volts, at 20 kHz. This is stepped up by a ferrite core transformer in the cascade base to provide the input to the Cockcroft-Walton full wave multiplier.

Each stage of the multiplier is in the form of a potted disk and the disks are, in turn, connected to each other with a clever set of studs and spacers. All of the disks are the same and they can be stacked up to 10 high (200 kV). The cascades that I picked up ranged from 4 to 6 disks and the rated output current was in the range of 100 mA. Quite lethal. A variety of names were on the equipment including Hivolt and Wallis.. A bit of investigating revealed that the company was UK-based had been through a number of acquisitions, hence the name changes.

I was able to get quite a bit of information on the units via the manufacturer's US sales office. The current name is HiTek Power and they can be found on the web at <http://hitekpower.net>. A major application for these particular supplies is in ion implanters and the units that

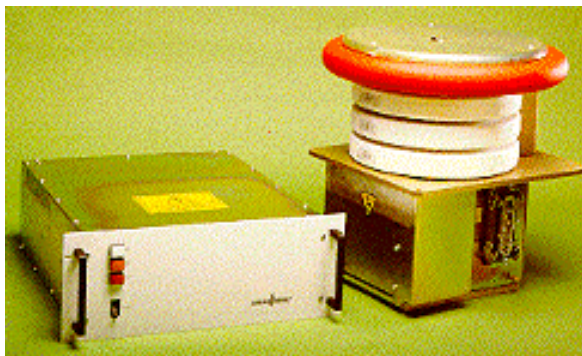


Figure 1 - HiTek Power OL Series Power Supply (photo courtesy of Hitek Power)



Figure 2 - The author's conversion with six-section 120 kV negative cascade, dual 10 kV rms transformers, water resistor and 1 Gohm divider resistor.

I got were from Eaton's semiconductor equipment division (now Axcelis). A complete 60 kV system is shown in Figure 1.

Having no access to 3-phase power and being somewhat fearful of 100 mA I really couldn't use or justify the inverter portion of the unit. Just to see if the cascades were functional, I tested them with an iron-core neon sign transformer (15 kV peak, center tapped). Each one that I tested was good.

From each cascade assembly I was able to harvest the following useful components: the ferrite core transformer (more about this later), the disks (all positive output with the exception of one six-disk negative stack), 1 G-ohm resistor for the output measurement and control circuit, plus a bunch of specialized hardware including sparkgaps, IGBTs, etc.

II. FIRST VERSION, 60 Hz

Since neon transformers are the leakage type and the output droops at higher power levels, I dug through my

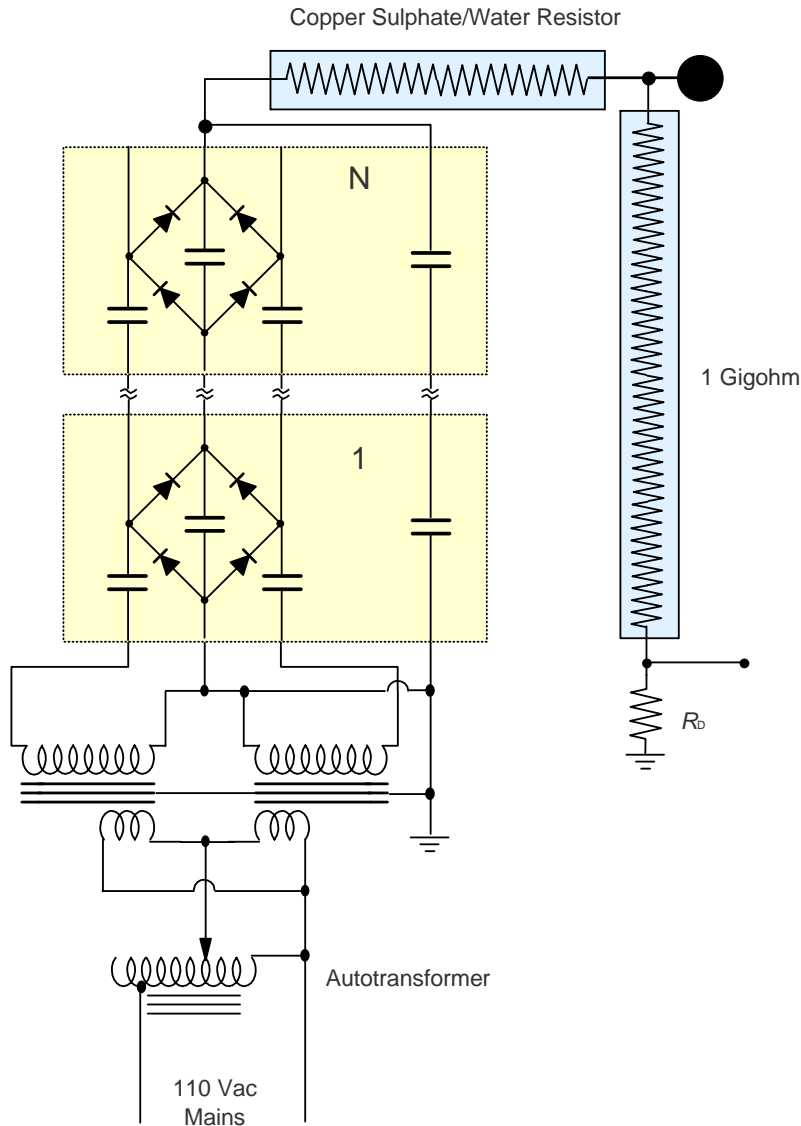


Figure 3 - Adapted Cockcroft-Walton High Voltage Power Supply. The disks are rated for 20 kV peak input. The AMP transformers are 10 kV rms each so care has to be taken to avoid overstressing the multiplier components. The water resistor is about 1 Mohm. Divider resistor R_D is sized for the input resistance of the meter used and desired voltage scaling.

junk box to find something more suitable for driving the cascades directly from the 60 Hz mains. In a dark corner of my basement I found two matched AMP Corp. 10 kV rms units. I have no idea what they were originally made for or the current specs but they have potted windings and C cores of fairly fine laminate.

The obvious disadvantage of this arrangement is that the efficiency of a low frequency CW multiplier is fairly low and any substantial current draw will cause the output voltage to drop substantially. This, of course, is the reason that multipliers are generally driven by high frequency power supplies. More stages and smaller capacitors worsen the performance.

All of that being said, a picture of the resulting power supply is shown in Figure 2. The unit was built on an old printer stand. The cascade shown is a 6-disk negative output stack (120 kV at no load). Under the shelf are the two transformers. To the left of the stack are (rear) a copper sulphate water resistor and (front) a voltage divider made from the salvaged 1 G-ohm resistor. Test leads from the divider go to a DVM which is mounted remote to the stand. To vary the output I have a 10 amp autotransformer (not shown). The schematic for the supply is shown in Figure 3.

III. UPPING THE INPUT FREQUENCY

As noted above, the efficiency of a voltage multiplier is related to the values of the capacitors, the input frequency and the number of stages. For sine-wave excitation:

$$V_o = 2nv - i \frac{8n^3 + 6n^2 - 2n}{12fC}$$

where V_o is the output voltage, n is the number of stages, i is the load current, f is the frequency and C is the value of each capacitor (equal capacitances are assumed).

Since the output is strongly influenced by the number of stages, the simplest stopgap measure is to only use the number of stages required to get the desired output voltage. Thus, for 40 kV, only two disks are used. However, for any load handling capability at high voltages, the only option is to increase the drive frequency.

I did a bit of searching and found out that some experimenters were successful in adapting common 12 Vdc to 120 Vac power inverters to run surplus 400 Hz equipment. Home Depot carries the Vector brand under their Husky Tool line so I picked up the cheapest one I could find (175 watts for about \$20) to tinker with.

The block diagram for the inverter is shown in Figure 4. The 12 Vdc input is first converted to a high frequency square wave which I measured to be about 50 kHz. A pair of transformers steps this up to about 180 V. The next stage is a full wave rectifier. The output stage is another inverter which converts the rectified dc to an approximate sine wave of 120 volts and 60 Hz.

The output stage is run by a Fairchild KA7500B SMPS controller IC. The frequency is determined by the capacitor (C_T) on pin 5 and the resistor (R_T) on pin 6. Each of these components is tied to ground, pin 7. This

Vector inverter has a fixed capacitor (0.047 μ F) and a resistor (51k) in series with a trimmer pot (30k).

I made some changes in the values of these components and determined that the output frequency of the inverter could be adjusted up to about 2 kHz before the output dropped off significantly. The test set up consisted of a simple lightbulb load, DVM to measure voltage and a PC oscilloscope to monitor frequency.

The best news was that the AMP transformers seemed to take multi-hundred Hz line frequencies in stride.

Next I acquired a 750 watt inverter and a surplus 5A 400 Hz autotransformer. The latter came from Fair Radio Sales in Lima, OH. The new inverter had the same SMPT controller IC and general architecture as the lower power unit. Figure 5 shows the circuit board and components of this inverter.

I removed the timing capacitor, the fixed timing resistor and its associated trim pot. New values were 0.047 μ F and 470 ohm respectively for the fixed components. I ran a two conductor wire out of the case to an externally mounted 100k pot. This arrangement permits operation of the inverter over a range of 150 Hz to over 2 kHz.

Compared with 60 Hz operation, the results with 400-600 Hz operation are spectacular. The only downside is that I run the thing off of my trolling motor battery so I have to keep it charged. However, it gives the battery something to do when it's not in the boat. A picture of the inverter mounted on the power supply stand is shown in Figure 6.

IV. MORE FUN WITH INVERTERS

Moving away from the voltage multiplier setup I proceeded to investigate the possibilities of using these inverters to drive a high voltage ferrite core transformer at the 50 kHz input stage frequency. With an output of a

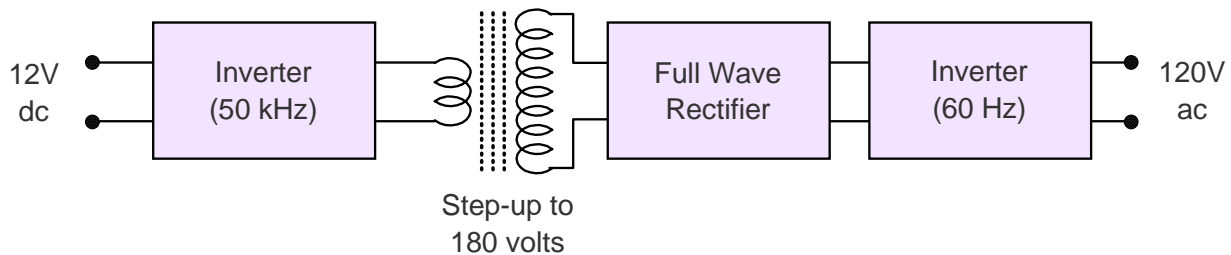


Figure 4 - Block Diagram of the Vector (Husky Tool) Inverter.

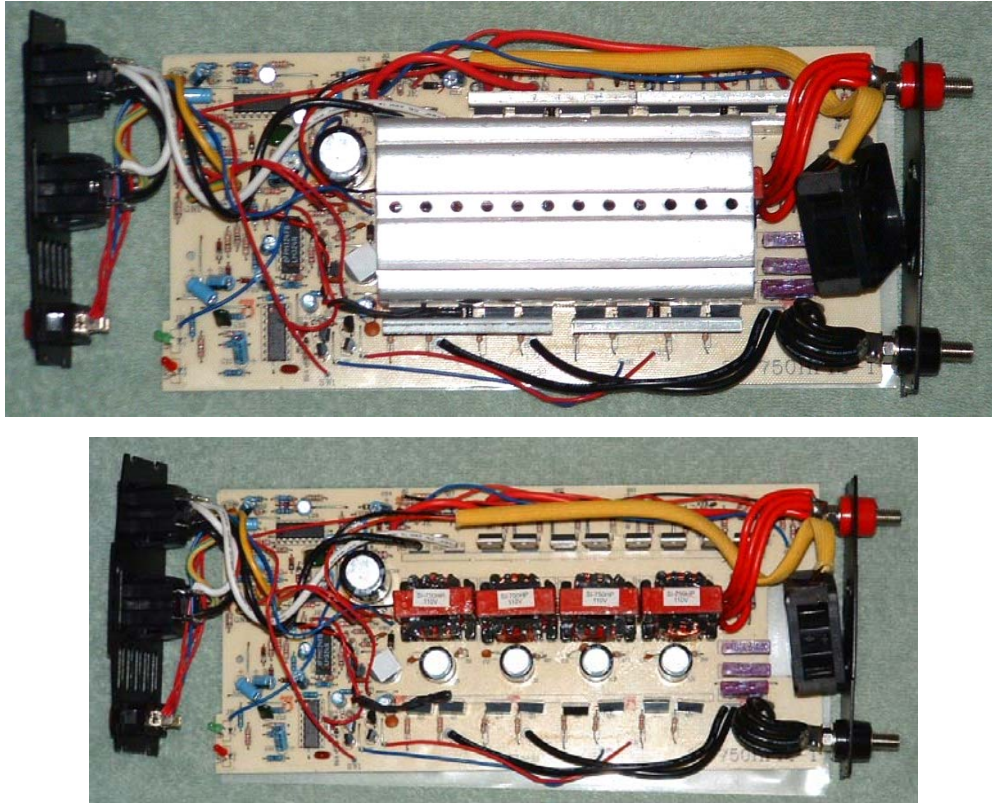


Figure 5 - 750 Watt Vector (Husky Tool) Inverter. The top photo shows the inverter with the case removed. The SMPS controller chip is at the upper left. The bottom photo shows the same inverter with the heatsink removed. The low voltage components are at the bottom of the board. Ferrite core step-up transformers are across the center and the line voltage components are across the top of the board.



Figure 6 - Power Supply with Vector 750 Watt Inverter. The 400 Hz autotransformer is in the box on the floor. In operation this would be remote from the stand. The frequency adjustment pot is above and to the left of the inverter.

few kV at several hundred watts I figured this could make a useful supply for my small plasma reactor.

For the subsequent step-up I took one of the large ferrite core transformers from a Hivolt cascade. These look like overgrown TV flybacks. The secondary is a double winding of 780 turns each with the center point grounded. The primary is 22 turns of very heavy Litz wire that is wound inside the secondary. The core section is 1- $\frac{1}{4}$ inch square and the overall core is about 7- $\frac{1}{2}$ inches on a side.

I got another inverter (this one was 400 watts) and connected the output from the step-up transformers to the outlet socket, thereby bypassing the rectifier and output stages. With this simple change I had a simple and cheap 180 volt, 50 kHz power supply. There is one downside in that in making this modification I also removed the overtemp circuit.

I pulled out the heavy primary winding and made a new winding consisting of 75 turns of #14 stranded hookup wire. To be safe I put a 4 amp fuse in series with the primary. A block diagram is shown in Figure 7 and a photograph of the completed transformer is shown in Figure 8.

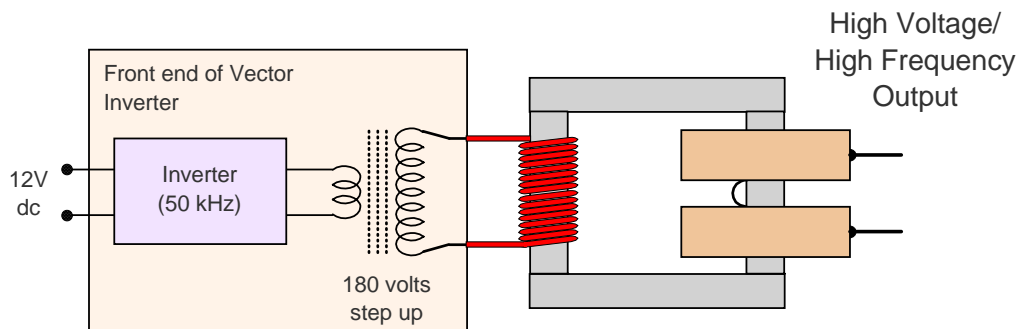


Figure 7 - Block Diagram of High Frequency/High Voltage Power Supply. In the author's version the primary was wound concentric with the secondary winding. Placing the winding as shown is entirely satisfactory.

The Hivolt/HiTek Power transformers are definitely overkill for any use I have - anyone wanting to replicate this could use a less massive ferrite core.

Driven by the 400 watt inverter, the output of this transformer provides a very hot arc. The open circuit voltage should be in the vicinity of 3500 volts but I did not attempt to measure it. In continuous operation the power supply did not seem to be taxed and the fuse stayed intact. For more power & voltage I believe I could safely remove a few primary windings.

To try out this supply, I connected it directly to the internal electrodes of my small oxygen plasma etcher. I have had some success using electronic neon sign transformers (basically the same thing as this, just much lower current output). The result was a very intense plasma. I also tried capacitively coupling the supply to the plasma and that also worked reasonably well.

V. NEXT STEPS

With regard to the Cockcroft-Walton multiplier the next logical step would be to replace the AMP transformers with ferrite core transformers wound in the correct ratio

to properly drive the cascade using the 180 volt high frequency output of an inverter. This would provide quite stunning performance.

With regard to the plasma reactor high frequency supply. I plan to investigate the use of a larger inverter (maybe 1000 watts) and also try some experiments with increasing the frequency beyond 50 kHz.

VI. ROLLING YOUR OWN

While I have to consider myself fortunate to have acquired (at an excellent price) the surplus Hivolt multipliers and related components, much of what is presented in this article can be accomplished with discrete components, readily available ferrite core stock, etc. The great find for me was discovering the ease of modification and utility of inexpensive power inverters.

For those who might be on the lookout for the cascade multipliers, ESS does maintain an eBay store (esssurplus) and the Hivolt products do show up from time to time.

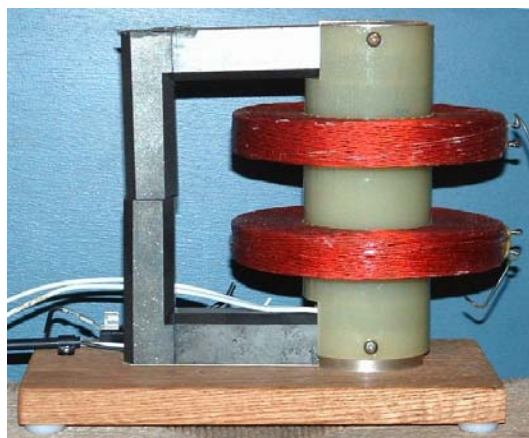


Figure 8 - Modified Hivolt Transformer on Wood Base. The primary is wound concentric with the secondary. Fuse holder is behind the core but is not connected in this photo. For a size reference, the oak base is 10 inches long.

Microwave Oven Plasma Reactor

Hideaki Page

I. MOTIVATIONS

I'm interested in doing some sputtering of various semiconductor materials. Constructing a DC sputtering system seems relatively straight forward. However RF systems are needed for semiconductors. Generating an RF plasma using a microwave oven seemed the simplest approach to accomplish this. Also diamond films can be grown under modest vacuum conditions using a methane/hydrogen plasma. These are long term goals. At present I am having fun generating the plasma.

II. FIRST EXPERIMENTS

To test the feasibility of the generation of plasmas using a microwave oven I constructed a quick and dirty chamber. I removed the turntable in the oven and fitted the vacuum outlet through the hole for the motor shaft. The fitting was a 10mm diameter threaded pipe fitting pirated from somewhere. I used common PVC piping to the rotary vacuum pump. All pipe fittings were fixed with jubilee clips and PTFE tape. I fabricated a simple gasket from a sheet of ~1mm thick PVC sheet. I had to press down on the jam jar and open the valve to the pump to get the system to seal. However, once sealed I couldn't move the jar. After this I simply closed the door of the oven and turned it on full power at first (600W in my case). There is a sudden eruption of colour as the plasma strikes. I believe the colour of the

discharge is typical for air. The first few tries didn't work. I think this was due to leaks on the pipe fittings. Once these were thoroughly reworked with the jubilee clips and PTFE tape I suddenly got results. I was very surprised that the crude seal work as well as it did.

The system worked until the PVC seal failed (burned) after a few runs of 10-20s each. The jam jar got very hot but didn't crack. Sometimes the oven's safety trip kicked in and I had to wait until everything cooled down. However these initial results were very promising.

III. SECOND SYSTEM

In order to have a greater working volume and to preserve the seal from the caustic effects of the plasma, I opted to push the bell jar into the microwave oven chamber though a hole cut in the bottom. I planned to use a metal grill placed in the jar to confine the microwave energy in the top and hence separate the plasma from the delicate seal. I chanced upon a large cylindrical glass container at a boot fair ("*Flea Market*" to us *Yanks - Ed.*), the standard source of much of my equipment, vacuum pump, microwave oven etc. Much to my surprise it didn't shatter under pump down. For a seal I've used some rubber pipe (used for aerating fish tanks). I split the pipe up its length and then work it round the lip of the jar. The two ends are fixed in place with super glue. The jar is simply placed on a metal

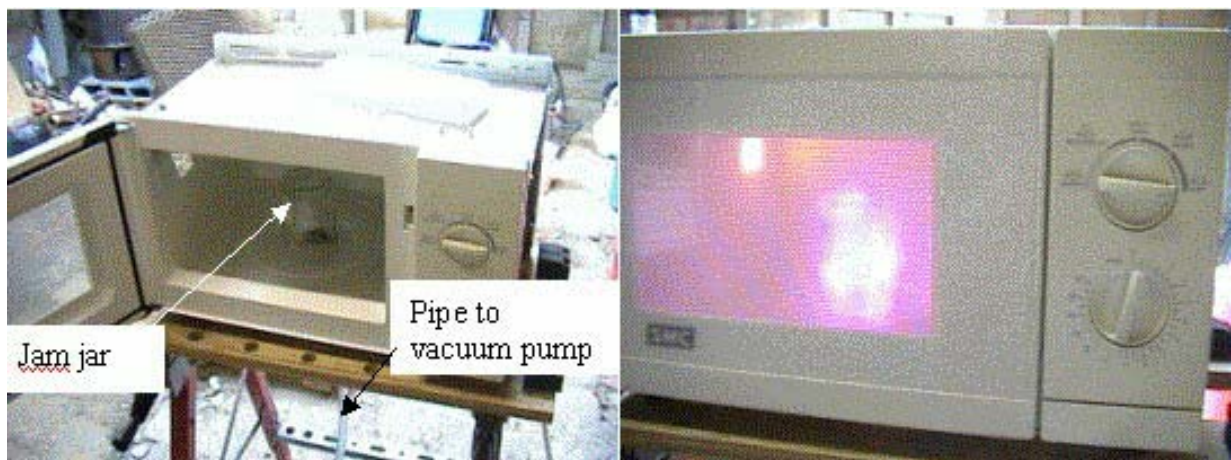


Figure 1 - Initial Microwave Plasma Generator

base with a little vacuum grease. I've made a number of vacuum chambers with Pyrex salad bowls using these rubber seals. I've used a circular steel base from an old lamp stand. I have problems trying to find thick metal bases for my bell jars. The steel based does bow under pump down but it does seem to effect the seal. I've also tried aluminium cooking pots as they seem to have a relatively thick based. However I've even managed to deform one of these on pump down. Maybe I could get better results with better materials but I'm working with what I have for the moment.

Figure 2 shows some bell jars made from common kitchen glass ware. The faint green tinge on the rim of each jar is the seal made from a split rubber pipe. The centre jar has a brass bolt electrode fitted though a hole ground in the top. Figure 3 is a detail of the baseplate shown at the center of Figure 2.

I cut a rough hole in the bottom of the microwave (see Figure 4). I was very worried that the vibrations from the cutting and filing of the hole could damage the magnetron. However I seem to be lucky this time. I left the whole rough cut to avoid unnecessary filing.

I made a cylindrical guard using a tin and piece of



Figure 2 - Bell jars made from common kitchen glassware.

grill to cover the lower half of the jar (see Figure 5). This way I could monitor the penetration of the plasma in the lower section without being cooked at the same time. The guard was flush with the bottom of the oven and the steel base. I sure that leakage of the ~12cm wavelength radiation though the small gaps left over was minimal. The jar in question has a smaller diameter at the lip than in the body. This put a stop to my initial idea of using a grill in limit the penetration of the plasma. I compromised by cutting a small circle of grill that would sit just inside the lip to the jar. First I pumped down the jar. Then I placed the cylindrical guard in place and finally I dropped the oven into place



Figure 3 - Base plate detail.

over the jar. When I turned the oven on the plasma struck immediately. I could drop the power down to almost half and maintain the plasma. The plasma did penetrate the lower half of the jar but the small circular grill seemed to be effective in stopping the plasma attacking the seal. The jar gets very hot and I'm worried about the lifetime of the system. I noticed that the discharge is not uniform. There were hot spots. These



Figure 4 - Oven with hole cut into bottom of cavity.

may arise from imperfections on the inner surface of the jar. There is also a non-uniform field distribution in the oven.

IV. FUTURE PLANS

I would like to work on a system to seed the discharge in the centre of the jar, maybe using a strategically placed metal point or an electrode connected to a high voltage unit. With this in place I would like to start some preliminary experiments on etching various materials with the plasma, even starting with air and



Figure 5 - Jar, baseplate and guard, separately (left), together (right).

working my way through different gases, argon in particular.

The weakness of the system is the glass jar. I'm not sure how long it will last. I sure that a lot of energy is wasted in heating up the glass rather than the plasma. Finally, I'm considering build a metal vacuum chamber that will double as a microwave resonant cavity. This should improve the efficiency of the system.

Editor's comments: There is a commercial adaptation of a standard household microwave oven that is offered as a low cost plasma cleaner & asher. This is the "Plasma-Preen." It is manufactured and sold by Plasmatic Systems, Inc. of North Brunswick, NJ. The developer of the product and the president of Plasmatic Systems is Aaron Ribner. Unfortunately, the company does not have a web site but the well-detailed patent (4,804,431) can be viewed on the Bell Jar's patent page. Here also will be found a link to the manual for this system.

Bubbles and Gel Candles

A project inspired by gel candle maker Deb Puerini

Steve Hansen

I. INTRODUCTION

As I've mentioned before, I regularly receive a variety of questions and requests from people who hear about this publication. The ones from the technical community are generally well reasoned. Some are obnoxious ("I'm a student at XYZ College and have a paper due tomorrow on vacuum gauges, please send me all the information you have..."). Then there are the ones from confused English majors who confuse vacuum bell jars with something written by Sylvia Plath and then accuse me of plagiarism. And then there was the "farmer" who wanted to know how to freeze dry 2 lbs. of dirt (on second thought, maybe he was trying to preserve anthrax spores?).

A while ago I got an email message from Deb Puerini, a dedicated gel candle maker who lives in Rhode Island. She wrote:

"I just stumbled upon your site from a Google search. I am a gel candle maker and recently heard about making gel candles bubble-free by (and I quote): '...that her Dad, an engineer, had built her some type of contraption that has a vacuum pump or something to cause the bubbles be under a vacuum pressure, and when they were poured they had no bubbles. I think he

should market the contraption instead of selling the candles.'

"Do you have a clue as to what this "contraption" might be? I remember in high school we had a huge glass jar (like the 5 gal water bottles) and the teacher put marshmallows in it and closed the top. It had a vacuum pump or something and the marshmallows got huge. Do you know what I'm talking about? Well if you could let me know if a thing like that could be made, I would be ever grateful if you could build such a thing to help us poor gel candlemakers who are sick of bubbles in certain candles!"

From this it was obvious that she was describing a vacuum deairing process but what the heck is a gel candle? As I quickly found out, gel candles, rather than using conventional paraffin wax, use a clear, proprietary mix of mineral oil and a polymer to which are added dyes and scents.

II. GEL CANDLE BASICS

After a bit of correspondence with Deb I learned that these candles are made by melting the gel, mixing in the various additives and then pouring the gel into a container with, at the minimum, a wick. Because of the number of degrees of freedom that one has with this clear gel, the candle maker will usually add a variety of other items. The photo of Figure 1 shows an ornate gel candle that has been made to look like a fish bowl, complete with glass fish. Since the candle maker inserts objects into the gel, the clarity of the gel is very important in many pieces and bubbles are something that the candle maker tries to avoid. The traditional way of doing this is to keep the gel hot for an extended period, permitting at least some of the bubbles to rise to the surface and break. Small bubbles and bubbles that have adhered to the immersed objects are the hardest to remove.

One of the largest manufacturers of this gel material is Pennzoil's Penreco Division (4401 Park Ave., Dickenson, TX 77539). Their gel candle product is called Versagel™ C. The product bulletin describes the generic candle making process as follows: 1) melting the raw gel at a temperature of about 100 °C and mixing in the additives, 2) pouring at about 90 °C and 3) letting the candle set in an oven at about 65 °C to remove bubbles. Hope (accompanied by a bit of cursing) is an important part of the process.



Figure 1 - Gel Candle (Fishbowl). Photo courtesy of Deb Puerini, used with permission.

III. A SIMPLE PROCESS

I figured that while deairing is a common and simple process (put the material under vacuum, pump down to enlarge, dislodge and burst the bubbles, and then let back up to atmosphere) there were some additional challenges here in terms of the gel nature of the material and having to complete the process before everything turns goeey again. So, I asked Deb for some sample candles with bubbles. These arrived within a few days.

My first attempt consisted of remelting the candles in the kitchen oven (boy did our house smell good), quickly transferring the candle to an improvised vacuum chamber and then pumping with a small rotary pump. Gauging was a simple Bourdon dial gauge, 0-30 inches Hg, atmosphere referenced.

Disaster. There was obviously some volatile component in the gel mix. The material bubbled out of control and splattered all over the vessel. (Deb informed me that bits of splattered gel are called gel boogers and that quite accurately describes the texture of this stuff.) On the plus side, the material (about half of the original amount) that remained in the candle container was bubble free.

The next attempt was more controlled. I added a pressure control feature and dropped the pressure in quick stages to about 20 inches Hg (gauge reading) where the bubbles got noticeably large and mobile, and then to 28 where they moved upward quickly and broke upon reaching the surface. In the range of 29 inches the material looked like some volatile components were

coming out. So, 28 inHg looked like a good lower pressure limit.

The final apparatus is depicted in Figure 2. The bell jar was an old pyrex jar that I got from Fisher Scientific many years ago. Inside dimensions are about 5.5" diameter and 8" tall. To keep any boogers from getting into the plumbing I put a PVC pipe cap with a small hole drilled in the side over the outlet port as a platform/baffle. The pipe nipple attached to the mounting flange is plugged with epoxy. Pressure is controlled by pinching the neoprene tubing with the pinchclamp. The pump, not shown, is a small Robinair 1.2 cfm pump, model 15200.

Figuring I'd done all I could, I packed everything up and sent it to Deb, the gel candle expert.

IV. FINAL RESULTS

Realizing that I was dealing with someone who'd never operated a vacuum pump, I waited in suspense. Two days after receiving the system Deb reported that she'd not only set it up, she had tortured a marshmallow chick, thereby replicating the classic vacuum experiment. The next day she reported results:

"Well I think I've got the system down.....the trick is to color and scent the gel, stir it up well, then stick it in the vacuum at 28 inches for at least 1 minute (still very hot) and then pour into the designated container. The proof of the pudding is waiting overnight to see what has happened during the cooling off period....well I must say this has *gotta* be the answer!!! I poured one

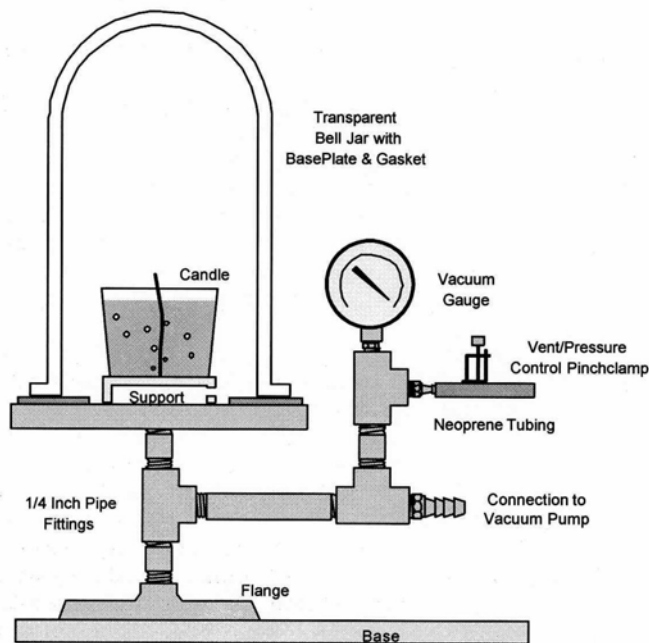


Figure 2 - Prototype Gel Candle Deairing Apparatus

last night with epsom salt at the base (for snow) and put a glass penguin in...this morning there is NOT ONE BUBBLE to be seen!!! Now the epsom salt usually presents a big bubbling problem, but wow!!! clear clear clear!!! I am sooooo excited about this!!! I poured another one just a few minutes ago with glass flowers and glass marbles in the bottom and even put glitter in....wow!

“I have ‘run the controls’ in all ways and methods, no matter how high the temperature of the gel is you will get some bubbles then you have to reheat again to get the bubbles out. Even then it always varies which is the most aggravating part. But with the vacuum method, it gets out so many bubbles as long as I have colored and scented the gel to my satisfaction, the bubbles will not come back. I’m sure if I stirred again after vacuuming they would come back. I did the most important test, silk flowers with gel, and put the container in the vacuum and it’s really something. Bubbled like all hell but then is still clear as anything!”

V. CONCLUSION

I have to say this was a fun project since I not only got someone introduced to using vacuum to practical ends but also I learned about the wonderful technology of gel

candles. (Now, can this gel stuff be used to pot high voltage electrical assemblies?)

For anyone that is interested in Deb’s excellent work, please visit her extensive Web photograph album at <http://www.netSnapShot.com/pcw/B?KEY=41&ACCOUNT=684>. She can also be reached by email at lovesalsa@cox.net.

FURTHER READING

An excellent resource for gel candle making is on the Web at:

<http://www.howtomakegelcandles.com/index.asp>

POSTSCRIPT (Feb 2006)

Deb and I turned this gelcandle debubbling process into a small business. Figure 3 shows one of our current offerings. Full details on this aspect of gelcandle making can be found at:

<http://www.belljar.net/gelcandles/main.htm>



Figure 3 - Gel Candle Deairing Apparatus with Metal Chamber

Cartesian Divers - Part 2

From Cartesian Divers to PID Pressure Control

Steve Hansen

I. INTRODUCTION

In the previous issue we looked at Cartesian divers and how they evolved into pressure measurement devices. In this final installment we will see how the Cartesian diver can be used as a simple pressure control device. The article will conclude with a look at more contemporary methods of controlling pressure within vacuum systems.

II. THE CARTESIAN DIVER AS A PRESSURE CONTROLLER

In Part 1 we concluded with Gilmont's Cartesian diver manometer as described in his U.S. patent 3,261,207. This patent also describes a pressure controller based on the Cartesian diver principle. The relevant illustrations are contained in Figure 1 on the next page. The text that follows is adapted from the description contained in the patent and is specific to the device in the right-most illustration.

Figure 1 depicts the invention as adapted for functioning as a control device ("manostat") to regulate the vacuum applied to a system.

The housing 2' is similar to that of the pressure gauge version except that the two outlet openings 6a and 6b are provided at the upper end of the tube 4' with the opening connected to a nozzle 60 which extends downward into the upper section of the tube 4', terminating in valve orifice 62. The float 16' is comprised of a thin-walled body 18' having an internal diameter only slightly less than the tube 4'. The lower end of the body 19' is constricted to form an opening 34' which has a small cross-sectional area as compared with the main body of the tube 18'.

The upper end of the body 18' has a soft elastomeric cap 24' that is in line with the opening of the nozzle 62. The device is connected to the system being controlled via port 6a and port 6b is connected to the vacuum pump. As the system is evacuated the pressure in the upper section of the chamber 4' is reduced and the float 16' rises. When the vacuum reaches a predetermined value the float will rise and the cap will contact the nozzle, closing the opening 62 and cutting off the vacuum pump from the system being controlled. When the pressure in the system rises, the float will fall opening the nozzle and the process will repeat as necessary to maintain a constant pressure in the system. Operation is enhanced by making the system more dynamic by introducing a small bleed in the system.

This improves the sensitivity of the control. The control pressure is determined primarily by the amount of gas that is present in the float 18'. Fine adjustment is achieved by means of the position of the piston 36' which is set by knob 44'. Moving the piston upward will raise the pressure set point, lowering will reduce the pressure.

Figure 2 shows a contemporary version of a Cartesian diver vacuum controller. This one is made by Ace Glass Corporation of Vineland, NJ.

For the remainder of this article we will skip to the present and look at automated systems for active closed loop pressure control of vacuum systems. A prerequisite for full understanding of the following is the article "Leaks, the Good, the Bad and the Ugly" which appeared in Volume 7, No 1. This article will also be in the second compendium.

III. MANUAL PRESSURE CONTROL

Consider the vacuum system shown in Figure 3 on page 18. Below. When the throttle valve (a variable conductance) is open and the leak valve (another variable conductance) is closed, the chamber will settle at a base pressure determined by the pump's throughput* and the sum of the sources of gases within the system. These would be from real and virtual leaks, permeation and outgassing and represent the overall gas load presented to the pump.

If we want the chamber to reach a specific pressure then we would open the leak valve by some amount, perhaps just under the desired pressure. Then we could close the throttle valve by some amount to get the system to the desired pressure.

While this technique will achieve the result of realizing a specific pressure in the chamber, there are a number of deficiencies:

- As system conditions change it will be necessary to continuously monitor and adjust the pressure control elements.
- The process requirements may require multiple gases as would be the case for a reactive process. Simple metering (conductance) valves cannot perform this task.
- It may be necessary to independently control the flow of each of the incoming gases while also controlling pressure.

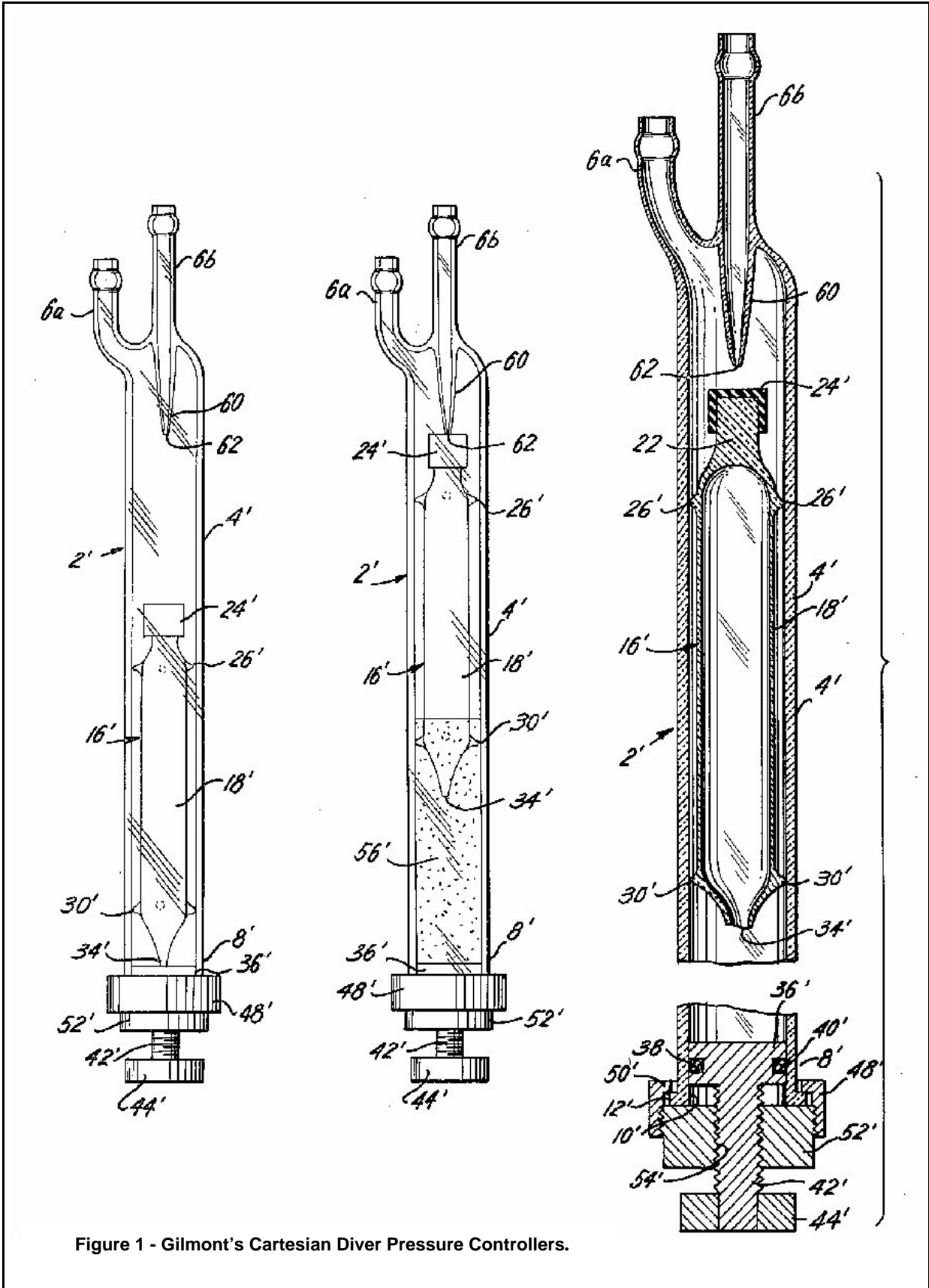


Figure 1 - Gilmont's Cartesian Diver Pressure Controllers.

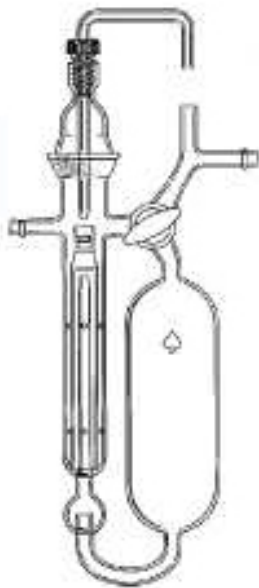


Figure 2 - Ace Glass 8741 Vacuum Regulator. "Increased sensitivity and versatility over other models, mercury requirement is reduced to 20-30cc. A control point within 1mm Hg can be fine-tuned by adjusting the height of the pump-out tubulation. The Cartesian driver float drops away from pump-out orifice on an increasing system pressure and reseals it when the reference pressure set within large bulb is reattained. Reference pressure is easily set. The spherical joint allows the attitude of the orifice to be adjusted to accommodate a small continuous leak without hunting of the float." \$430.12 in 2007.

IV. CLOSED LOOP CONTROL

In "Leaks: the Good, the Bad and the Ugly" we saw how closed loop control is applied in a mass flow controller with the control input being supplied by the flow sensor with a solenoid proportioning valve as the variable flow control element. The same approach can be applied on a larger scale to a vacuum system. Figure 4 on the next page represents an extension to the

previous example with closed loop pressure control. This particular configuration is referred to as upstream pressure control. This is because we are driving a valve that is upstream of the chamber.

Proportioning valves are an important element in vacuum systems. Figure 5 depicts the general configuration of a solenoid proportioning valve.

This type of valve uses the magnetic field developed by a solenoid to move a magnetic armature

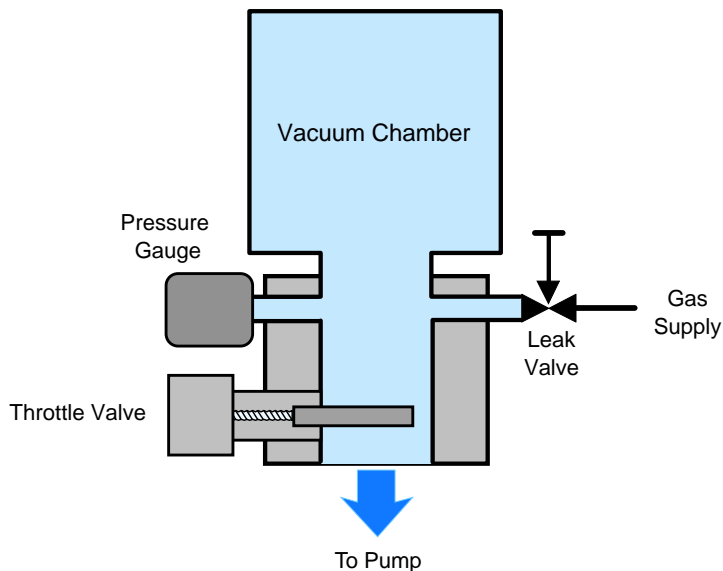


Figure 3 - Manual Pressure Control Elements

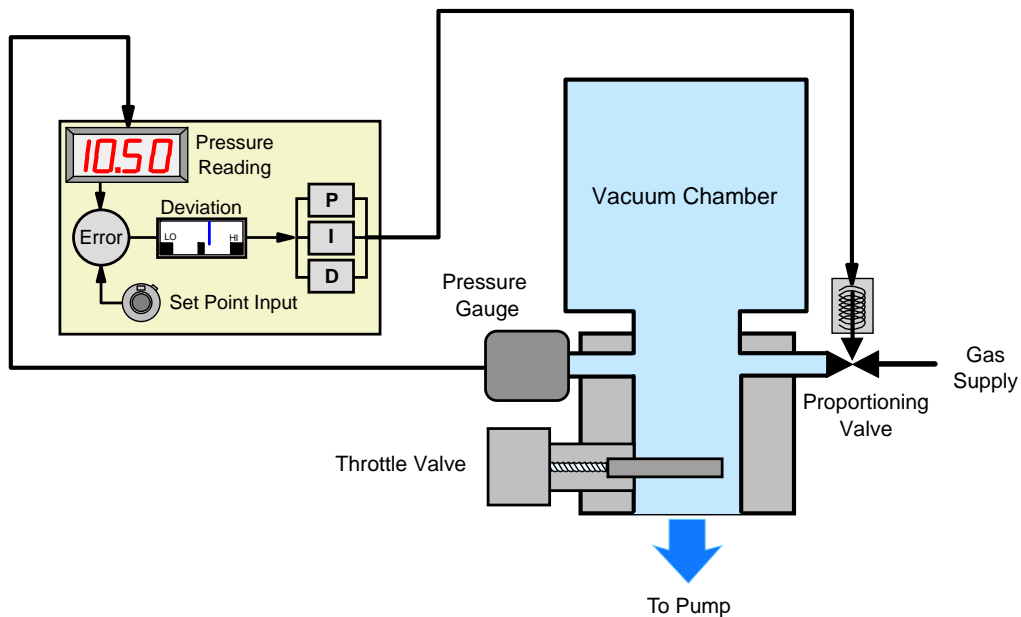


Figure 4 - System with Upstream PID Pressure Control

that is within the valve. A flat spring centers the armature and also provides a countering force to the magnetic field. The usual arrangement is normally closed - when no current is applied to the solenoid the face of the armature rests on the orifice. This face may have an elastomeric material to seal the valve or it may be a metal-non sealing configuration. It should be noted that control valves of this type are not meant to be positive shut off devices. If a positive shutoff is required, there should be a dedicated isolation valve downstream of the control valve.

The action of the valve is as a variable conductance with very high resolution. Proportioning valves are designed to be used as part of a closed loop control system with constant feedback. They are not designed as "set and forget" valves as the armature position will wander without constant correction.

The orifice is sized to the flow range required by application, and manufacturers will offer a range of flows and guidelines for selection. A typical offering would be several ranges from 10 sccm to 10,000 sccm. with the flow rating based on the orifice size under typical upstream and downstream pressure conditions

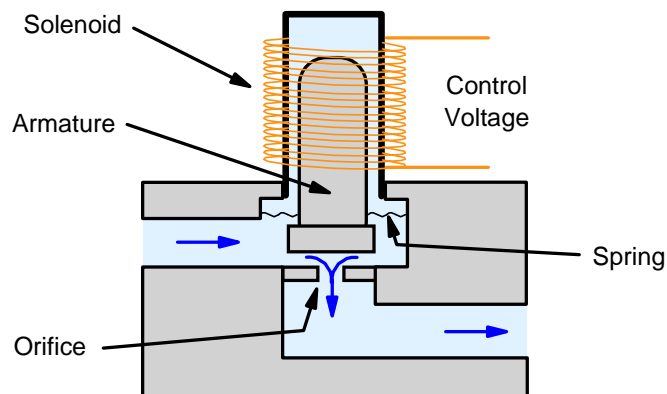


Figure 5 - Solenoid Proportioning Valve

with nitrogen gas. A detailed discussion of orifices is contained in another previous article, "The Speed of Sound, Shock Tubes and Potato Cannons." This article was in Volume 8, No. 2 and will also be included in the Second Five Years compilation.

V. PID CONROL

PID stands for Proportional - Integral - Derivative. Referring back to Figure 4, the box on the left hand side is the pressure controller. This controller has two inputs. The first is for the setpoint. This is the adjustment for the desired system pressure. The second is the actual pressure input from the vacuum system's pressure gauge. The application usually requires a capacitance gauge due to the need for accuracy, response speed and gas-type insensitivity.

These two signals feed into an error circuit. The error circuit determines the difference between the two inputs and then provides an output related to the error. To the left of the error element is a deviation indicator that provides the operator with a graphical representation of the magnitude of the error and whether the error is on the high side of the setpoint or below.

The error signal then progresses to the PID circuitry. The PID elements function as follows:

Proportional: This element provides a response that is instantaneous and is a directly proportional to the amount of error. It is also called gain. If the proportional setting is too low, there will only be a weak response to a large error. Conversely a high proportional setting will produce too

large a response to a small error with the result that the system may become unstable and oscillate.

Integral: This element provides an additional signal that is directly related to the length of time that an error signal has existed. For example, if the pressure almost reaches the setpoint but there exists a persistent error, the integral element will integrate the error with time and provide a correction.

Derivative: This element provides a signal that is proportional to the rate of change of the error signal. It slows down the response as the system approaches the setpoint. This element is used to reduce the amount of overshoot that may be produced by the other elements but will also slow down the overall time for the system to reach the setpoint.

As a thought experiment, think about your actions when you step into a small shower stall and turn the water on after entering. You become part of a closed loop control system and unconsciously are using each of the elements described above. Depending upon the temperature of the water, your proportional element may be set very high.

Some representative response curves are shown in Figure 6. "A" represents the ideal response to the setpoint change. The response in "B" is slow with no

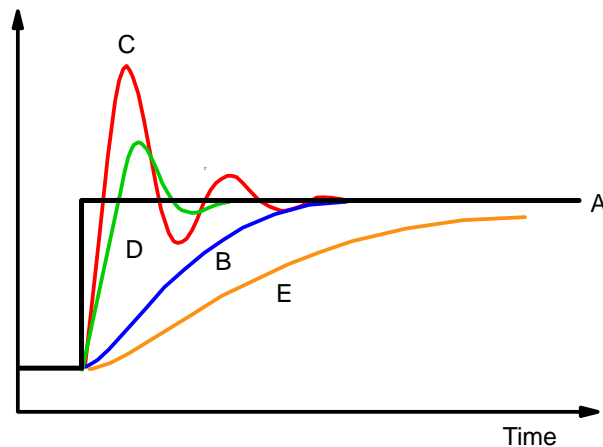


Figure 6 - System Response to Various PID Controller Settings.

overshoot. In this case the derivative element would be fairly strong. In the case of “C” the system reaches the setpoint quickly but overshoots and oscillates about the setpoint for a few cycles. Increasing the proportional term would probably drive the system into oscillation. “D” also overshoots but the system settles fairly quickly. Finally, “E” represents a very slow response system with a persistent error that requires attention from the integral element.

The type of response that is applicable to a given vacuum system is highly dependent upon the application. In some cases overshoot is not desirable but on the other hand, time is money and getting to setpoint quickly can be very important in terms of manufacturing efficiency.

Upstream control is the simplest pressure control mode and can permit a single gas (or a premix of gases) to enter the system. Common examples of systems using upstream control include freeze dryers and single gas sputter deposition. Both require accurate pressure control and a specific gas for the process - nitrogen for freeze drying and argon for sputtering.

VI. RATIO UPSTREAM CONTROL

This configuration is required where it is necessary to control pressure and also introduce multiple gases where the mass flows of the gases have to be supplied in a specific ratios due to reaction requirements.

One of the most common examples of a process that uses ratio control is reactive sputtering. The parameters that must be controlled are the chamber pressure and the ratio of argon to the reactive gas (oxygen or nitrogen). A typical ratio for this application might be 95% argon to 5% oxygen.

The solution is to replace the simple proportioning valve with a pair of mass flow controllers. Each gas is controlled by one of the MFCs in such a way that the proper mass ratios are maintained.

Referring to Figure 7, the output of the pressure controller is fed to a ratio control device. This simply apportions the total signal required between the two MFCs. If MFC “A” is defined as the master controller that is delivering some time varying Q , then MFC “B” will deliver some constant fraction of what “A” is delivering. The net result is a constant split or ratio of Q between the two MFCs but the total flow is adjusted in response to the pressure controller.

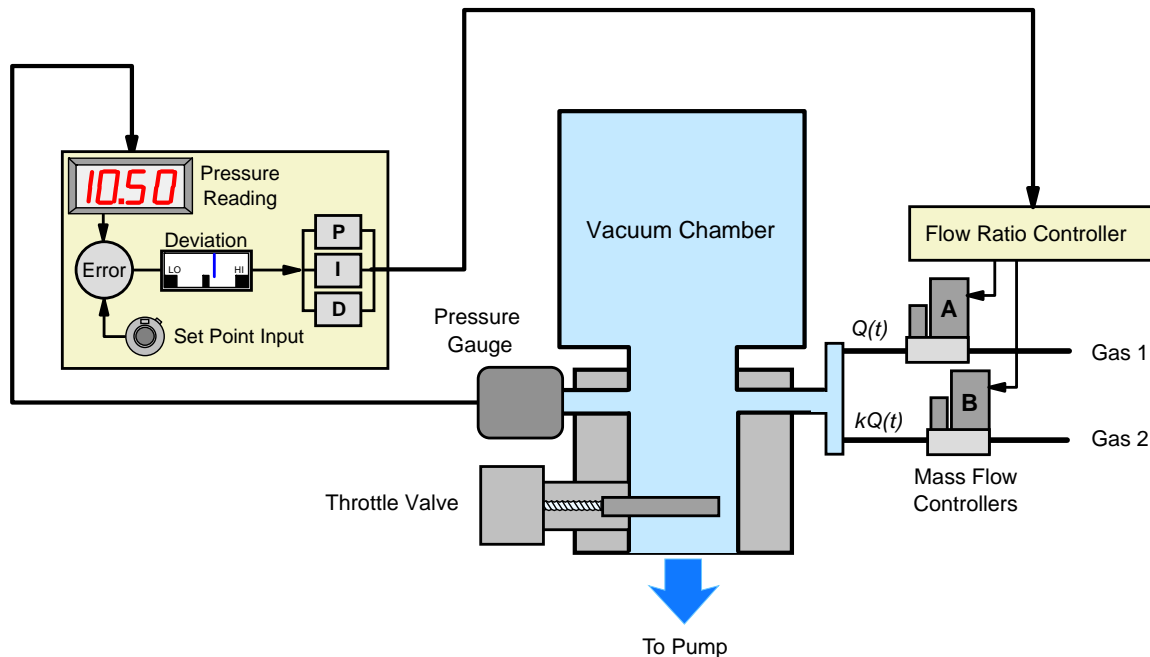


Figure 7 - Ratio Upstream Control

VII. DOWNSTREAM CONTROL

This is the most complex and flexible of the pressure control methods. Downstream control is utilized when it is necessary to control the absolute quantities of each gas admitted to the system while also controlling pressure. Each gas will be controlled by an MFC, each one of which is providing a specific Q . This setup only controls the gas input to the system, not the pressure.

The pressure is controlled with a motorized throttle valve that is located between the chamber and the pump. The name of this configuration is derived from the fact

that the pressure control element is downstream of the chamber. Figure 8 depicts this configuration.

Downstream control is commonly used with processes such as chemical vapor deposition and reactive etching where the reactant gases must be admitted not only in a specific ratio but also at a specific rate. This precludes ratio control as a method. In addition to this, the process pressure also has to be controlled within tight limits. Some processes may also change the gas flow rates during the process or introduce other gases through additional flow controllers.

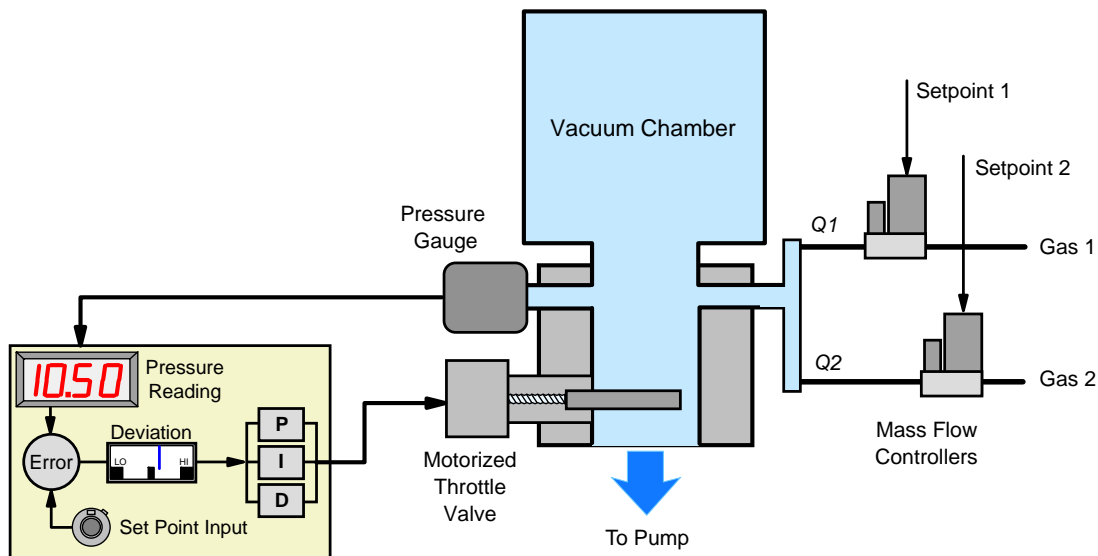


Figure 8 - Downstream Control