Lesker Tech "...vacuum science is our business."

Volume 4, Issue 1 January, 2005

Pumping Up Your Applications

Part 3A

Introduction

[This issue's gestation period was so long I'd like to blame someone else. Unfortunately, mea culpa. It is also so physically long I divided it into part 3A and 3B. I refuse to make another four part trilogy.]

New readers, please don't start here! Indeed, all readers are urged to skim through *Lesker Tech Volume 3, Issues 1 & 2* once more before jumping into *selecting the right pump by* ***understanding the application.***

Let's be clear. If you make the wrong '*horses for courses*' pump choice, an application can destroy the pump, ruin product, raze the building, or give you an early case of *rigor mortis*. To heighten your awareness, I start with five examples of **bad pump choices**. If the writing style suggests I'm making light of these situations, you couldn't be more wrong. In two cases, people might have died!

Then comes **applications classification** based on the physical and chemical nature of the *stuff* heading towards the pumps. Here *stuff* means, collectively: gases, vapors, aerosols, droplets, bulk liquids, dust, and particles of similar *chemical reactivity* or *physical nature*.

Three final introductory comments:

- Nothing written here really applies to the semiconductor industry. The amount of villainous *stuff* used by those folks is way beyond the modest aims of this article.

- I cannot over-stress this: I'm only offering a basic compatibility list. Your specific application may have so many, un-anticipate-able 'gotchas' that it's a death-trap waiting to be sprung. For any application, you **must** use your own smarts and process knowledge to make your final pump choices. Relying solely on my 'long distance' diagnosis is putting Janet Jackson in charge of your wardrobe—something will malfunction.

- If you disliked chemistry at school, you'll hate this.

Five Killer Apps

(Perhaps I should point out—we had no part in designing or building these systems.)

Ever heard John Madden talk about some football team's line-backers? "When you least expect them—Boom! They're right there." That sums up vacuum-related killer apps. In this short list, some had serious safety implications while others were Oscar-quality in the 'Surreal Pump Choice' category.

1. Pure Gas killer.

At first sight, pumping xenon with a turbo pump is the ultimate non-issue. It's non-reactive, like argon, and heavy. What's the big deal? Xe's mass is ~3x greater than Ar's and, with some dubious extrapolation, we can predict its compression ratio in the pump is about ~4x higher. While that doesn't sound huge, it roughly doubles the exhaust gas temperature. And that's a problem.

An R&D facility's first experiments with Xe destroyed three turbos in quick succession before the pump manufacturer accepted it wasn't just 'operator error'. The manufacturer investigated, promptly blew up another pump, and became instant believers. Pumping Xe is quite tricky.
(See www.lesker.com/PumpingXenon.cfm)

2. Process Gas killer.

A lady (that's not chauvinism—you'll see) wrote to TechInfo, "*After sputtering aluminum, there's no smell. But when I sputter selenium, the chamber smells very bad.*" I shot back an **URGENT** email forbidding chamber sniffing and explained, the system was making H_2Se. According to various MSDSs, H_2Se is ~200 times more poisonous than the chemically similar H_2S (rotten eggs), yet shares the sneaky characteristic of paralyzing your olfactory nerves, making it odorless just before you keel over and play permanently dead.

The system was small and the argon sputter gas was evacuated though an untrapped oil-sealed rotary vane pump. So we had: backstreaming hydrocarbon oil vapor (which contains hydrogen); sputtered Se atoms; and a plasma to stir up reactions. What better H_2Se generator is there? Why give the questioner's gender? OK, so you're a guy. Before reading this, could you imagine asking a tech info service why your chamber stank?

3. Backing Pump killer.

CDs are coated with aluminum in a high speed process so they're cheap. ($16.00 is cheap?) A CD-coating equipment maker was introducing a new product line which had a medium speed turbo, a small backing pump, and an exceptionally tiny foreline volume. The time scale for the chamber cycling between two pressures (as the process requires) is seconds.

During tests on three pre-production systems, all three pumps crashed in quick succession. When dismantled, the turbo's bottom rotor blade set, *in each pump*, had chewed into the stator blades above. In the ensuing 'headless chicken' phase we were asked to help explain.

The turbo was correctly sized for the pumping time allowed. But the foreline. . . ? Its minuscule volume coupled with an inadequate backing pump meant its calculated pressure spiked over 10 torr. Whatever pressure was reached, apparently it was enough to cause the rotor's blade tips to bend up—like a helicopter going for maximum lift—and slash into the stator above.

4. Pump Fluid killer.

A company casting metals under vacuum used large chambers pumped by big diffusion pumps. When in operation, the melt-furnace's heaters were at ~1800°C, well above the flash-point of the pump's silicone oil. But, who cares? The furnace wasn't switched on until the chamber reached 10^{-5} torr—what's to explode? Except the inevitable happened. Something in the foreline let loose during a melt and air roared back through the diffusion pump, carrying with it scads of silicone oil vapor.

The explosion didn't injure anyone only because the chamber's bottom surface had a large unused flange. Its bolts sheared and the flange blew down into the cage-like structure supporting the chamber. Many corporate 'suits' eased their shirt collars with crooked fingers as the dust settled on that little event. But they increased the budget to include inert fluid for all the diffusion pumps.

5. No Pump Capacity killer.

A metal refining company made 40 tons of titanium bar with something like 10 times the allowable H_2 concentration. Degassing Ti involves heating to high temperature for many hours and pumping away the H_2 as it evolves. For this job the company re-commissioned an old 90' by 7' diam. vacuum furnace.

Originally the furnace had three 30" diffusion pump stations at the middle and ends. But some joker had persuaded the company to dump the 'old technology' diff pumps and re-fit with cryopumps of similar diameter. We were called in when these 'modern wonders' plainly did diddly for H_2.

It took just a few minutes to find the cryopump's spec sheet. There, large as life, was its total H_2 capacity before regeneration was needed. Trivial arithmetic showed one furnace-load of titanium required ~350 such pumps.

And the beautiful golden statuette goes to. . . ?

Classifying Applications by Chemistry

In **Table 2**, I've grouped the 'Pumped *Stuffs*' by (loose) similarities in chemical reactivity or physical properties. (Where's Table 1? Ha! You didn't read *Lesker Tech Volume 3, Issue 2*, did you!) The drill-down in **Table 2** identifies some of the specific chemicals in that sub-group, but obviously only a fraction of all possible *stuffs*. With luck, the more detailed sections given later provide a framework for informed decisions about the appropriate pumps for your application. Just make sure you identify all the 'gotchas'.

Notice I've identified each sub-group with a letter: **A**, **B**, **C**, etc. This is a navigational aid. For example, if you're about to pump *spontaneously combustible gases*, the table label is "**J**". To find pumping suggestions, look for the section sub-headed "**J**" (in Part 3B)

OK, So What Pump?

Applications often involve multi-component mixtures so consider **all** categories of *stuff*. Think, "Which stuff scares me the most? What makes me run for my flack jacket, leather underwear, scuba gear, and Darth Vader helmet?" Head for the section dealing with that stuff and choose the pump.

To rate compatibilities between pumps and applications, I've invented a **Rating Scale**.

Pump Rating Scale

No.	Meaning
1	OK (in most cases) *if. . .*
2	Possibly OK to so-so *if. . .*
3	Poor choice
4	Won't work and/or dangerous

Table 2 - Applications Classified by *Stuff*

Pumped *Stuffs*	Sub-Group		Typical Examples
No stuff added		**A**	
Passive		**B**	Air, N_2, CO_2, CO, He, Ne, Ar, Kr
Corrosive	Plasmas	**C**	He, Ar, N_2, O_2, CF_4, CF_4+O_2
	Halogens	**D**	F_2, Cl_2, Br_2, I_2
	Halogen acids	**E**	HF, HCl, HBr, HI
	Lewis acids	**F**	$AlCl_3$, BF_3, BCl_3
	Others	**G**	H_2S, NH_3, NO_x, SO_2, etc
Flammable	Hydrogen	**H**	H_2
	Hydrocarbons	**I**	CH_4, C_2H_2, C_2H_4, C_2H_6, etc,
	Spont. combust.	**J**	B_2H_6, AsH_3, PH_3, SbH_3
	Oxidants	**K**	O_2, O_3, H_2O_2, ethylene oxide
	Others	**L**	H_2N_4, CH_3NH_2, $(CH_3)_2NH$, H_2Se, etc
Solvent		**M**	Water, benzene, toluene, ethanol, methanol, acetone, MEK, methylene chloride, etc
Monomeric		**N**	Styrene, methyl methacrylate, acetylene, 1,3-butadiene, methyl acetylene, vinylidene chloride, etc
Particulate	(existing)	**O**	$AlCl_3$, SiO_2, SiO, flakes from prior depositions
	(formed in pump)	**P**	$SiH_2Cl_2 \rightarrow SiO_2$, $BCl_3 \rightarrow$ (hydrated boron oxide?), etc
Droplets		**Q**	H_2O, quenching oil, epoxy, etc
(High Temp)		**R**	Melting, degassing, substrate heating

The "*if . . .*" lets me to add qualifiers for the particular application. That is, I'm trying to address issues that start with phrases like: *these pumps are only suitable for this application if they are/have. . .* or; *if you add . . . to the pumping system*.

In this list of issues, the 'operative' word in the rating is bold-italic:

- ***particle*** filtration for pump fluids
- ***acid***/alkali filtration for pump oils
- ***dust*** filtration for gases
- ***bleed*** of inert gas for bearings (turbos)
- ***bleed*** of inert gas for oil case (oil-sealed pumps)
- ***mag-lev*** bearings (turbos) to avoid lubricating greases
- gas ***ballast*** using inert gas
- ***inert*** pump fluids
- ***corrosion*** resistance design
- ***semicon***ductor industry versions of the pumps noted
- ***examine*** often for corrosion or accumulated deposits
- ***regen***eration causes high concentration of *stuff* near the absorbate

And some obvious, but often forgotten slew of cautionary caveats and generous generalizations about pump selection:

- If *stuff* is really corrosive, even a corrosion resistant pump's life may be short.
- If *stuff* is flammable, a pump doesn't change that.
- For capture pumps, during regen, *stuff* is present in high concentration near the pump's absorbate. If a reaction occurs, the pump's capacity for light gases is shot.
- A transfer pump's exhaust contains *stuff* that went in the inlet. Depending on the *stuff's* nature, the pump's exhaust gas may need: high dilution; venting from room/building; or exhaust gas abatement.
- Pre- and post-pump traps/filters mostly work. But it's a good idea to believe they don't. And remember, as you open a trap, you are exposing *stuff* to air and yourself to *stuff.*
- Oil-sealed pumps sometimes trap *stuff* in the oil.
- Liquid ring and steam ejector pumps may handle nasty *stuff* but you must then deal with contaminated waste water.
- For corrosive *stuff,* frequently inspect the pump's ports for etching or solids build-up. But inspect with caution—stuff may still be in the pump.

Finally—every application has: an effective pumping speed; required base and working pressures; and pump throughput questions that must be addressed and answered (for help, see many earlier *Lesker Techs*).

Now we are ready to make a stab at choosing a pump from the selection tables below. But again heed my warning: ***at best, this is just a list of possibly compatible pumps.***

A. No Stuff Added

Select a pump mechanism that, in addition to reaching the desired pressure, doesn't *intrude*. For example, if a hint of oil vapor will cause problems, use dry pumps. If vibration upsets your apple-cart, use vibration-free pumps. If 'utilities' are restricted at the installation site, don't select a pump that needs N_2 gas, cooling water, 3-phase power, or whatever you don't have. If loud continuous noises turn you into a basket case, buy a quiet pump. In summary, use common sense!

B. Passive Gases

Excluding helium for a moment, just about any pump will work. Again, the selection must reach the needed pressure and not intrude. It must also handle the total gas load when the process is going full tilt. For high gas loads, look at transfer pumps. For even higher gas loads, buy your way out. . . meaning, select bigger pumps or more of them. But be smart—figure out the necessary Effective Pumping Speeds *while still designing the system*. You don't want incompatible gas flows and pump capacities to come as an operational surprise.

For helium the issues are:
- Doesn't react—reactive capture pumps won't work
- High VP at low temperatures—cryos won't work
- High velocity—turbos have poor compression ratios

Obviously, no other inert gases react but they're OK with cryos and turbos. For He see **Table 3** for suggestions.

Table 3 - Pumping Helium

Vacuum	Pump Selection
UHV	You're in trouble!
High Vac	• Diffusion: 1 • Turbo: 3 • Hybrid turbos: 1 • Cryos: 3 - 4
Rough	• Rotary vane: 1 • Rotary piston: 1 • Dry piston: 2 • Scrolls: 2 -1 • Roots: 3 - 4
Coarse	• Diaphragm: 1 • Screw, Roots, Claw: 3

C. Corrosive Gases—Plasmas

I know. As soon as you saw Ar listed as a corrosive, you thought, "Is he out of his gourd?" The annoying fact is, a plasma converts just about anything to an oxidizing agent. In case your corrosion chemistry is rusty, go to en.wikipedia.org and search, separately, for *corrosion* and *oxidation*. If a phantom *wiki* editor hasn't struck, you'll find *corrosion* implicates oxidation and *oxidation* includes the phrase: ". . . *oxidant removes electrons from the substance*." That is, anything which attracts an electron is an oxidant. Guess what. . . any positive ion wants an electron in the worst way. Added to that, Ar^+ has exactly the same electronic shell configuration as atomic chlorine—an exceptionally corrosive element (see **D**).

Oh sure, there are differences. If Ar^+ hits a grounded metal, it grabs an electron and runs. The Cl atom, while snagging its electron, sticks to the metal as a chloride. But what if Ar^+ (or any other + ion) hits an insulator, like an O-ring, pump oil, or grease and whizzes off with the electron? Can molecular bond destruction be far behind?

In a sense, Ar^+ is a *temporary* oxidant. Once it gets its electron, it's back to being inert.

I lump plasmas into either *OK* or *PIT* (pain-in-the. . .) types. A good example of an *OK* plasma is a magnetron sputter gun operating with argon. The Ar^+ is formed in a (small) plasma volume. Any ion that escapes has opportunity to neutralize and lose energy before hitting the pumps. A *PIT* plasma either: (a) fills the chamber with plasma and/or; (b) makes chemical nasties from innocuous starting gases. Examples of *PIT* (a) are chamber-cleaning plasmas and some types of plasma etching. The active species formed near the pumping port have little opportunity to 'un-form' and there's trouble in pump-city.

The classic example of *PIT* (b) is CF_4+O_2 mixture used in aggressive plasma etching. Other than the cautions urged when pumping O_2, the start gases aren't a problem. But the plasma products, even from small localized plasmas, include F_2 (a *permanent* oxidant). It doesn't matter where it's made—watch out when that goodie hits the pumps!

How does this all play into pump selection? For *OK* plasmas, it's OK to use 'normal' high and rough vacuum pumps. But regularly check the oil's pH to avoid corrosion surprises. At the first hint of trouble, change oil. Compared to a pump rebuild, an oil change costs peanuts.

For *PIT* plasmas, my suggestions are in **Table 4**:

Table 4 - Pumping *PIT* Plasmas

Vacuum	Pump Selection
High	• Diffusion: 1, *inert ** • Turbos: 1-2, *corrosion, bleed* • Cryos: 1-2, *regen* • Getters: 4
Rough	• Rotary vane: 1, *corrosive, inert*, ballast, bleed* • Roots: 1, *semicon* • Scrolls: 4 • Dry piston: 2-3
Coarse	• Diaphragm: 1, *corrosion* • Screw, Claw: 1, *semicon, ballast*

* *vapor backstreaming may give F^+ in plasma*

D. Corrosive Gases—Halogens

As corrosive elements go, the halogens are the periodic table's Hannibal Lectors, each with its own special niche of nastiness. The compatibility between the dry gases and construction materials used by the vacuum industry is, according to one web site, given in **Table 5**:

Table 5 - Halogen Compatibility

Halogen	Attacks	OK With
Fluorine	304L, Ti, Cu, cast iron, buna	316L, Al
Chlorine	Al, Ti	Cu, 304L, Viton®
Bromine	All of them	Viton®
Iodine	304L, 316L, Cu, cast iron	Ti, Al, bronze, Viton®

I think the "Attacks" column is a little light. The resistance of 304L and 316L stainless steels to chlorine isn't demonstrably wonderful. Could it be that 'dry' means 'super dry' and I've only seen the affects of wet gas?

But is this level of detail necessary when selecting a pump? Aeons ago we suggested a ordinary mechanical pump for an application with lots of iodine vapor. A week later the pump quit, its exhaust valve propped open with lumps of brown sludge (ferric iodide formed from the cast iron). Yeah, it's best to note the details.

Halogens in vacuum processes are often accompanied by halogen acids, so, the appropriate pump guides are combined in **Table 6**.

E. Corrosive Gases-Halogen Acids

Although less information is readily available on halogen acid gases, it's reasonable to assume they are as corrosive as halogens—with a twist. Chances are, if a halogen doesn't react with a material, the halogen acid will. Ain't chemistry fun? When choosing a pump for halogens and halogen acids:

- Choose the most corrosive resistant versions available.
- Those needing fluids must be filled with Fomblin®.
- Elastomer seals/gaskets must be Viton® or Kalrez®.
- If the pump has a bleed option for bearing protection, use it, with high flows of inert gas.

Use **Table 6** as a guide but, most importantly, if you don't want to be suddenly surprised by those linebackers, examine the pump regularly.

Table 6 - Corrosive Gases–Halogens & Acids

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 2, *corrosion, bleed* • Cryos: 1, *regen* • Getters: 4
Rough	• Rotary vane: 1, *corrosion, inert, ballast, bleed* • Screw, Roots, Claw: 1, *semicon, ballast* • Scroll: 4 • Dry piston: 4
Coarse	• Diaphragm: 1, *corrosion* • Liquid Ring: 2-3, *corrosion* • Steam ejectors: 2-3, *corrosion*

F. Corrosive Gases—Lewis Acids

Any definition of a Lewis acid includes phrases like 'electron-pair acceptor' and who needs the aggravation of learning what that means? Ignore the definition, the simplest examples are: BF_3 (gas), BCl_3 (liquid), and $AlCl_3$ (solid).

Add a little residual water to these babies and **shazam!** instant halogen acid gas and a solid mess. And just who's daft enough to get these arcane chemical wonders in their vacuum chambers? Oh, only those decidedly-non-daft folks making almost every computer chip on the planet.

Since we've looked at pumping halogen acid gases in **E**, it's the Lewis acid solids we must watch for. That means:

- Filtration for ***dust*** particles ahead of rough pumps
- Oil filtration for ***particles*** in oil-filled rough pumps
- ***Acid***-removing filtration in oil-filled rough pumps

Use **Table 6** as a guide, add as much filtration as you can, and employ the old fire-station drill-frequent *inspection* and lashings of *maintenance*.

G. Corrosive Gases—Others

These are a hotchpotch of alkaline, acid, and neutral materials that are selectively corrosive. There's a problem trying to advise you about the interaction of pump construction materials and gases. 'Informed' opinions vary. . . often wildly for elastomer reactions. I chose what I think are reliable sources.

• **Hydrogen sulfide** (H_2S) attacks Cu, 304L, cast iron, buna, but is OK with Al, 316L, Viton®.

• **Ammonia** (NH_3) attacks Cu, Viton®, but is OK with Al, SS, cast iron and so-so with buna. Inspect gaskets regularly and replace.

• **Nitrogen oxides** (NO_x) covers a multitude of sins: N_2O is a giggle since it doesn't attack anything; no-one mentions NO (except the makers of Levitra®, Cialis®, and Viagra®); NO_2 with water gives an acid which corrodes most metals but is OK with Viton®. And N_2O_4 chews the socks off all elastomers, Cu , and Al.

• **Sulfur dioxide** (SO_2) attacks 304L and buna, but is OK with 316L, Viton®.

Clearly, you must look at the particular gas's properties and the pump's constructional materials. Often, you won't find a pump that is completely attack-proof. So, choose one with a minimum of critical bits that react. Try to find pumps with bearings that are outside the vacuum volume or have a bleed option. And make sure the gasket materials are compatible.

Table 7 - Pumping Other Corrosive Gases

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 1, *corrosion, bleed* • Cryos: 1, *regen* • Getters: 3-4
Rough	• Diaphragm: 1, *corrosion* • Rotary vane: 2, *corrosion, inert, acid* • Screw, Roots, Claw: 1, *semicon* • Scroll: 3-4 • Dry piston: 3-4
Coarse	• Liquid Ring: 1-2 • Steam ejectors: 1-2 • Screw, Roots, Claw: 1, *semicon*

Table 7 is only a vague guide. Pump compatibility starts, of course, with the chemical nature of *stuff*.

[Part 3B will follow shortly to complete this discussion. All trademark information will appear in 3B.]

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If you believe a coleague would enjoy reading *Lesker Tech*, please feel free to forward this issue along with our compliments.

As always, your comments and suggestions are valued and welcomed.

Kurt J. Lesker Company

Lesker On The Road

Date	Event	Location	Booth
Feb28 - Mar 3	PITTCON 2005	Orlando, FL	4519
MAR 14-15	FL AVS & Microscopy Soc.	Orlando, FL	
MAR 15-17	Semicon China	Shanghai, China	4575
MAR 21-23	APS (Amer Physical Soc)	Los Angeles, CA	601
MAR 22-23	No CA AVS & ICMI	Santa Clara, CA	133

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