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Practical Vacuum: Good Design, Procedures and Maintenance Equals Good Vacuum

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Vacuum is used to control the environment in many heat treatment processes and the vacuum pumping systems used may be simple or complex. This article offers practical recommendations to handle operational issues that can arise with these systems to optimize performance, reliability and safety.

The application of vacuum in industrial processes is widespread with the heat treating industry using vacuum in a broad range of processes, many of which place particular demands on the vacuum pumps. Today, there are many types of vacuum pumps available, from traditional oil-sealed rotary vane pumps to the so called "dry" pumps developed for use in the semiconductor industry, and which have found their way into industrial processes. As well as a choice of pumping technologies, a range of installation and configuration possibilities have arisen to ensure that pumps operating in difficult applications have the best chance to provide a reliable, consistent and cost-effective vacuum solution. Pumping-system configuration can be just as important as the selection of the pump technology itself, and making what may appear to be small changes in configuration can make significant improvements to vacuum pump reliability and reduce intervention. This article is intended to provide the practical means to enhance vacuum pump performance and reliability for a range of vacuum heat treating processes.

Fig. 1. Oil sealed piston pump with mechanical booster and oil purifier

Why use vacuum?

Industrial heat treating processes such as hardening, tempering and annealing have traditionally been carried out at or near atmospheric pressure either in air or in a controlled atmosphere. The cost of providing a vacuum environment for these processes is not insignificant, typically requiring a substantial chamber designed to withstand atmospheric pressure, batch rather than continuous processes and the cost of providing and maintaining the vacuum plant and equipment. Despite this, the benefits of the vacuum process can offset these disadvantages by providing:

- Unoxidized bright parts with no subsequent cleaning required
- Energy efficiency by eliminating heat loss via conduction or convection
- Low environmental emissions of waste gases and heat
- Reduced use of process gas
- Fast, uniform heating, which reduces distortion
- Consistent and reproducible product quality
- Faster processing to increase product throughput

Typical vacuum heat treatment processes and problems

Hardening: Nitriding

Nitriding, carried out at temperatures between 500 and 600°C (930 to 1110°F), increases surface hardness by nitrogen diffusion. Piston rings, gears and crankshafts are nitrided to improve fatigue and wear resistance. Nitrogen and ammonia, decomposed at furnace temperatures, provide the free nitrogen for diffusion into the metal surface.

Typically, Roots-type mechanical boosters with oil-sealed piston or dry backing pumps are used to remove ammonia and flammable hydrogen by-products from the process. In general, the process does not produce significant amounts of particulates. Hydrocarbon lubricants used in oil-sealed pumps can accumulate moisture released under vacuum from the large surfaces of the process load. This moisture, combined with ammonia passing through the pump, can cause corrosion of pump components and attack the pump lubricant. Additionally, ammonia adsorbed into the pump lubricant may attack pump elastomers (shaft seals in particular) causing oil leakage. Dry vacuum pumps do not contain lubricants in the swept volume and they do not retain moisture from the process, while the pump shaft seal purge protects sealing and drive components from ammonia. As such, dry pumps are resistant to corrosion (as long as water vapor condensation is avoided) and can, therefore, operate reliably on this process.

Hardening: Ion nitriding
Ion nitriding is a process where a nitrogen gas plasma between 1 and 13 mbar (0.75 and 10 torr) is used to diffuse nitrogen into the product when heated to a temperature between 500 and 600°C. Typical gases used are nitrogen and hydrogen, and the lack of ammonia avoids many of the problems associated with conventional nitriding.

Hardening: Low-pressure carburizing (LPC)
LPC is carried out at pressures below 50 mbar (37.5 torr) and temperatures between 790 and 1080°C (1455 and 1975°F). LPC produces a controlled diffusion of carbon into the component surface and when followed by quenching and tempering, results in an increase in surface hardness. Typically pure hydrocarbons like propane, ethylene and acetylene decomposed at furnace temperatures above ~ 800°C (1470°F) provide the free carbon for diffusion. With the exception of acetylene, these gases can form soot and sticky tar-like deposits in the furnace and pumps resulting in damage or pump lock-up when shut down.

A flow of "solvent" oil through the pump mechanism can be used to dissolve and remove tars. Oil-sealed vacuum pumps (Fig. 1), by design, use a solvent in the form of the hydrocarbon lubricant, which may contain detergents and dispersants to aid in the solubility of tars. They require only an oil purifier circuit to continually remove any insoluble particles and regular oil changes to provide a reasonably robust pumping solution. As would be the case in most particulate applications, the predominantly noncontacting rotary piston pump mechanism is more robust than rotary vane type pumps, where vanes and stator operate in physical contact and can suffer wear in the presence of particulates.

Dry pumps (Fig. 2) require a solvent flush through the pump mechanism to be configured, or tar build-up can eventually stop the pump or cause a restart failure. However, a wider range of lower cost solvents can be used, because the solvent flush does not need to provide lubrication.

In LPC, it is essential to use inert nitrogen purges on the pump (e.g., via gas ballast, or via seal purge) to both remove residual air and reduce flammable gas concentrations due to the flammability of source and by-product gases. It is also important to ensure exhaust gases are vented safely.

Hardening: Heating and cooling (quenching)
A heat treatment may include convection heating of the product to the required temperature and a controlled gas cooling process (quench) to provide the right material properties, or simply to allow the product to be removed from the furnace without oxidation. Gases (Fig. 3) used to aid convective heating/cooling may be flammable (hydrogen) or asphyxiant (argon, helium and nitrogen) and cannot be vented to the working environment. Typically, they are removed via the vacuum pumps before opening the chamber, and some of these gases have properties that pose specific problems to the vacuum pumps.

Hydrogen presents a flammability risk with all mechanical pumps and, similar to helium, is a low molecular weight gas,

Fig. 2. Multistage drypump with mechanical booster and solvent oil re-circulation system

Filter selection and maintenance
All metallurgical processes (except perhaps simple tempering and annealing) will generate contamination in some form, so a filter should be used by default to protect vacuum pumps. Questions about filters are: Which type of filter is best, where should it be located, how big should it be, as well as are filters fitted properly, are they correct for the pressure and flow required and are they maintained?

Filters are sized according to the required volume flow rate expected and to minimize pressure drop (which translates to pumping speed loss). Therefore, their specification depends on their location in the vacuum pumping system. Because mechanical vacuum boosters are generally tolerant to most types of contamination, the economical solution would be to place the filter in front of the primary (backing) pump since the volumetric flow rate here is lowest and a small filter can be used.

Practical experience in metallurgical applications indicates that for flow rates up to ~3,000 m^3/h (1,765 cfm) for general duties, metal mesh filters offer the best compromise between filtration efficiency, impact on vacuum performance and operating costs. In contrast to paper and synthetic fiber disposable elements, metal mesh elements are generally easy to clean, are resistant to clogging, can be wetted without blocking and can tolerate high gas temperatures. Note that in general, metal mesh filters can be oil-wetted to improve capture efficiency if the backing pump is an oil-sealed type where a little oil carryover is unimportant. Oil carryover to a dry pump may wet the mechanism causing fine dust passing the filter to "hang up," so the mesh should be kept dry for these types of pumps.

Cyclone separators are recommended for use in applications generating very high loads of larger particles. They will not clog and can be located at the chamber to protect all the vacuum pumps because they offer minimal impedance to gas flow. Packed columns of Pall rings (or Raschig rings) can also be very effective without loosing

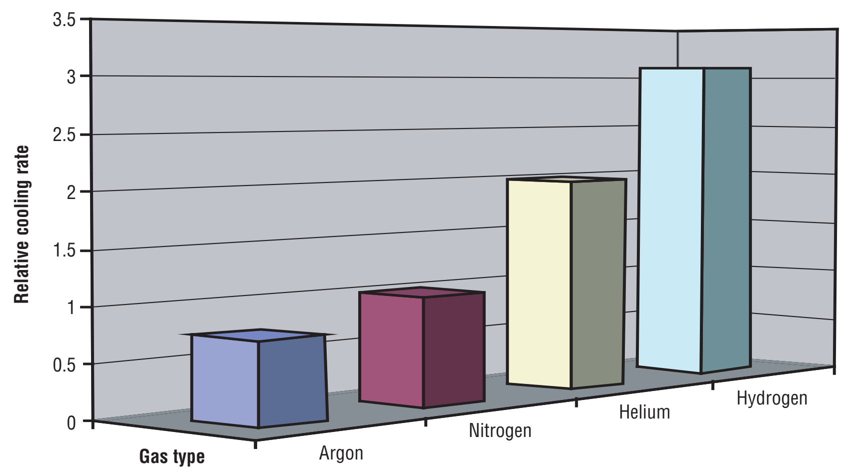

Fig. 3. Relative cooling rates for quench gases

which may be difficult to pump via some dry pumps with their internal clearances (leakage). The low viscosity of hydrogen makes this gas particularly difficult to pump. Oil-sealed pumps have no such difficulty with light and low-viscosity gases due to oil sealing of the pump mechanical clearances.

Argon reaches a high gas temperature during compression due to its high gamma and poor thermal conductivity. For dry pumps where the rotors are immersed in the gas and able to lose heat only through conduction, elevated rotor temperatures can risk closure of pump clearances unless a purge gas with a lower gamma (air, nitrogen, etc.) is used to reduce gas temperatures and improve thermal conductivity. Oil-sealed pumps may run hotter on argon, but generally do not have problems due to a more isothermal compression and oil wetting, which aids heat exchange.

Vacuum de-oiling processes

Many manufacturing processes use lubricants to aid the formation of complex stamped or formed powder metallurgy (PM) product shapes, which must be removed at later stages of manufacturing. Vacuum furnaces offer a clean, high-temperature environment to distil lubricants from product surfaces while preventing component oxidation and discoloration.

Products are heated to a temperature between 180 and 300°C (355 and 570°F) at pressures of ~0.01 mbar (~0.0075 torr), and the lubricant vapor is removed by the vacuum pumps. Typical systems use one or more stages of Roots-type mechanical boosters with oil-sealed or dry backing pumps. Oil condensed at the cold chamber wall and pumping line inlet baffle is collected for reuse, but further condensation occurs on compression within the vacuum pumping system. Mechanical boosters are unaffected by oil condensation and drain freely, but oil-sealed pumps can flood, thereby suffering lubricant dilution unless condensation in the pumps can be prevented. Traps should be used to intercept oil draining to the pump from either inlet or exhaust pipe work.

Dry pumps have the capability to pass condensates without the problems associated with oil-sealed pumps, but care must be taken to limit the flow of liquids to ensure hydraulic lock of the pumping mechanism is avoided on both process and start-up. The temperatures within dry pumps can also cause decomposition of condensed oil vapor, leading to mist and smoke emissions, which requires consideration of their flammability and environmental impact.

too much vacuum performance, either oil-wetted or dry as appropriate. For very large chamber systems, a single large system filter, such as a multibag type, may be the only practical proposition.

Maintenance tips

Consistent maintenance of all types of filters will give the highest rewards. While many heat-treating processes produce trace contaminants which are oily or corrosive, it should be noted that many fine metallurgical dusts can be reactive or even pyrophoric in the dry state on contact with air. Adequate safety procedures must be in place to deal with dry filter cleaning in such applications.

For metal mesh filters, this may typically include isolating and "inerting" the filter with nitrogen, removing the whole filter assembly to a protected location and performing controlled oxidation of the dry metal dusts in a safe manner. When fully oxidized and cooled, careful but thorough water washing may be the best way to clean the mesh element(s), but personnel should wear adequate protective equipment as reactive by-products may be liberated during cleaning. Dry the element thoroughly before reinstallation. Manufacturer's recommendations for servicing vacuum pumps should always be the first consideration to ensure long pump life, especially in harsher environments.

Dry pumps will usually benefit from a controlled shutdown regime at the end of the process, especially with harsh, dusty processes and those that generate corrosive contaminants, such as nitriding. The procedure should involve running on atmospheric air for 10 to 15 minutes, throttled to around 700 mbar (525 torr) inlet pressure to blow residual dust through the pump and to dry the pump out. Note that with an all-dry pump set, the exhaust silencer and exhaust line requires routine checking and cleaning of any accumulated process dust to avoid potential problems.

For oil-sealed pumps, a shut-down regime of 15 minutes running isolated on full gas ballast, or a small inlet purge should help clean the oil and drive out adsorbed and condensed moisture and corrosives.

Vacuum pump safety issues

Potential problems with pyrophoric dusts have been mentioned previously, but also consider any harmful, toxic or flammable risks associated with the gases being pumped. Such risks should be clearly identified by a risk assessment for the process, conducted and documented by the process operator.

Apart from any issues with toxic gases, the key concern is with any flammable gases liberated by the process, because these will be pumped though the vacuum system and exhaust lines and eventually out into the environment. Even though the location of the vacuum system may not be a designated hazardous area, flammable risks can still arise inside vacuum pumps, pipe work and exhaust line if flammable gases and vapors are pumped. Appropriate safeguards should be in place to prevent any possibility of a flammable gas mixture being created, which could be ignited by some malfunction or other occurrence (for example, friction, particle impact, static discharges, etc).

Note that for the vast majority of flammable gases, ignition cannot occur at pressures lower than 60 mbar (45 torr). Therefore, flammability issues usually arise only after that point in the vacuum system where the gas is compressed to above this pressure level. This certainly occurs within the backing pump and the exhaust line, and depending on process pressure, can occur in one or more stages of mechanical boosters.

The recommended approach is to ensure that any mixture of flammable gas with air always remains safely below the lower flammable limit (LFL) or lower explosive limit (LEL), so no ignition is possible even if a source of ignition occurs. Below-LFL operation is usually achieved by process gas dilution; that is, injecting nitrogen into the vacuum pump inlet or its purge or gas ballast ports. If the flammable gas concentration at the pump outlet is then below the LFL, further mixing with air in the pump exhaust line reduces the gas concentration further below the LFL and away from a potential hazard.

Some processes may operate with very high levels of flammable gas, such as hydrogen, and inert gas dilution is impractical or involves high cost. In these cases, operation above the upper flammable limit (UFL) or upper explosive limit (UEL) may be justified. The key requirement in this case is to prevent the hydrogen concentration from going below this level at any point in the vacuum system where the pressure exceeds 60 mbar, including exhaust lines through to any abatement system provided.

Air leaks into the chamber, piping, pumps and exhaust line must be prevented, and appropriate process control measures and procedures must be incorporated to ensure this is always achieved. Typical precautions include:

Vacuum brazing

Brazing under vacuum avoids oxidation of the joint during heating and eliminates trapped pockets of gas that impede full braze penetration. The final product is clean and the joint quality is consistent. Oil-sealed pumps and mechanical Roots-type boosters are used with vapor boosters or diffusion pumps to provide a vacuum environment of $<1 \times 10^{-3}$ mbar ($<7.5 \times 10^{-4}$ torr). The pumps receive small amounts of particulates together with solvents and residues from the braze paste, binder and stop-off used to prevent the braze from flowing where it is not required.

Problems occur when solvent-based binders are replaced by more environmentally friendly acrylic-based binders, which can polymerize in oil-sealed pumps to build hard deposits. Gas ballast can be applied to reduce problems, but the pump lubricant always adsorbs some of the pumped vapors and some polymerization is inevitable with these types of pumps. Polymerization in dry pumps can be reduced or avoided by configuring gas ballast or pump stage purges to hold the vapor partial pressures below the condensation point.

Optimizing the vacuum system

A completely optimized vacuum system is probably rare in the real world. However, there are a number of issues that should be considered if installing a new vacuum system or if improving an existing setup. Key to any successful vacuum system is an understanding of what you want the system to provide to the process in terms of vacuum performance and duty cycle. In addition, it is essential to ensure you are getting this performance and that any factors limiting the effectiveness of the system are clearly identified and addressed. There are several important points to consider.

Pump type

Is the pump type appropriate for the application? Considering primary (backing) vacuum pumps first, while rotary vane pumps are usually a low cost solution, they are not well-suited to metallurgical treatment processes where pumps may be subjected to process dust, which can result in rapid vane and stator erosion. General advice would be to use either oil-sealed rotary piston pumps or dry running pumps. The former have a lower installed cost, but require higher maintenance to retain vacuum performance as lubricant contamination and mechanical wear progresses. The latter have higher installed cost, but lower maintenance as the noncontacting mechanism without lubricants provides more stable long-term performance. Further, while dry pumps are well-suited for use in applications generating dry particulates or corrosive vapors, an oil-sealed pump mechanism may be preferred in applications where oils or tars are produced, provided that the oil is filtered and regularly replaced. Mechanical (Roots) vacuum boosters are often fitted as secondary pumps, and although the mechanism has an inherent ability to pass contaminants straight through into the backing pump, these will also require appropriate cleaning on really dirty processes.

Pump-down time

Is there sufficient backing pump capacity and proper use of mechanical booster pumps?

- Routine system leak checking
- Inert gas purging or a pre-evacuation cycle before starting the process to remove residual air
- Permanent sealing of any fixtures and fittings on the pump that may be opened to air
- Inert purge of the exhaust lines to prevent suck back of air when the pumps are stopped
- Full routine maintenance of all vacuum pump seals

To minimize the risk of air leakage into the vacuum pumps, it is also strongly recommended that only mechanical booster pumps with canned motors or oil-flooded shaft seals are used in these applications. Also, oil-sealed primary pumps should use inert oil box purging and a pressure feed seal-lubrication system, which is alarmed if the oil pressure is lost. For dry pumps with gas seals, these should be permanently plumbed to an inert gas supply and alarmed if the seal flow is lost.

The linear relationship between pump speed and chamber pump-down time implies that simply adding more pump capacity reduces pumping times, but the real world is not always so simple. The simple relationship should hold true where a target pressure is within the range of primary pumps alone (e.g., down to around 1.3 mbar, or 1 torr). However, the capacity of the mechanical booster(s) and the method used to drive them become important where the target pressure is significantly below this. Starting booster(s) at atmosphere using variable frequency (inverter) drives, bypass valving or hydrokinetic drives to control the differential pressure can add pumping speed (usually increasing as the inlet pressure drops) to make significant improvements to the pump-down time. Switching on a direct-drive booster at a higher pressure may also help, but issues of motor current (power) and thermal overload must be addressed including seeking expert advice.

The vacuum chamber

How much does the vacuum chamber leak, are there a minimum of fixtures and fittings inside and how clean do you keep the internals? Operation above 0.13 mbar (0.1 torr) will largely avoid issues with gas loads due to surface outgassing and typical chamber leakage. However, below this pressure, these two phenomena combine to supply an increasingly significant gas load to prevent or slow further reduction in chamber pressure.

A leak represents a constant gas load to the chamber, and the total chamber leak rate (sum of many small leaks) should be quantified (via a chamber pressure rise test clean, dry and empty) located (may require a leak detector) and dealt with as required. Surface outgassing represents a gas load that is directly proportional to chamber internal surface area and decays with time under vacuum. Surface outgassing is mostly water vapor absorbed onto chamber internal surfaces when open to atmosphere and desorbed when the system is repumped. To minimize this outgassing load, reduce the internal surface area and avoid construction materials having adsorbent surfaces. Ideally, use polished metal surfaces, remove porous, rusty and corroded materials, and minimize any exposed elastomers. If possible, keep the chamber closed and filled with desiccated air when not in use. Regularly clean out all dirt and dust (which provide extremely large surface areas for water to adsorb). Good housekeeping assists good vacuum!

Vacuum fore line

Is the vacuum fore line adequate for the operating pressure and gas flows required, can it be shortened/widened/straightened if necessary and is it routinely cleaned? As noted previously, systems that operate down to around 0.13 mbar (0.1 torr) usually are in the viscous-flow regime where fore line conductance (the ability of the pipe to pass gas flow) is related to both pressure and pipe geometry. However, we pass through the transitional-flow regime as pressure reduces, and eventually into the molecular flow regime at ~0.001 torr, where line conductance is independent of pressure and simply a function of line geometry. In practical terms, this means that for operating pressures below ~0.1 torr, systems should always aim to have fore lines as short and straight as possible and with a diameter at least the size of the vacuum pump inlet flange. Expert advice can always be used to identify any particular limitations for existing systems.

Another practical issue with geometry is the risk of accumulating dirt, dust, tars and oils in the fore line. Contamination with such materials is a fact of life in many systems, so fore line design should collect/trap such materials where they can be readily removed and safely disposed of. For example, minimize horizontal pipe runs, which accumulate sediment, and use "T" connections to force gas flow upward, while catching particles or liquids in the downward leg terminated with a removable flange (and drain valve if required) to facilitate cleaning. When backing diffusion pumps, a cold vertical section at the diffusion pump outlet flange assists in coalescing any forestreaming diffusion-pump oil, which can then drain back to the vapor pump rather than carry over to the backing pump. **IH**

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