



About the columnist . . .

Daniel H. Herring is president of The Herring Group Inc., Elmhurst, Ill., a business specializing in practical solutions to problems facing the heat treating and sintering industries (Web: www.heat-treat-doctor.com).

Dan Herring has spent more than 25 years working for furnace equipment manufacturers in a variety of roles, including senior management and new business development.



Now an independent entrepreneur, Dan lectures, writes, teaches, and consults. A frequent speaker at local, national, and international conferences, he is dedicated to advancing the state of the art in thermal processing. He has published more than 100 technical papers, written three books, and contributes to Heat Treating Progress and other industry publications.

Dan's credentials include his appointment as a research associate professor at the Thermal Processing Technology Center, Illinois Institute of Technology, Chicago. He also is active on several HTS Committees.

Generic and practical information presented here is not intended to replace or supplement federal, state, and local codes, government standards, insurance requirements, company policies and procedures, or common sense. In addition, all equipment manufacturers' instructions and operating and maintenance manuals should always be thoroughly read and followed. Further, personnel training should be provided unequivocally to everyone who will be associated with operating such equipment.

The Why, When, and How of Leak Checking a Vacuum Furnace

The use of vacuum furnaces is becoming more popular since they offer a number of processing advantages, especially in critical applications, provided they are properly maintained and kept in good operating condition.

A common problem experienced by almost every user is that, over time, leaks develop that are both damaging to product quality and to furnace internal components. In extreme cases, the problem is obvious: the furnace will not pump down and/or the hot zone shows signs of oxidation. Small leaks, which are more common, often go undetected because the pumping system can more than offset any air infiltration. However, even small leaks can cause continuous and sometimes catastrophic damage. Thus, leak checking should become a routine part of any good vacuum furnace maintenance program.

What is a vacuum leak?

A leak is an opening such as a crack or hole that allows a substance to be admitted to or to escape from a confined space. A vacuum system leak allows air to be admitted into the vacuum vessel. Suspect areas on vacuum furnaces include threaded and brazed joints, fittings that have been improperly sealed or installed, and damaged (cut, worn, melted, or dirty) O-ring seals, especially around doors. Components that ro-

tate or reciprocate are other prime leak sites.

What is leak testing?

In general, leak testing involves measuring the amount of leakage (vacuum degradation) over time in the vacuum environment. The "leak-up" rate of a vacuum system is a function of actual (real), internal (through), and virtual (outgassing-related) leaks in the chamber or vacuum system (see table).

Real vs. virtual: Outgassing is the release of low-vapor-pressure contaminants present in the vacuum system. Sources of these contaminants include volatile liquids, dirt, grease, oxides, water vapor, and gases adsorbed on or in surfaces and pores. These contaminants outgas at different rates at different vacuum levels and temperatures. To minimize outgassing, cleanliness is essential, especially of workpieces, part baskets, and fixtures introduced into the furnace. It's also a good idea to limit the time that an open furnace is exposed to the factory environment.

Outgassing is often made obvious during the process cycle by a large "spike" or rise in pressure during heating. It also can be detected by comparing successive leak-rate values after long pump-down cycles. If the leak rate improves (decreases) with each successive pump down, then outgassing is suspected. If the rate re-

Potential sources of vacuum system leaks

Type	Potential Source
Actual (real) leaks	Compression seals Welds and brazed joints Shaft seals and bellows on valves Flexible connectors in piping Threaded joints on vacuum gauge and plugs, for example. Static gasket seals on sight ports, feedthroughs, manifolds, and air-operated cylinder glands.
Internal (through) leaks	Process gas delivery system Vent gas exit system Seals between adjacent internal volumes Improper gas ballast, gas purge, partial pressure control systems, and backfill valves.
Virtual (outgassing) leaks	Residual solvents Residual liquids (water, cooling fluids) in blind holes and restricted passageways. Pockets of trapped gases or liquids High-vapor-pressure materials Porous materials and condensed by-products from processes already run.

THE HEAT TREAT DOCTOR *Continued*

mains essentially the same, then an actual (real) leak may be considered the primary cause.

In most cases, both leaks and outgassing are present simultaneously. Bear in mind that real leaks are linear and outgassing is not. Thus, for example, a 4 hour leak-rate test, with measurements taken at 30 minute intervals, will generate a linear rate. The outgassing curve will deviate from linearity, yielding the true leak-up rate.

There are many methods used to measure leak rate and various types of leak-tracing agents. (The choice of tracing agent usually depends on the measurement method used.) Selection of the best method for a specific application requires consideration of economics, accuracy, tolerance to environmental conditions, leak-rate requirements, and equipment limitations.

Why do I need to leak test?

Everything leaks. And although a leak may be extremely small, it still may pose a problem. Leaks can be inherent in the material, created during

the manufacturing process, be introduced during maintenance or repair, or occur over time due to wear, fatigue, and stress. The possible source of a leak may be revealed by the answer to the question, "What was the last area worked on or modified?"

However, the question that really matters is this: "Can the system tolerate the leak?" In other words, can the process and equipment survive and be unaffected by the leak? The answer is almost always, "No."

How do I run a leak test?

A furnace leak-up rate test does not locate the leak, it only quantifies it. The furnace must be clean, cold, empty, and outgassed to obtain a true leak-up rate value. If it isn't, which is often the case, a conditioning cycle should be run.

How to condition: A conditioning cycle — bake out, burnout, cleanup — involves heating the equipment to 50–100°F (30–55°C) higher than the furnace's normal operating temperature (but lower than its maximum operating temperature), soaking at temperature

for 2 to 4 hours, and then vacuum cooling, usually overnight. Fixtures, baskets, parts, and work thermocouples are removed from the furnace. The first 2 hours of the cycle should be run in a partial pressure of 1000 microns (1.3 mbar) of nitrogen. (1 micron = 1 micrometer of mercury.) This is to ensure that heat transfer takes place to the normally colder areas of the hot zone and furnace interior, since vacuum alone is an excellent heat-loss insulator. The balance of the cycle is run in the lowest obtainable vacuum, which will facilitate vaporization of unwanted process by-products.

If it is not possible to perform a conditioning, the alternative is to pump down to 1 micron (0.0013 mbar), or to the lowest attainable value of the pumping system (if it is above 1 micron), followed by an inert gas back-fill to approximately 10 in. Hg (255 torr or 340 mbar). This is followed by another 1 micron pump down and, finally, by at least 1 hour of additional pumping.

Leak testing: To perform a leak-up



Giving nature a helping hand

Ammonia neutralizing systems

Visit us at ASM Heat Treat, Stand 607

	Nitrex Inc. USA michigan@nitrex.com +517.676.6370	Nitrex Metal Inc. Canada nitrex@nitrex.com +514.335.7191	Nitrex Metal GmbH Europe nitrexgmbh@nitrex.com +41.26.493.3254	www.nitrex.com
---	--	---	---	-----------------------

THE HEAT TREAT DOCTOR *Continued*

rate check, pump the furnace down to ultimate pressure with the heat turned off and the furnace cold, at 70°F (20°C) or below. Record the vacuum level and the time. Next, isolate the furnace from the pumping system by closing the vacuum valve(s) to the chamber. Allow at least an hour to obtain an accurate leak-up rate. (This step is often shortened to just 5–30 minutes, but this is poor practice and should be avoided.) Record the time and vacuum level. The leak-up rate is the difference in the vacuum levels divided by the elapsed time and is expressed in microns/h (mbar/h). For most vacuum applications, a leak-up rate above 10 microns/h (0.013 mbar/h) in the heating chamber is unacceptable — the leaks must be found and corrected. Note that leak-up rates between 50 and 100 microns/h (0.067 and 0.13 mbar/h) are not uncommon for oil-quench tanks.

Test methods: There are three general categories of leak detection procedures:

- Effect-of-leak types: pressure decay

(differential, increase), vacuum decay

- Amount-of-leak types: mass flow (inside/out, outside/in, accumulation), carrier gas, residual gas analysis (RGA)

- Traditional types: immersion, sniffing

The most common procedures for detecting leaks in vacuum furnaces are the solvent and mass spectrometer procedures. Descriptions follow.

Solvent: simple, effective

The use of a solvent test method is simple but effective for locating intermediate sized leaks, normally in the 400 to 1000 micron (0.53 to 1.33 mbar) range. A solvent such as acetone (preferred) or alcohol is carefully sprayed on a suspect area and any change in vacuum level inside the chamber, based on the vacuum gauge reading, is noted. Allow enough time (up to 20 seconds) for a pressure increase to occur. This procedure is more sensitive at lower pressures. Be sure to observe all required safety precautions when using hazardous solvents, including proper ventilation and spill

containment. Remember that these solvents will remove paint.

If a leak is located, a temporary sealant such as Glyptal red alkyd lacquer (Glyptal Inc., Chelsea, Mass., www.glyptal.com), Kinseal clear vacuum sealant (Kinney Vacuum Div., Tuthill Corp., Springfield, Mo., www.kinneyvacuum.com), or vacuum seal putty can be used to patch the area and allow leak checking to continue. A common mistake is to neglect to permanently fix the problem after testing is completed.

Spectrometer: accurate

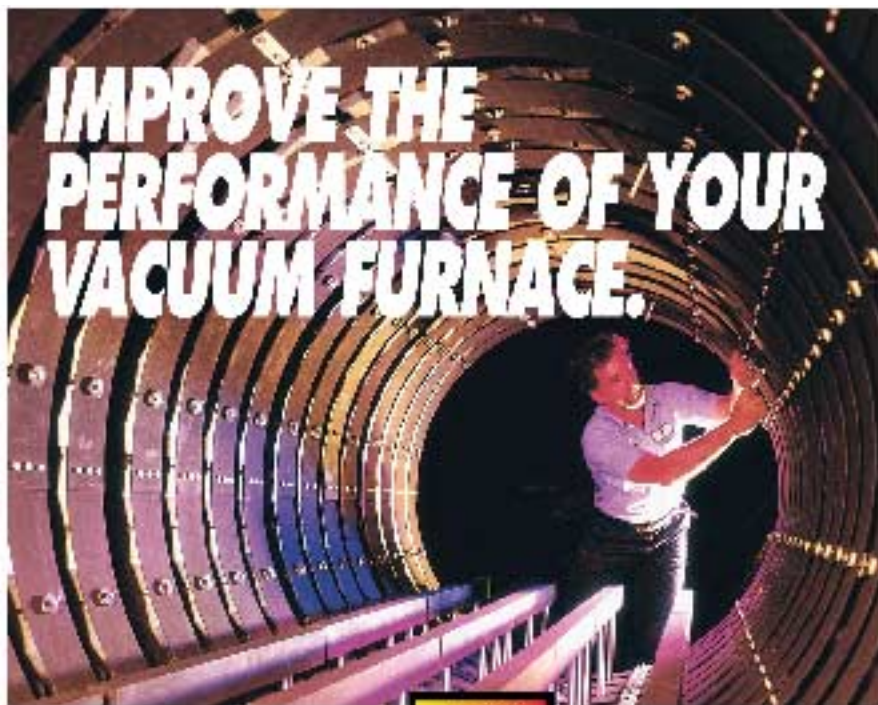
A helium mass spectrometer is a highly accurate instrument for locating very small leaks or leaks in hard-to-reach areas. In some instances, it is necessary to “bag” or isolate a specific area on the furnace and inject helium into the contained space. The dynamic, nondestructive technique is often used to check parts that have moving seals or those that may leak only during the transition from pressure to vacuum (or vice versa).

A mass spectrometer can detect extremely small amounts of helium (or another tracer gas). When the gas enters the spectrometer tube it is ionized and accelerated. These high-speed charged particles are then exposed to a magnetic field perpendicular to their direction of motion. What results is a force perpendicular to both the velocity vector and magnetic field (see figure, top).

This force causes the particles to follow a curved path, the radius of which depends on the mass of the particle. Thus, allowing separation of the particle stream into different ions. A properly positioned collector plate (ion detector) enables the concentration of any gas to be very accurately measured (see figure, bottom). Every electron given up by the collector plate equates to the presence of one helium ion. The amount of helium collected is then converted to a leak rate.

Helium is the tracer gas of choice because it is inert, nontoxic, relatively inexpensive (in small quantities), and not easily absorbed. Helium also easily flows through small leaks and has only a trace presence in air (5 ppm).

Tips: Vacuum pumps are very efficient at pumping large atoms and molecules (water and hydrocarbon vapors, oxygen, and carbon dioxide,



- VFS' Premium Aftermarket Support includes:
- Rebuilding and replacement of hot zones
 - Upgrading of pumps and quench systems
 - Rebuilding of control systems
 - Large spare parts inventory
 - Fast, efficient field service



Call VFS today.
Tel: 215-723-8125
www.vfscorp.com
E-mail: sales@vfscorp.com

Designed To Meet The Highest Standards. Yours.

for example), but inefficient at pumping helium. This allows a greater amount of helium to reach the mass spectrometer for measurement.

Mass spectrometer leak testing requires that the unit be exposed to helium leaking for only 3 to 4 seconds. However, as with the solvent test method, a dwell or lag time between test areas is needed to prevent false readings.

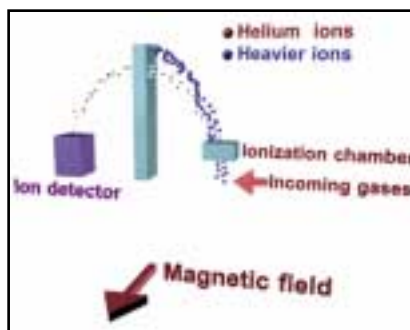
Backfill lines . . . and more

There is no acceptable minimum leak size. Since the vacuum chamber is not capable of determining how many leaks are present or their sizes, the effect is cumulative. For example, five small leaks can be more harmful than one large leak if the sum of their leak rates exceeds that of the large leak.

When leak testing a vacuum furnace, do not forget to check the gas backfill lines (including all fittings) from the gas supply to the furnace. It's good practice to install vacuum-tight shutoff valves near the source (just inside the building if using a cryogenic system located outside) and at the equipment. These lines are often pressurized and "soap tested" when first installed. However, that does not guarantee that they do not leak. The gas inside backfill lines often travels at near supersonic velocities and a pinhole leak in a line will draw in air via a venturi action. Always perform leak-up tests first with the backfill lines closed and then with them open (up to the shutoff valve near the source) to confirm that the lines are not leaking. The backfill lines may be evacuated via the vacuum furnace back to the source and the line sets may be vacuum checked utilizing a helium leak detector.

In addition, clean and accurate vacuum measuring devices are essential for obtaining a meaningful value of leak-up rate. The need for periodic checks and annual calibrations of all vacuum instruments against known standards cannot be overemphasized.

Finally, there are differences between North American and European leak-up rate specifications. In North America, the specs usually involve a fixed leak-up rate and do not take into account the chamber volume. (OEM specifications for new equipment vary from 2 to 10 microns/h [0.003 to 0.013 mbar/h]) In Europe, the leak-up rate



Principle of the mass spectrometer. Top: Helium gas is ionized and accelerated. The ions are then exposed to a magnetic field perpendicular to their direction of motion. A force perpendicular to both the velocity and magnetic field vectors results. Bottom: This force causes the particles to follow a curved path, the radius of which depends on the mass of the particle. This allows the particle stream to be separated into different ions. An ion detector measures the helium concentration, which is then converted to a leak rate. Courtesy Future Technologies Inc., Bay City, Mich. (www.futuretechnologies.com).

factors in chamber volume and is expressed in units such as mbar-L/s (millibar-liter per second).

Good (leak) hunting! HTP

Selected references

- *Practical Vacuum Techniques*, by W.F. Brunner and T.H. Batzer: Krieger, 1974.
- *A User's Guide to Vacuum Technology*, by J.F. O'Hanlon: John Wiley & Sons, 1980.
- *High Vacuum Technology: A Practical Guide*, by Marsbed H. Hablanian: Marcel Dekker Inc., 1990.
- "Basic Vacuum Practice," 3rd Ed.: Varian Vacuum Products Training Dept., Varian Assoc., 1992.
- "Leak Detection Applications & Techniques": training course, Varian Inc. Vacuum Technologies.

Acknowledgements: Important contributions to this column were made by James E. Grann, Grann's Refrigeration, Rockton, Ill., and Diego Calvo, Future Technologies Inc., Bay City, Mich., as well as various vacuum furnace OEMs.

How useful did you find the information presented in this column?
 Very useful, **Circle 277**
 Of general interest, **Circle 278**
 Not useful, **Circle 279**



Brains & Brawn

*New Axiovert 200L
 Performance Driven Microscope*

Stable and flexible, Axiovert 200L inverted metallurgical microscope is ready to handle your every command. From analyzing microstructures, measuring particles and grain size to handling large and bulky specimens.

News:

- Innovative EC (Enhance Contrast) optics generate superb images with highest contrast and resolution.
- Exclusive contrasting techniques C-DIC and C-TOPO will increase your performance and productivity.
- Manual or motorized systems combined with digital imaging for flexibility, comfort, optimum performance, and ease of use.

With fine-tuned, perfectly matched components, new super-b optics, 8 documentation ports and its modular design, Axiovert 200L is ready to meet your every need now and in the future.

Smart Microscopy Made Easy

Carl Zeiss
 Microimaging, Inc.
 Ithaca, NY 14850
 1-800-442-4020
micro@zeiss.com
zeiss.com/micro



We make it visible.