# PREVENTING DISCOLORATION IN VACUUM HEAT TREATMENT: GUIDELINES FOR CONTAMINATION CONTROL

## Contamination in vacuum furnace heat treatment can have a significant impact on the final product's quality, so operators must be aware of the potential sources of contamination, the chemical reactions involved, and the color identification for discoloration.

**By JANUSZ KOWALEWSKI**

Discoloration in vacuum heat treatment can indicate contamination, leading to potential product quality issues and process inefficiencies. Even minor contamination can compromise part integrity, causing oxidation, residue buildup, and surface defects. This article explores the key sources of contamination in vacuum furnaces, including outgassing, backstreaming, leaks, and particle generation. Learn how to identify, evaluate, and prevent contamination using best practices for furnace maintenance, clean-up cycles, and process optimization. From understanding oxidation limits to identifying metal sublimation effects, this article provides actionable insights for heat treaters looking to improve quality control. By implementing proper contamination prevention techniques, manufacturers can enhance process reliability, extend furnace life, and ensure high-quality, defect-free heat-treated parts.

### INTRODUCTION

Vacuum furnace heat treatment is critical in many industries, including aerospace, medical, nuclear, additive manufacturing, automotive, and commercial heat treatment. It involves heating materials in a vacuum with optional specialized gas environments to achieve specific mechanical, physical, and metallurgical properties. However, contamination may compromise the process, which can negatively affect the final product's quality, reliability, and performance. Preventing contamination is crucial for ensuring the quality and reliability of heat-treated materials and the repeatability of the vacuum furnace functionality. This article provides an overview of several sources of contamination that vacuum furnaces are exposed to. The following are several contamination-related issues — often identified by changes in color within the visual spectrum — along with strategies to understand and prevent them.

#### *Contamination Fundamentals*

Vacuum furnace contamination is a physical or chemical occurrence during heat-treatment operations. Contamination can occur through leaks (real or virtual), diffusion pump backstreaming, outgassing substances from parts or fixture surfaces and cavities, process gas impurities, alloy sublimation, and air permeation through seals and washers. It's important to consider how contamination affects the hot zone's physical condition, the integrity of the load surface, and process duration during heat-treatment processes.

It's also important to consider the evaporation of alloys during high temperatures and vacuum conditions. When temperatures and vacuum levels are elevated, alloy evaporation can speed up and cause alterations in the metal's chemical composition, color changes, and decreased or uneven hardness. This excessive evaporation can cause hot-zone components such as ceramic insulators, power feedthroughs, insulation, and heating elements to have a shorter lifespan. Additionally, it can reduce the accuracy and lifespan of gauges.

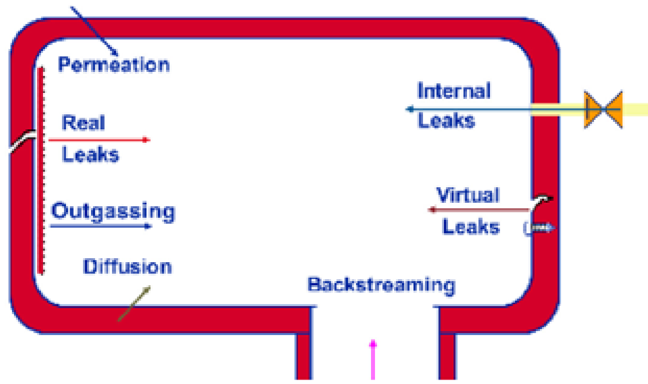


**Figure 1: Possible sources of leaks in a vacuum furnace. [1]**

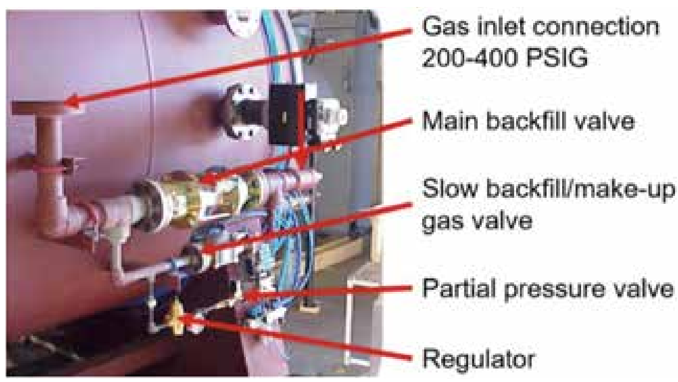


**Figure 2: This picture illustrates at least 16 possible sources of leaks in gas partial pressure and cooling systems if not maintained correctly. (Courtesy: Ipsen)**

Locating vacuum leaks in furnaces can be a challenging and time-consuming task for service engineers, as there are numerous potential sources for leaks (see Figures 1 and 2)

### POSSIBLE SOURCES OF CONTAMINATION

#### *Air Contamination*

The primary source of contamination from air is leakage. Even a tiny amount of air leaking into the furnace can cause significant contamination, as the air contains oxygen, nitrogen, and other gases that can react with the heat-treating processes; e.g., argon purity problems caused by air leaks present as yellow/straw, dark yellow, or blue. At the same time, nitrogen air leaks appear as blue/dark blue or black. Leaks can occur at various points in the vacuum furnace system, including the chamber, seals, valves, inert gas supply service/lines, and leaking pumps.

Other sources of contamination from air include inadequate purging of the vacuum chamber and incomplete evacuation of residual gases. These sources can lead to surface contamination or the diffusion of unwanted gases into the material.

### *Process or Cooling Gas Contamination*

Cooling and partial pressure gases are sources of discoloration in the vacuum furnace. The requirement for vacuum furnace is high gas purity. The low purity may come from the gas supplier (very unlikely), or more likely from leaks in the piping, or buffer tank. The internal condition of the buffer tank is important to be free from rust, grease, paint chips, or any other contaminants. It is recommended to purge the buffer tank before first used.

### *Water Contamination*

Small water leaks can be difficult to troubleshoot, whereas a catastrophic leak is obvious and most likely to damage the hot zone beyond repair. If the small water leak is constant, it is most likely heat induced; you will not be able to pump down below a certain pressure, and your leak up rate will be affected. The furnace will run, but the parts and hot zone will become contaminated with every run.

The most common potential water leak locations are the heat exchanger and internal welds. In the heat exchanger, there are many brazed joints that see both hot and cold environments.

This causes the solder connections to become porous. Internal cracks happen and may eventually leak over time.

Very small leaks will open during the cooling process or quenching, allowing water to vaporize into the hot furnace. The best way to identify the leak is to standardize testing procedures.

Water contamination in vacuum furnaces may also come from residual moisture on the parts surface or cavities. When the parts are heated, the moisture evaporates and reacts with the metal, causing surface defects, discoloration, or even changes in mechanical properties. Other sources of water contamination are leaks on water cooled power feedthroughs and, although less likely, water-cooled convection fan motor or internal vessel welds such as thermocouple ports as shown in Figure 7. Typically, water leaks produce dark green/purple or black discoloration. In standardizing load material, the discoloration is more constant. Note: 304 SS reacts green and can be used as an indicator.

One way to identify sources of contamination is to repeat the production cycle that resulted in discolored parts but use vacuum cooling instead of gas cooling to prevent any gas from entering the furnace while it is hot. If the pieces come out cleaner with vacuum cooling, it may suggest contamination from impure gas or a water leak. To confirm, proceed with the next step of the process to determine if there is a water or air leak. Run the same cycle with a maximum soak temperature of 1,000°F and use force cool. If the parts are clean, the gas system can be ruled out as the cause of contamination, and the water leak is the most likely culprit. It's worth noting that low-temperature cycles may not clean up as well as high-temperature cycles. If the material processed at a lower temperature with the same gas produces cleaner parts than at high temperatures, a heat-induced water leak is usually the cause.

### *Material Contamination – Pre-Heating Operation*

Inadequate cleaning or preparation of the material before introducing it into the vacuum furnace leads to contamination from the materials in heat treatment. The materials may retain residual oils, greases, and other contaminants, which can vaporize and cause surface defects or severe damage during the heat treatment. Some questions to ask include:

» Before heat treatment, has the fixture undergone sandblasting?

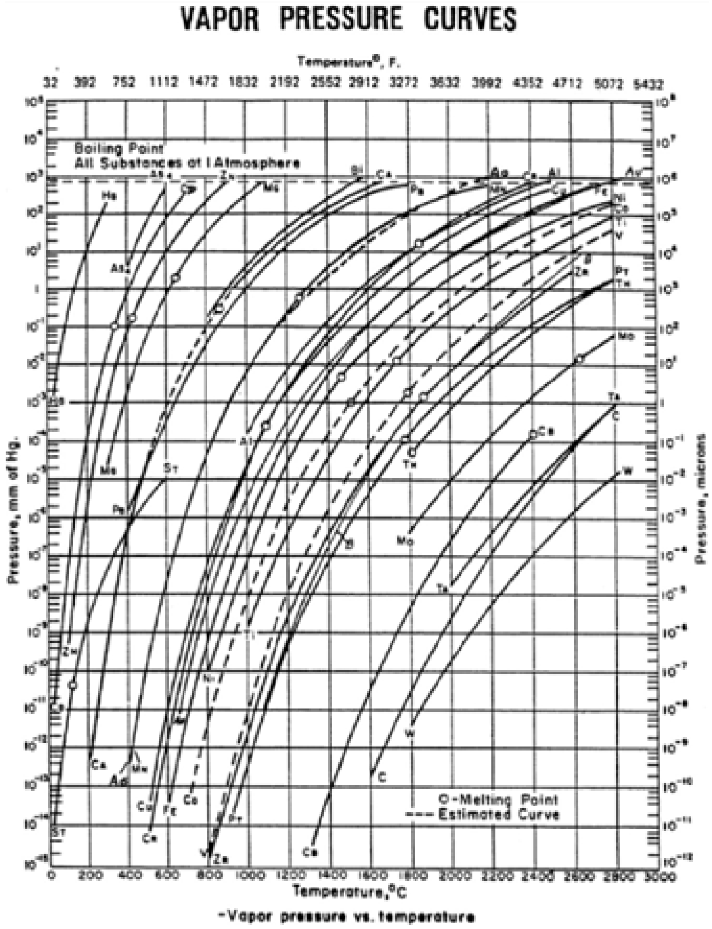


Figure 3: Vapor pressure curves for various elements. [2]

Figure 4: Zinc oxide dust on furnace hot zone shell. (Courtesy: Ipsen)

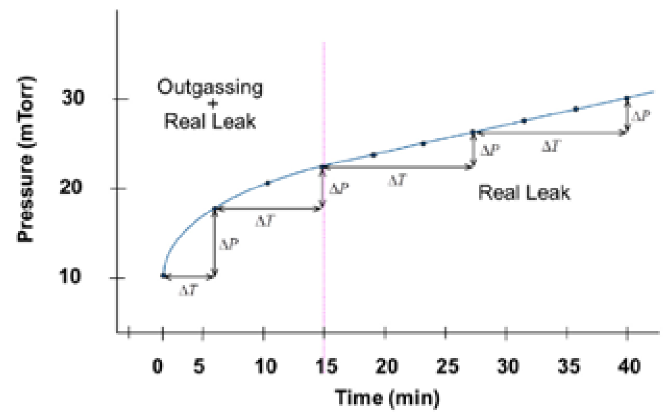


Figure 5: This graph shows the actual vacuum leak and outgassing behavior. [3]

Figure 6: Sample of stainless-steel parts contaminated by gas from the rusty inside of a buffer tank. (Courtesy: Ipsen)

Figure 7: Left – internal vessel weld. Right – Graphite hot zone exhibits significant water damage caused by a heat exchanger leak, resulting in prominent forest green discoloration. (Courtesy: Ipsen)

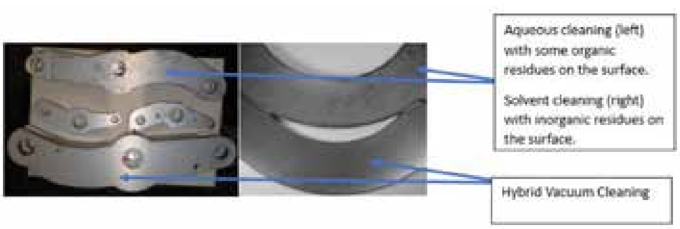


Figure 8: This picture shows the difference between only aqueous a hydrocarbon and hybrid cleaning results. (Courtesy: HEMO)

Figure 9: Discoloration caused by copper material during the brazing process. (Courtesy: Ipsen)

If so, what type of shot material was used, and have any other materials been previously run through the sandblasting system that could lead to cross-contamination?

» Is the shop fork truck transporting items that could contaminate or damage the fixtures?

» Could contaminants from the upstream parts washers affect the parts before entering the furnace?

Contamination of parts and hot zones can occur between 200°C and 260°C (400°F -500°F), as well as during the cooling phase. At higher temperatures (600-800°C and above), contaminants may evaporate from parts, contaminating the hot zone insulation or fixtures. If boron is present in the cooling fluid during machining before heat treatment, it can lead to soft spots and slight discoloration on the parts after low-pressure carburizing.

Properly cleaning parts and fixtures is essential in preventing discoloration, spats, softness issues, contamination, and unnecessary extension of the chamber evacuation time. All organic (non-polar) contaminants should be cleaned with solvents (hydrocarbons, modified alcohols, or a mixture of both). For cleaning all inorganics (polar) contaminants such as salts, soaps, emulsions, coolants, polishing pastes, protective paints, dirty hand marks, dust, scale, rust, grinding, abrasive and blasting residues, and even chips themselves, aqueous solutions will work.

All state-of-the-art cleaning machines are hermetically closed-safe systems, using vacuum technology for cleaning and drying, and distillation with limited sewage disposal, and emission-free. While solvents effectively remove grease and oils (degreasing) but never salts, organic contaminants will always remain if you use only aqueous cleaning media. Therefore, if you do not precisely know all impurities on the surface of your parts before HT (commercial heat treater), the best cleaning technology will be a mixture of both, called a hybrid.

### *Material Contamination – Oil, Grease, and Filler*

The color of the vacuum furnace depends on several factors, including the brazing material used, partial pressure, and temperature. Typically, a reddish/copper color is seen during vacuum copper brazing processes, while a silver color indicates a clean furnace without leaks when using nickel brazing in a high vacuum.

During the investigation, good questions to ask are:

» Which types of O-ring greases, oil valves, and cooling motor fan grease are used?

» Do the stop-off paint or fiber materials used for part separation contain boron nitrate?

During vacuum processes, parts, fixtures, and metal parts can stick together or become contaminated due to metal-to-metal transfer. This process is known as diffusion and requires specific conditions to occur:

» Metals that are soluble in each other.

» High temperatures.

» Low furnace pressure (high vacuum).

» Some contact pressure (which may be the weight of the part itself).

The exact condition is a function of all of these and needs to be analyzed on a case-by-case basis. This can be avoided by keeping the parts separated with a proper metal using a ceramic as outlined in prevention of eutectics or utilization of partial pressure to negate sticking.

### *Color Identification*

Contamination often appears in different colors depending on its type and source. For example, gray or black discoloration may suggest carbon contamination, while green or blue hues can indicate the presence of copper. Recognizing these color variations is crucial, as they help pinpoint the source of contamination and enable customers to take the necessary steps to prevent future occurrences.

Contamination layers less than 0.1 µm are transparent, and human eyes cannot distinguish the color correctly. Customers can identify discoloration based on the thickness of the contamination layer in the heating chamber or the part. Tool steels are less sensitive and can be heat-treated under a low vacuum level. Stainless steel and super alloys are susceptible to even the slightest traces of oxygen in the vacuum system.

Testing vacuum levels on various materials produced the following results: A standard furnace (not equipped with high vacuum) can produce bright D2 and M2 parts, although discoloration may occur if the furnace is contaminated. In contrast, a high-vacuum furnace consistently delivers bright results across all tested materials once the 10- Torr range is achieved. No burnout is required before heat treatment, and high-vacuum cycles evacuate more quickly, resulting in shorter overall cycle times.

Below is a general guideline during vacuum heat treatment to avoid contamination due to improper vacuum level(s).

### *Air Contamination*

Observing specific colors during the heat-treatment process helps identify the presence of air leaks. For argon purity problems resulting from air leaks, one may see yellow/straw, dark yellow, or blue colors. Conversely, air leaks involving nitrogen manifest as blue/dark blue or black colors. These distinct colors act as visual indicators, enabling operators to promptly identify and address the source of the leaks, thus detecting potential air contamination.

### *Water Contamination*

Water contamination is a common issue in vacuum furnace heat treatment, as it can lead to the formation of undesirable colors on the surface of the treated parts. The presence of water in the vacuum furnace can form various oxides, including iron oxide and chromium oxide (silver and green), which can cause the sections to become discolored. These oxides can also lead to reduced corrosion resistance and other adverse effects on the properties of the treated parts. Following a water leak, iron compounds FeO and Fe3O4 are identified by their respective dark brown and black hues, whereas Fe2O3 exhibits a red shade. Commonly, water leaks result in the appearance of brown (small leaks) or black stains (severe or prolonged leaks). The discoloration remains relatively consistent within the same batch of material.

### *Material Contamination*

Outgassing is a phenomenon that occurs in vacuum furnace heat treatment where volatile substances are released or evaporated from materials being heated in a vacuum environment. These substances can include residual gases, water vapor, solvents, lubricants, and other volatile components present in the material. Outgassing can have significant implications for the heat-treatment process and the quality of the treated materials.

### *Contamination Prevention*

To prevent contamination in vacuum furnace heat treatment, operators must minimize all potential sources of contamination. This includes:

### *Air Contamination*

Use the following steps to prevent air contamination:

**Proper Sealing and Vacuum System Maintenance:** Ensure the vacuum furnace and its components are properly sealed to prevent air ingress. Regularly inspect and maintain the seals, gaskets, valves, and other details to ensure their effectiveness. Conduct routine maintenance and testing of the vacuum system, including checking for leaks and verifying the efficiency of pumps and valves. Address any issues promptly to maintain a high-quality vacuum environment.

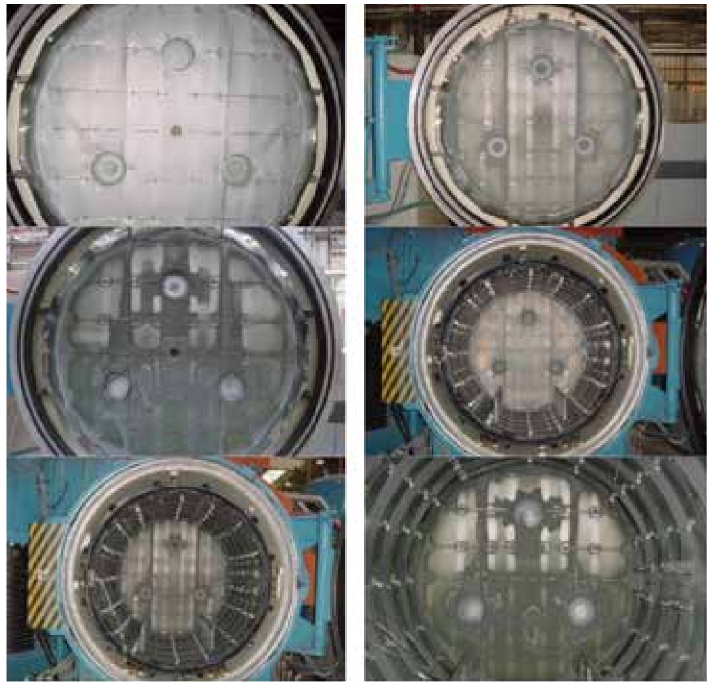

**Figure 10: Example of discoloration of the hot zone with passing time: The oxidation process in the metal hot zone takes a long time, and the first thing seen in the chamber will be intermediate colors. Later, the color of molybdenum changes from green to dark brown (MoO3 light green, MoO2 – dark brown).**

**Purging and Backfilling with Inert Gas:** Purge the furnace chamber with an inert gas, such as argon or nitrogen, before starting the heat-treatment process. This purging displaces residual air and helps create a clean, oxygen-free environment. Backfill the furnace chamber with the same inert gas after purging to maintain the desired atmosphere during the heat treatment. This prevents air from re-entering the chamber and contaminating the materials.

**Monitoring and Control:** Install pressure sensors and leak detectors in the vacuum system to continuously monitor the chamber pressure and detect potential leaks. Regularly calibrate and maintain these monitoring devices for accurate readings. Use an oxygen analyzer to measure the oxygen content in the backfill system and verify that it remains within the desired low levels. Implement appropriate process controls to ensure consistent and reliable vacuum conditions throughout the heat treatment.

### *Water Contamination*

Preventing water contamination in vacuum furnace heat treatment is crucial to maintaining the quality and integrity of the treated materials. Follow these steps to avoid water contamination:

**Thorough Drying of Materials:** Before placing materials in the vacuum furnace, thoroughly dry them to eliminate any moisture content.

**Effective Vacuum System Design and Maintenance:** Ensure the vacuum system is adequately equipped with moisture traps, condensers, and other components to remove and collect any moisture that may be present in the chamber. Regularly inspect and maintain the vacuum system, including checking for leaks, to prevent the entry of moisture-laden air.

**Controlled Heating Rates:** Implement controlled and gradual heating rates during the heat-treatment process. Doing so allows sufficient time for any residual moisture to evaporate without causing excessive water vapor accumulation in the furnace chamber.

**Vacuum Pumping and Purging:** Use efficient vacuum pumping techniques to evacuate moisture and other gases from the furnace chamber before the heat treatment begins. Employ purging with dry and inert gases, such as argon or nitrogen, to displace any remaining moisture and create a dry atmosphere within the chamber.

**Monitoring and Control:** Install moisture sensors or dew point analyzers to monitor and control the moisture levels within the backfill system during the heat treatment process. Regularly calibrate and maintain these monitoring devices to ensure accurate readings.

**Proper Workspace Environment:** Maintain a clean and dry working environment around the vacuum furnace to minimize the potential introduction of water sources that could contaminate the process.

### *Material Contamination*

Establishing measures that effectively avert material contamination during vacuum furnace heat treatment is crucial to safeguarding the integrity and quality of treated products. To achieve this objective, one can implement the following actions:

**Material Selection and Certification:** Choose materials specifically designed and certified for vacuum heat-treatment processes. Consult material suppliers or manufacturers to ensure the materials are suitable for the intended application and free from contaminants that could cause issues during the heat treatment.

**Proper Material Handling and Storage:** Implement appropriate handling procedures to prevent the introduction of contaminants onto the material's surface. Use clean gloves, tools, and equipment during material handling to avoid contact with substances that could contaminate the materials. Store materials in controlled environments, i.e., clean and dry storage areas, to minimize exposure to potential contaminants.

**Controlled Furnace Atmosphere:** Establish and maintain a controlled atmosphere within the vacuum furnace. This may involve purging the furnace chamber with inert gases, such as argon or nitrogen, to displace residual air and minimize the potential for contamination. Regularly monitor and control the gas composition and purity within the furnace to maintain the desired atmosphere throughout the heat-treatment process.

**Inspection and Quality Control:** Implement rigorous inspection and quality control procedures to detect material contamination before, during, and after heat treatment. Use non-destructive testing techniques, visual inspections, and analytical methods to identify any surface defects, contamination, or irregularities.

**Operator Training and Awareness:** Provide comprehensive training to operators on proper handling, cleaning, and packaging procedures to minimize the risk of material contamination. Foster a culture of awareness and attention to detail among operators to ensure they are vigilant in identifying and preventing contamination.

## CLEAN-UP CYCLE PROCEDURE [4]

Use the following steps to perform a clean-up cycle in your vacuum furnace, ensuring it is properly outgassed and vacuum-tight before processing critical loads.

**CAUTION –** Make sure all foreign materials are out of the furnace. Close the furnace and pump down to a high vacuum. Perform a leak check using a helium mass spectrometer or suitable rough checking means.

**CAUTION –** Do not proceed with the clean-up cycle until all gross leaks have been repaired. Heat the furnace to 100°F (56°C) above your normal continuous operating temperature (refer to manual for details) and hold for at least 2 hours using partial pressure of 1,000 microns, then 2 more hours under a high vacuum condition but not less than 2,100°F.

**CAUTION –** Do not exceed the furnace's maximum continuous operating temperature. Turn off the element power and allow to vacuum-cool to 150°F (66°C) or less. Allow the furnace to cool overnight if possible.

After the clean-up cycle, immediately check the furnace leak rate by closing the vacuum valves and timing the rate of rise in vacuum level. A furnace is considered vacuum tight and outgassed when the leak rate averages 5 microns (0.665 PA) per hour, or less, during a 4-hour period.

If the leak rate is excessive, after thorough cleaning and outgassing, check for vacuum leaks using a helium mass spectrometer. (A fitting to connect the mass spectrometer to the furnace is provided at the roughing pump inlet connection.)

If a mass spectrometer is not available, a rough check can be made by evacuating the chamber and spraying the suspected fittings and joints with acetone and noting any changes in system pressure on the vacuum gauge. A repeatable change in the indicated pressure indicates a leak in the effective 1,000- to 5,000-micron range. If the range cannot be achieved, backfill positive pressure and snoop for significant leaks.

***Regular maintenance and inspection are necessary to ensure the vacuum furnace's integrity and prevent contamination.***

## CONCLUSION

Contamination in vacuum furnace heat treatment can have a significant impact on the final product's quality. Operators must be aware of the potential sources of contamination, the chemical reactions involved, and the color identification for discoloration. Prevention through proper cleaning, vacuum level, temperature, and appropriate materials and seals is critical to ensure a successful heat-treatment process. Regular maintenance and inspection are necessary to ensure the vacuum furnace's integrity and prevent contamination. All hot zones run to fruition, and their specific life cycle end. Maintaining your leak rates, watching for process changes, and running scheduled cleanup cycles (tailored around your specific trace elements) can significantly increase overall operational equipment efficiency (OEE) and profit.

## REFERENCES


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## ABOUT THE AUTHOR


Janusz Kowalewski is sales director for Southeast Asia at Ipsen USA.