# Hot zone contamination

This sixth article in our series on vacuum heat treating examines contamination of the normally high-purity vacuum environment and explains how to determine if the hot zone is at fault.

by WILLIAM R. JONES

One of the major justifications for vacuum processing is to insure a bright, oxide-free heat treated or brazed product. When the result is oxidized or dirty parts, often after consultation with the furnace operators and the maintenance department, the verdict is a "contaminated hot zone" caused by a mysterious gremlin. Before concluding that this is the case, there are several factors that should be ruled out first. Most oxidation occurs from air leaking into the vacuum chamber or air leaking into the inert gas backfill lines, or from contaminated gas from the source of gas supply.

| Time (minutes) | Vacuum pump-down pressure (Torr) | Rate of rise Microns Hg/hr |
|---|---|---|
| Start | 750 | |
| 10 | $1 \times 10^{-3}$ | 150 |
| 15 | $5 \times 10^{-4}$ | 80 |
| 30 | $1 \times 10^{-4}$ | 50 |
| 60 | $3 \times 10^{-5}$ | 10 |
| 90 | $1 \times 10^{-5}$ | 3 |
| 120 | $8 \times 10^{-6}$ | 2 |

**Table I: Typical interrelationship of time, vacuum pump down and rate of rise, for empty and clean furnace**

Backfilling lines, valves, pressure regulators and components go through considerable stress from intermittent high-velocity gas flow, first cooling and then re-warming to ambient temperature. Everything starts out "bubble tight," yet with service of weeks or months, joints or components fail and start to leak. Soft solder joints may crack and teflon tape-sealed threads (Note 1) start to leak. No leaks can be permitted, as high-velocity gas will cause aspiration of air. Wherever possible, a gas reservoir or surge tank should be used—of sufficient capacity to handle the gas volume for backfill and located up close to the vacuum furnace, to reduce stress on the entire gas distribution network. The backfill valve (Note 2) should be mounted on the furnace with a short run to the surge tank, with hard brazed joints to minimize potential gas leaks.

The source of inert gas supply should be reviewed. Large liquid sources of nitrogen or argon are known to be of the best quality, with smaller dewars of less quality and cylinder gas of the least quality. If the gas source is questioned, a trace oxygen measurement (Note 3) on each dewar or gas cylinder should be performed. A satisfactory gas measurement can be made in four hours; normal readings would be 1 to 2 ppm by volume. Readings over 10 ppm are cause for gas rejection.

## Leak testing

In order to insure that the vacuum furnace is tight, a leak rate or pressure rise measurement should be made. To avoid confusing outgassing of the hot zone with a real leak, either (A) the furnace should be pumped down for an adequate outgassing period (see **Table I**); or (B) the empty furnace should be heated to near-maximum operating temperature, soaked out for one hour, gas quenched to near ambient temperature and then re-pumped down for approximately 30 minutes, without opening the furnace to air. Following either prolonged vacuum pumping (A) or the thermal outgassing cycle (B), the vacuum pumping valve is closed and the vacuum decay of the furnace chamber noted. At the beginning of closing off the vacuum valve, the pressure of the furnace vacuum gauge is noted. After 10 minutes, the vacuum gauge reading can be noted again.

As an example, if the start pressure is 10 microns Hg and after 10 minutes the vacuum rise is to 12 microns Hg, this is a rise of 2 microns in 10 minutes, or a rate of rise of 12 microns Hg per hour. Some tight aircraft brazing cycles may allow no more than a 2 micron Hg per hour specification, where an automotive copper brazing or tool steel processing furnace would permit a leak rate of up to 25 to 50 microns Hg per hour. Generally, a degassed vacuum furnace will have no trouble passing a 10 micron Hg/hour leak rate test.

Leak rates higher than 10 microns Hg per hour are questionable and may be a result of insufficient vacuum pump-down time (see **Table I**), or insufficient thermal bake-out. However, if this has been ruled out, the conclusion is the vacuum furnace is leaking and the leaks must be located and fixed. High vacuum leaks are most often located with a helium mass spectrometer leak detector (see Note 4). This instrument is physically connected to the vacuum pumping system and helium gas sprayed around the chamber. Helium leaks into the chamber through a potential crack or joint and the leak detector responds with a meter indication or sound alarm.

Helium leak detection is often tricky; even though no leaks are found with the leak detector, this does not mean the vacuum furnace is leak-free, only that the leak or leaks have not been located. The final test is always the rate of rise test, always after proper vacuum pumping or thermal degassing. Note from **Table I** the possibility for misleading information from short vacuum pump-down time. Note

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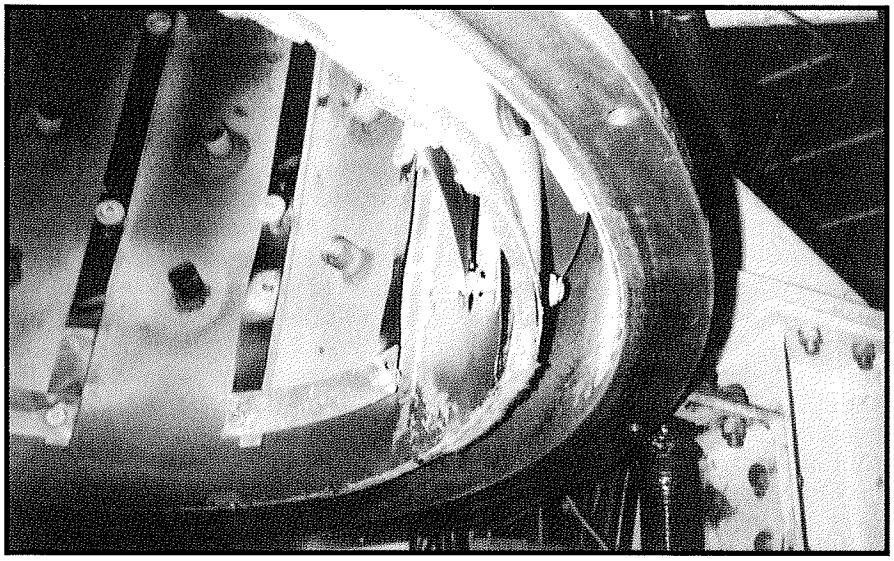
also the relationship between the vacuum gauge reading and rate of rise. If the vacuum gauge pressure reading is not down, the leak rate will correspondingly be poor. Therefore, a *properly working* vacuum gauge on a *given* vacuum furnace and vacuum pumping system is an excellent indicator of the overall vacuum furnace integrity.

Exposure of the vacuum furnace to atmospheric air on work loading and unloading is another unbalancing factor. It is well known that a vacuum furnace hot zone should never stand open to air for any prolonged period of time, because moisture will contaminate the furnace internal. What is not as well understood is that exposure to air for only a few minutes substantially affects the re-pump-down characteristics of the vacuum furnace (see **Table II**).



***"White" contamination appears around the cold wall of a vacuum chamber and the outside of a hot zone assembly in a bottom-loading vacuum furnace.***

## The hot zone

Materials of construction for the hot zone, as well as the overall cleanliness of the hot zone and vacuum furnace chamber, affect these results. Clean, all-metal hot zones will show the least effect from air exposure (moisture), where over-insulated hot zones[3] of more than two inches of alumina felt or board perform poorly because of the basic hygroscopic nature of this material.[1] On heating, residual gas analyzer data indicates that water is continuously released even to hot zone temperatures in excess of 2100°F.[2] So initial prolonged vacuum pump down is only the start of the problem.

Having determined that all else is correct, can the hot zone be contaminated? Operating experience indicates that eventually this may be the case, due to excessive dirt in the furnace acting as a gas "sponge." However, this will not normally occur until after several years of continuous operation. In one recent experience, the leak rate was low, one or two microns Hg per hour, with a good vacuum level of under $1 \times 10^{-5}$ Torr. (Pump down was considerably longer than normal.) Burn-out cycles to 2650°F for one hour no longer would restore performance. Sensitive alloys like 17-4 PH and titanium alloys would not process clean. 300 Series stainless steel was beginning to show yellow and dark areas, and the furnace was set aside for less critical applications, where bright work was not a factor. The furnace then deteriorated further.

The furnace hot zone was pulled out of the furnace for inspection, and it was noted that the entire outer hot zone assembly was coated with between 1/8- and 3/16-inch of dirt. The same was true for the entire inner cold wall for the vacuum chamber. This was thoroughly scraped out of the furnace internals and cold wall area, and then washed down with solvent, mineral spirits, followed by acetone. One full cubic foot of dirt and debris was collected. This was made up of dehydrated oils, grease, binders, and an assortment of products of bake-out from hundreds of workloads. This 50-inch furnace was in a commercial heat treat shop and was exposed to numerous applications. It was observed that improper product degreasing was a factor, and a new and larger vapor degreaser was installed to help clean up products entering the furnace. One of the products run in this furnace was coils of tubing difficult to clean on the I.D. After clean-up and restart of the vacuum furnace, and a few "run in" cycles, the furnace was restored to fully satisfactory, clean operation as was the previous experience.

Discussion with commercial heat treat shops shows that others have similar experiences with various vacuum furnace manufacturers. For some applications and installations, it may be necessary to schedule a hot zone tear-out and furnace scrape and clean-up on a regular, once-every-two-year basis.

| | Pressure in Torr | |
|---|---|---|
| Time (minutes) | $N_2$ release | $N_2$ release and 1 minute open to air |
| Start | 750 | 750 |
| 10 | $1 \times 10^{-3}$ | $2 \times 10^{-3}$ |
| 15 | $5 \times 10^{-5}$ | $1 \times 10^{-4}$ |
| 30 | $1 \times 10^{-5}$ | $5 \times 10^{-4}$ |
| 60 | $8 \times 10^{-6}$ | $2 \times 10^{-5}$ |
| 90 | $7 \times 10^{-6}$ | $9 \times 10^{-6}$ |

**Table II: Vacuum pump down (typical), for empty and clean furnace**

In another situation, at a captive braze shop, another furnace ran in production for seven years without any problems. The exposure to varied and dirty applications was not the case in this operation.

A contaminated hot zone may be the problem, but one should reach this conclusion only after ruling out every other possibility. HT

**NOTES**

1) Preferable to tape, SWAK (anaerobic pipe thread sealant, TFE; manufacturer is Cajon)
2) Solenoid-type Asco Model 821OD22
3) Teledyne Model 315 trace oxygen analyzer range 0-10 ppm
4) Alcatel model ASM110 CL turbo helium mass spectrometer leak detector

**REFERENCES**

1) Hot zone alternatives, C.R. Pierce, *Heat Treating*, July 1988
2) Vacuum—another atmosphere? W.R. Jones, *Heat Treating*, Oct. 1986
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