Partial Pressure Vacuum Processing - Part II: Applications

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This article is based on a presentation at the Furnaces North America '96 Conference, held in Dearborn, MI, September 24 - 26, 1996 and sponsored by Industrial Heating. In part two, partial pressure vacuum processing cycles for different materials are reviewed. Information regarding the evaporation of base metals and reactive processing, was presented in the September '97 issue of Industrial Heating.

Vacuum processing has been established as a premier way to insure consistency, cleanliness and to minimize distortion of heat treated parts. In recent years the range of vacuum heat treating applications has broadened, as higher vacuum levels and more intense positive pressure cooling capabilities have become fully commercialized. However, partial pressure processing is another side to vacuum processing that remains relatively obscure, yet offers significant benefits.

As was stated in part one of this series, partial pressure atmospheres are used during vacuum heat treating to eliminate evaporation of the alloys in the base material. This is accomplished when gases flow into the vacuum furnace hot zone during processing. It is necessary because the base material heat treatment requires processing temperatures higher than the evaporation points of the alloying elements.[1] The partial pressure gas atmosphere prevents evaporation from occurring. A secondary, but necessary, benefit is to prevent the evaporating elements from coating the inside of the vacuum furnace and destroying heating elements and hot zone components.[2]

Some possible inhibiting factors in using partial pressure processing are the expense of purchasing and operating high vacuum diffusion pumps, and many early furnaces have not been properly outfitted for partial pressure gas operation. The following processes and cycles should help one to see the advantage of partial pressure gas operation and its feasibility with most vacuum furnaces that are commercially available today.

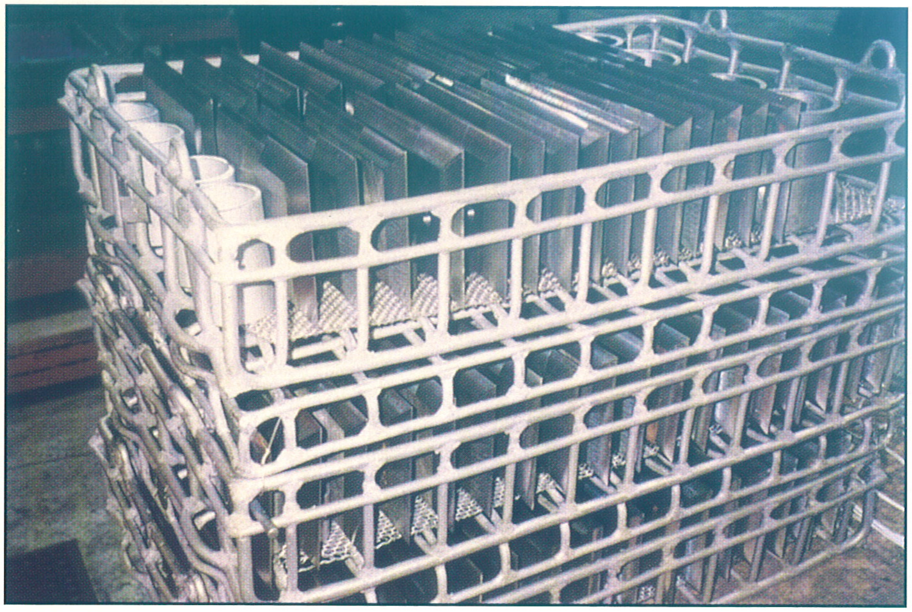

Fig. 1 A-2 tool steel prior to vacuum heat treating in partial pressure nitrogen atmosphere.

Partial Pressure Processing Cycles

The majority of examples discussed in this article will illustrate the use of nitrogen, which is the most commonly used gas in partial pressure cycles. However, because of the properties of various alloy elements, and the need to attain certain metallurgical, mechanical, or electrical properties, gases such as argon and hydrogen are also utilized.

Some materials adaptable to partial pressure processing, such as titanium base alloys, are best processed in argon rather than high vacuum to avoid gettering of these materials. Also, this is the best procedure to accomplish an improved purging of contaminating gases such as water vapor, hydrogen and carbon monoxide from the hot zone. Stainless steel alloys are best processed in hydrogen partial pressure to provide a reactive oxide reducing agent.

The following seven examples reveal a variety of vacuum processes that can be accomplished with the use of partial pressure gas operations. The examples demonstrate the gases, materials and processes that can be utilized for various heat treating, vacuum brazing and sintering cycles. Abbreviated cycles are given to point out the distinction of the partial pressure cycles.

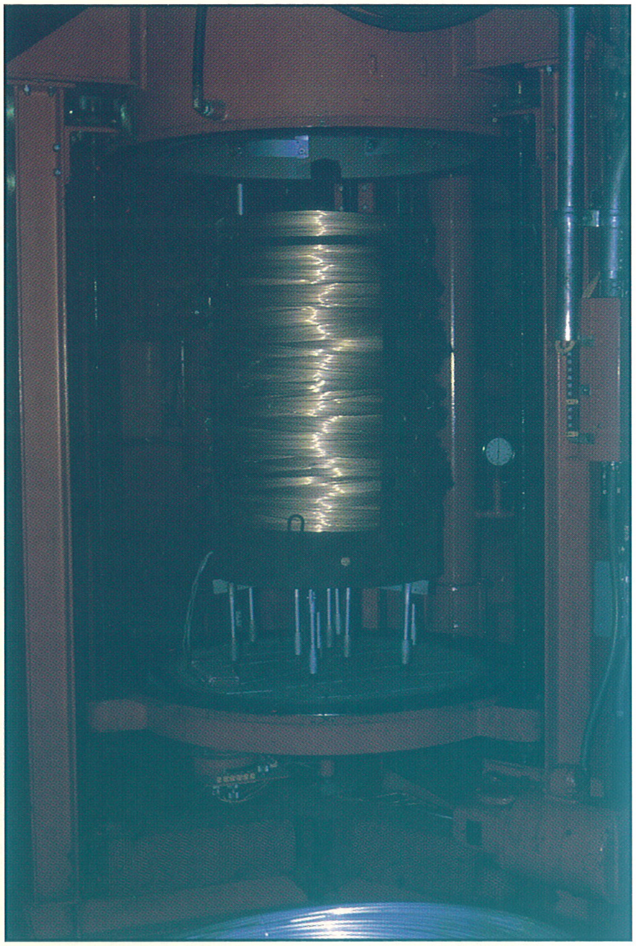

Fig. 2 Brass tubing loaded into a vacuum furnace after running in partial pressure nitrogen atmosphere.

Example #1: Partial pressure nitrogen processing, rather than high vacuum processing, is used for hardening of tool steels, such as A-2, H-13 and 440 stainless steel to avoid evaporation of base materials (Fig. 1). A typical processing cycle is:

1. Pump down to 50µ Hg, Introduce 50 SCFH partial pressure - nitrogen gas flow for a pressure of approximately 1 torr.
2. Ramp at 30°F/minute to 1725°-1775°F. Hold at temperature for 30 minutes (minimum at the core of the part).
3. Backfill with nitrogen, rapid quench to ambient, temper to desired Rockwell hardness.

Example #2: Partial pressure nitrogen is also used to anneal nonferrous materials such as brass and aluminum, particularly tubing (Fig. 2). The purpose of partial pressure is to avoid evaporation of the high vapor pressure elements present in brass or aluminum and to eliminate internal tubing lubricants that would otherwise be considered contaminants in a gas furnace atmosphere. A common brass annealing cycle is:

1. Pump down to 50µ Hg or less. Introduce 80 SCFH gas flow of nitrogen.
2. Ramp at 30°F/minute to 1000°±25°F.
3. Switch to -5 inches Hg (near atmospheric pressure) when work thermocouple is at 1000°±25°F.
4. Hold at 1000°±25°F for 30 +5/-0 minutes.
5. Nitrogen quench to ambient.

Example #3: Because of the material's composition, orthopedic implants (Fig. 3) are run in partial pressure argon. The partial pressure will prevent evaporation of chrome or nickel from these materials.

1. Pump down to 50µ Hg or lower.
2. Ramp at 35°F/minute to 1800°±25°F (furnace temperature).
3. Introduce partial pressure argon at 75 SCFH gas flow, approximately 1 torr.
4. Ramp at 35°F/minute to 2175°±10°F.
5. Hold at 2175°±10°F for 4 hours (+30/-0 minutes).
6. Argon gas quench at 2 bar minimum positive pressure to ambient temperature.

Example #4: Vacuum brazing utilizes partial pressure nitrogen gas in this copper braze cycle (Fig. 4). This partial pressure will prevent the evaporation of copper, the brazing alloy.

1. Mechanical pump down to less than 50µ Hg.
2. Ramp at 20°F/minute to 1875°F.
3. Introduce 150-175 SCFH nitrogen gas flow, approximately 5 torr.
4. Hold at 1875°F for 30 minutes.
5. Ramp at 10°F/minute to 2025°-2050°F.
6. Hold at 2025°-2050°F for 30 minutes.
7. Furnace cool to 1800°F. Backfill with nitrogen and quench to ambient temperature.

Example #5: Mumetal and Carpenter 42 alloys (Fig. 5) are annealed in a hydrogen atmosphere using this cycle. The hydrogen partial pressure gas is to

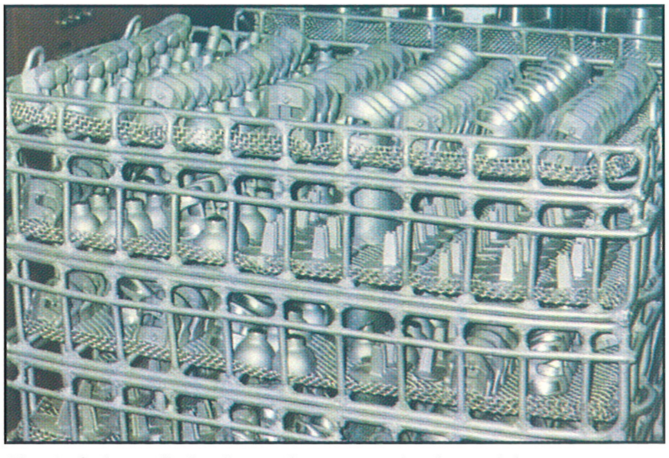

Fig. 3 Orthopedic implants after processing in partial pressure argon atmosphere.

Fig. 4 Heat exchangers after copper braze cycle.

Fig. 5 Mumetal and Carpenter 42 alloys after hydrogen magnetic anneal.

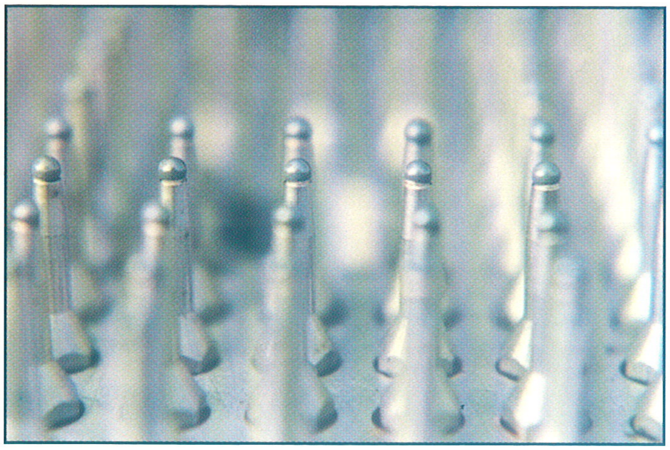

Fig. 6 Glass-to-metal seal diffusion bond after processing.

act as an active oxide reducing agent in this process cycle.

1. Fixture parts for minimum distortion and sticking.
2. Pump down to 50µ Hg or lower.
3. Ramp at 30°F/minute to 1000°F, furnace temperature at 1000°F.
4. Introduce 20-30 SCFH partial pressure hydrogen gas flow.
5. Ramp at 30°F/minute to 2044°±34°F. Hold at 2044°±34°F for 5 hours±15 minutes.
6. Furnace cool to 900°±34°F. Hold at 900°±34°F for 4 hours±15 minutes.
7. Backfill and nitrogen gas quench to ambient temperature.

Example #6: Diffusion bonding in a glass-to-metal seal (Fig. 6) occurs with partial pressure using argon gas.

1. Pump down to 50µ Hg or lower.
2. Introduce argon gas flowing 75-100 SCFH.
3. Ramp 30°F/minute to 1400°F.
4. Hold at 1400°±10°F for 10 minutes.
5. Ramp at 30°F/minute to 1650°±10°F.
6. Hold at 1650°±10°F for 20 minutes.
7. Vacuum cool to 1400°F or lower. Backfill nitrogen gas cool to ambient temperature (170°F or lower).

Example #7: High temperature sintering for powdered metal parts (Fig. 7) also requires argon partial pressure.

1. Pump down to 50µ Hg or lower.
2. Ramp at 30°F/minute to 900°±10°F.
3. Hold at 900°±10°F for 30 minutes.
4. Introduce partial pressure argon gas flowing 100 SCFH.
5. Ramp at 10°F/minute to 1800°±10°F. Hold at 1800°±10°F for 30 minutes.
6. Ramp at 10°F/minute to 2210°F. Hold at 2210°±10°F for 2 hours.
7. Cool to room temperature. Furnace cool to 1400°F or lower, argon backfill and cool to room temperature.

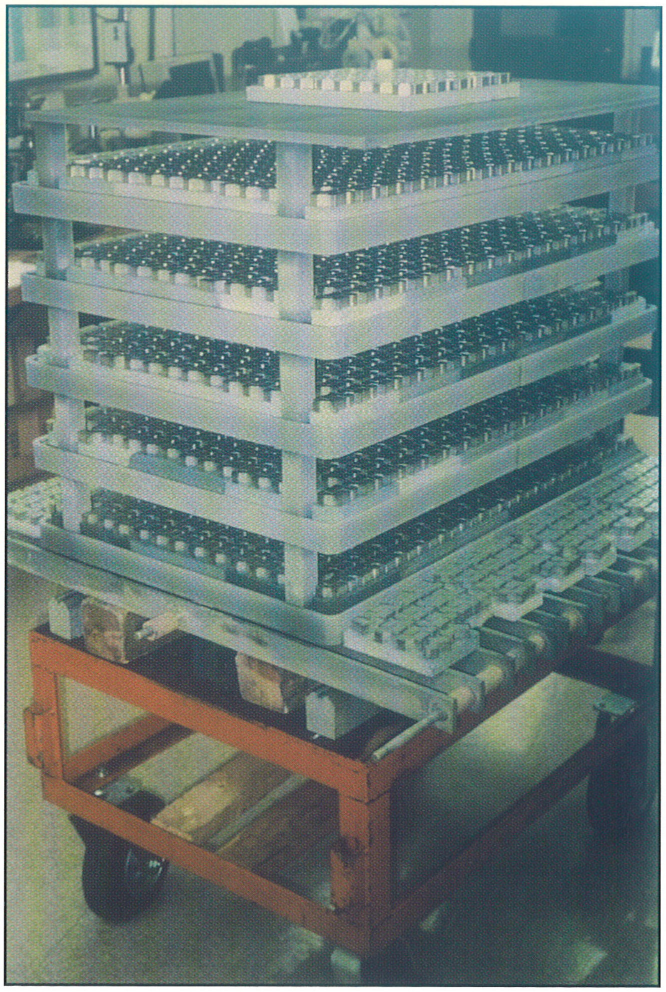

Fig. 7 A load of stainless steel parts after sintering in argon atmosphere.

CONCLUSION

As shown above, partial pressure atmospheres can be used in everyday heat treating and brazing operations without the use of high vacuum diffusion pumps. It is not necessary to pump down to deep vacuum levels in order to process various materials as shown.

The examples given here are only a few of many processes developed for commercial operations. Probably one-half of Solar's production is specified in partial pressure units, rather than high vacuum, for reasons already stated. Although others have specified partial pressure operation in the past, often this has been rejected as a result of poor performance (most notably oxidation or reacted work). This has been overcome by two factors heretofore overlooked: 1) injection of the partial pressure gas directly into the furnace hot zone at one or more locations; and 2) by controlling the partial pressure injection gas stream as a continuous flow rate (monitoring this gas flow with a standard atmospheric rated flowmeter, i.e., in SCFH units) rather than a specific operating pressure (often in an "off-on" objectionable mode).

REFERENCES

[1] CRC Handbook of Chemistry and Physics.

[2] Hot Zone Contamination, William R. Jones, Heat Treating, January 1989.