

VACUUM GAS-NITRIDING FURNACE PRODUCES PRECISION NITRIDED PARTS

A vacuum furnace and control systems developed for nitriding yields uniform, precision nitrided components in reduced cycle times with controlled white layer to customer specifications.

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Currently, nitriding is carried out predominantly in pit type vertical furnaces with metal alloy retorts to hold the work load during the nitriding cycle. The large thermal mass of these furnaces requires long heat-up and cool-down times. Another factor is that the ammonia nitriding gas cracks not only on the work load but on the metal retort too. In time, this leads to non-uniform nitriding of the work, and the retort has to be conditioned before uniform nitriding can be restored. In contrast, the much smaller thermal mass inherent in vacuum furnaces (as well as other features) offers an opportunity of designing a more desirable vacuum furnace for providing efficient uniform nitriding. Such a furnace was designed and developed over several years to replace traditional retort gas nitriding^[1].

Furnace Considerations

Two of the main factors in constructing a vacuum furnace system for nitriding are the physical design of the furnace proper and the nitriding atmosphere control system. The furnace

that was developed is a horizontal, single-chamber, front-loading furnace equipped to case harden a stationary load of various materials by means of nitriding gas or gas mixtures during the heat cycle. After nitriding, the material is cooled rapidly by external gas cooling at a positive pressure in a single chamber of the furnace (Fig. 1). The chamber is separated into an inner portion and an outer portion.

The inner portion (work chamber) consists of graphite insulation, eight vertically arranged heating elements, and a number of strategically placed graphite baffle plates. Piston-driven graphite port plugs are closed during the nitriding cycle, providing a gas seal, and are opened at the end of the nitriding cycle to provide rapid and efficient cooling. Two graphite fans at the top of the inner portion circulate the nitriding atmosphere uniformly within the work chamber. This system provides a uniform distribution of the nitriding gas, and thus produces uniform case hardening of the work load (Fig. 2).

The hot zone includes a work zone of approximately 36 in. wide x 30 in.



Fig. 1 — Vacuum furnace and control system.

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high × 48 in. deep. It is made completely of graphitic materials that are inert to the anhydrous ammonia used in nitriding.

The furnace provides rapid, uniform

convection heating and rapid cool down. Thus, the cycle time is reduced significantly over retort-type nitriding. However, in addition to an efficiently designed vacuum furnace, it is neces-

sary to have an atmosphere control system that provides precise control of the nitriding atmosphere. The control system was supplied by Super Systems Inc. (Cincinnati, Ohio).

Precise control of the nitriding atmosphere is a prerequisite to providing the required microstructure in the nitrided work load. The structure is dependent on the degree of dissociation ($VN_2 + H_2$)^[2] of the ammonia or a factor called the nitriding potential (K_n)^[3].

The possible phases that can be present in the nitrided layer include alpha (α) phase (a solid solution of nitrogen in ferrite), gamma-prime (γ') Fe_4N , and epsilon (ϵ) Fe_2-3N . Some or all of these phases may be present. A structure at the surface (white-layer) can be composed of γ' and/or ϵ . Controlling the ammonia concentration by flow or by diluting with N_2 or H_2 will dictate the degree of dissociation, and, in turn, the phases that will be present (Figs. 3 and 4). The K_n value can be calculated using the following formulas: For ammonia-nitrogen mixtures:

$$K_n^{(+N_2)} = \frac{1 - BR}{\{1.5(BR - N_2^o) / (2 - N_2^o)\}^{1.5}} \quad (1)$$

For ammonia-hydrogen mixtures:

$$K_n^{(+H_2)} = \frac{1 - BR}{\{BR(1.5 - H_2^o) + 0.5H_2 / (2 - H_2^o)\}^{1.5}} \quad (2)$$

where BR is the burette reading, N_2^o or H_2^o is the amount of nitrogen or hydrogen, respectively, added originally, and H_2 is the hydrogen from dissociation.

The values of K_n calculated from equations (1) or (2) in conjunction with the *Leher Diagram* (Fig. 5) may be used to determine the structures that will be present during nitriding with ammonia mixtures^[4]. The critical values of K_n from the *Leher Diagram* for the phase boundaries versus temperature at one atmosphere are shown below.

Temp., °F	K_n ($\alpha - \gamma'$)	K_n ($\gamma' - \epsilon$)
925	0.30	2.2
975	0.23	1.5
1000	0.21	1.3
1025	0.20	1.2
1050	0.16	1.1

There are various automated instruments used to determine the degree of dissociation. Measuring the percent of hydrogen works well if nitriding using pure ammonia or a mixture of am-

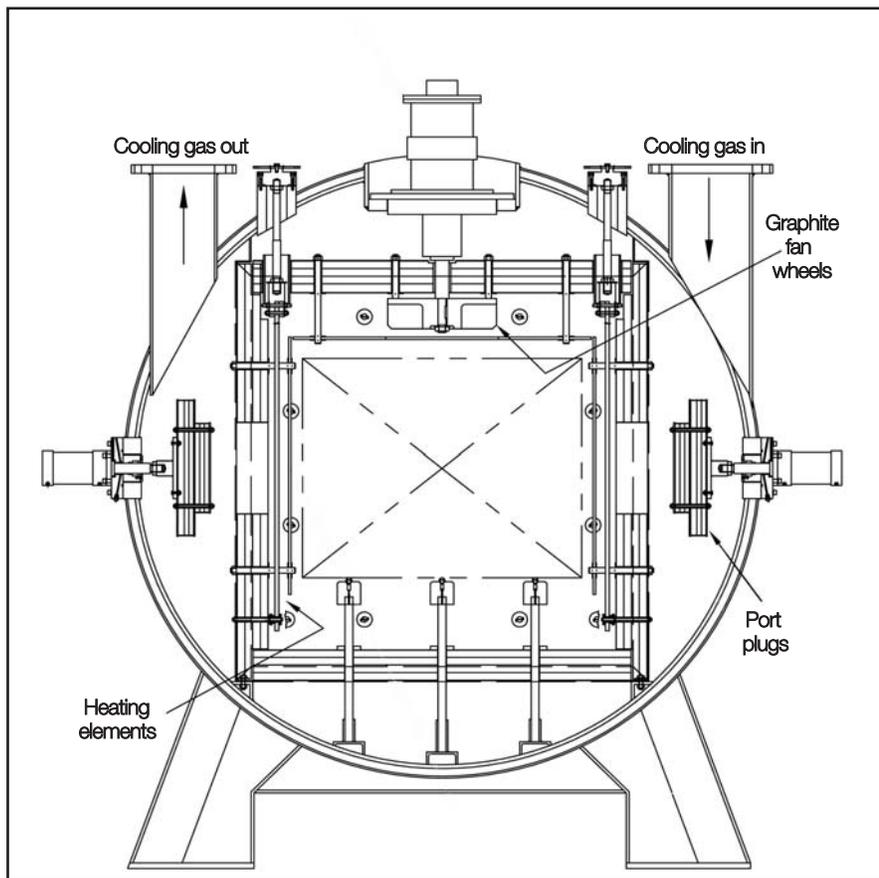


Fig. 2 — Schematic of furnace hot zone and vacuum chamber.

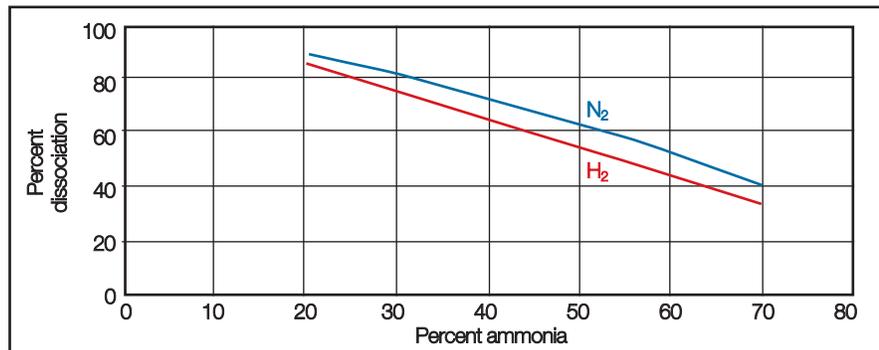


Fig. 3 — Dissociation curves of $NH_3 + N_2$ or H_2 .

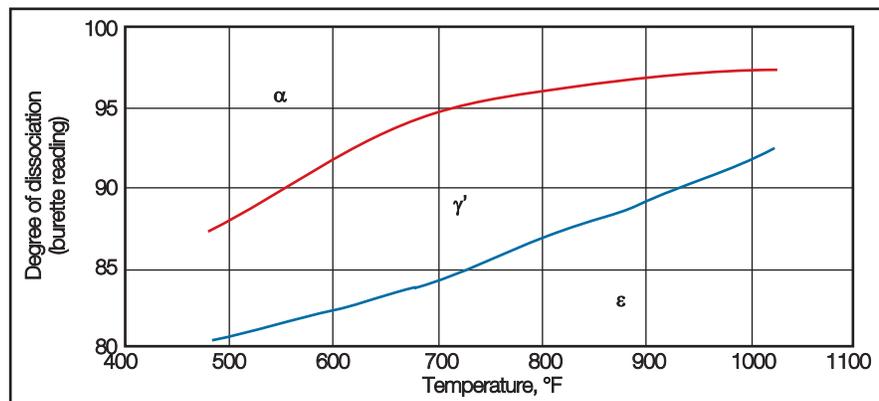


Fig. 4 — Possible nitrided phases for a 20% $NH_3 + N_2$ mixture at 1 atmosphere.

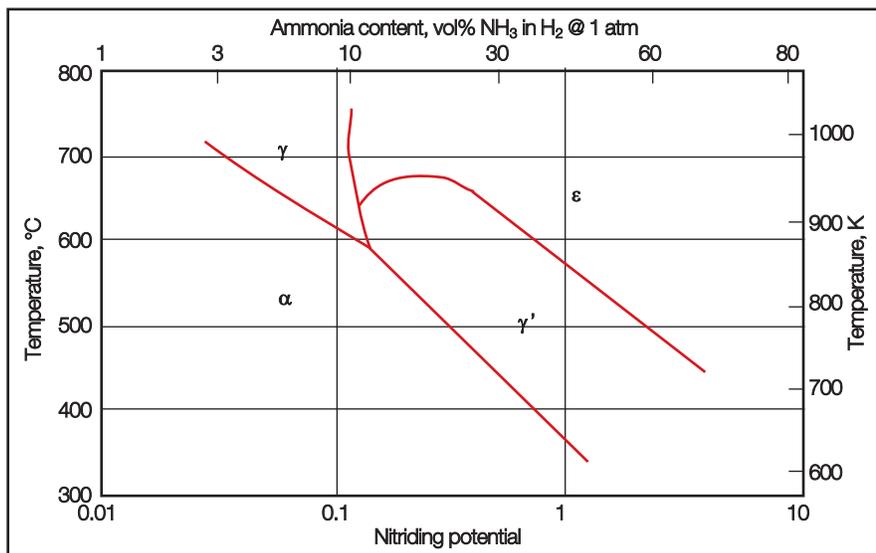


Fig. 5 — Lehrer diagram

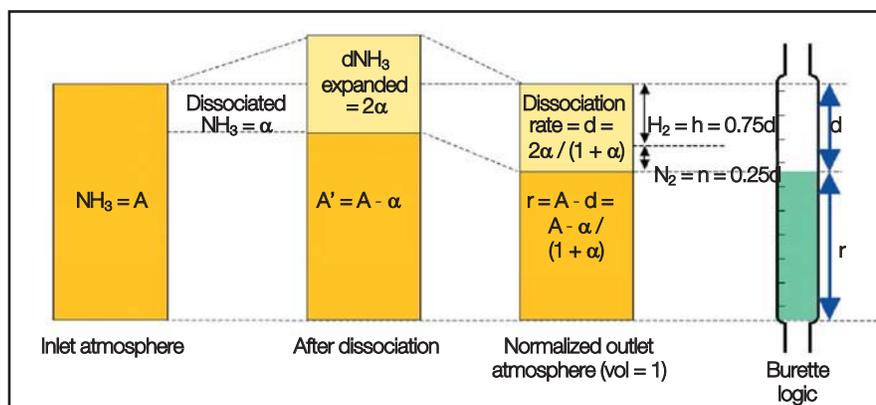


Fig. 6 — Dissociation burette schematic for ammonia.

monia and nitrogen. A good check on the automated degree of dissociation instrument is can be performed using a nitriding manual burette (Fig. 6)^[5].

Nitriding Cycle

A typical nitride cycle includes start up, nitride, and cooling the workload. Start-up steps are:

1. Place the workload directly in the furnace or in alloy steel baskets that are placed on graphite hearth rails.
2. Close the furnace door and gas port plugs to seal the furnace from leakage of gas.
3. Evacuate furnace outer chamber and hot zone to approximately 10^{-2} torr to remove essentially all of the air from the furnace.
4. Backfill furnace with nitrogen to approximately to 0.5 psig (800 torr) via a back fill valve.
5. Introduce partial pressure of nitrogen through a gas inlet, and start gas circulating fans.
6. Heat furnace to set nitriding temperature (up to 1400°F).
7. Pump out a portion of the nitrogen at the set temperature to a set

pressure below 800 torr.

8. Backfill furnace with ammonia to a set furnace pressure of 800 torr.

9. Introduce partial pressures of ammonia and nitrogen continuously at a fixed ratio with flow controllers, which are set to control the flow as required by the specification. Gas is vented to atmosphere via a gas purge valving arrangement.

The gas is circulated and is also fed into a nitriding gas analyzer that determines the hydrogen concentration. The results are fed back into the control system that regulates the gas flow to the desired ratio.

Cooling the workload involves:

1. Shut off heat and ammonia flow, and pump down furnace to about 1 torr.
2. Backfill furnace with nitrogen when pressure of 1 torr is achieved to a pressure of about 1,010 torr.
3. Automatically, a mechanism opens gas port plugs, and the blower and circulating fans are turned on to gas cool the workload. The gas is circulated through an external cooling heat exchanger, where the cooled gas is recir-

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Fig. 7 — Representative parts for vacuum gas nitriding.



Fig. 8 — Cross section of vacuum gas-nitrided surface.

culated to the furnace to further cool the workload to the desired temperature.

Typically, using this vacuum furnace, cycle time can be reduced by up to 50% of the time required using conventional retort-type furnaces. Furthermore, a variety of materials have been nitrided successfully to customers' specifications; including 4140, 4150, 4330, and 4340 steels, Nitralloy, and H-11 and A-2 tool steels. Figure 7 shows representative

parts ready for nitriding, and Fig. 8 shows a cross section of a nitrided case.

Conclusion

The development of this furnace and control systems allows the company to provide nitriding services that yield uniform, precision nitrided components with controlled white layer to customer specifications. Single stage, and two stage (Flo) processing have been developed that provide superior metallurgical properties more efficiently, for virtually any application.

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References

1. U.S. Patent application dated December 13, 2009
2. V.M. Zinchenko, et. al., Nitrogen Potential, 3rd Lakhtin Memorial Lecture, 9th Seminar of the Intl. Fed. for Heat Treatment and Surf. Engrg., 21-23 Warsaw, Poland, Sept. 2003.
3. D. Jordan, et. al., Low Torr Range Vacuum Nitriding of 4140 Steel, *Heat Treating Progress*, (8), p 33-38, Mar./April, 2008.
4. E. Lehrer, *Z Electrochem*, Vol. 36, p 383-392, 1930.
5. J. Darbellay, Gas Nitriding: An Industrial Perspective, MSE 701 Seminar, March 22, 2006.

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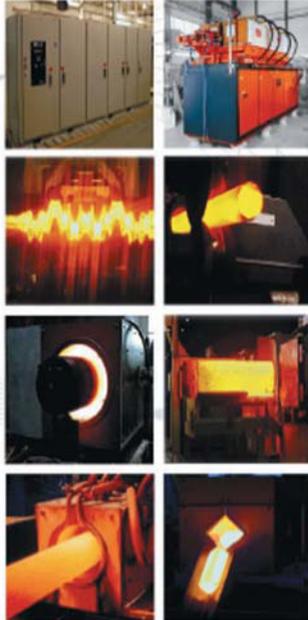


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