The Future of the Integral Quench Furnace

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Abstract

Welcome to the 21st Century and the future of the integral quench furnace. The future means safer processing, simple, yet advanced automation, and environmental compliance, all with the proven performance of low pressure processed component parts. The future furnace combines the benefits of low-pressure vacuum carburizing (LPC) technology with atmosphere integral oil quenching. The future means less gas usage, tighter process control, better temperature uniformity, effortless hardening and increased productivity at a highly competitive price point. Added benefits are seamless annealing, normalizing, stress relief, and high temperature hardening processes.

Introduction

Atmosphere and low-pressure vacuum gas carburizing are well known and long established technologies, which have heretofore rarely competed in the same commercial space. The lessons learned from both processes is that a combination of the workhorse benefits of atmosphere and the superior metallurgical and processes of vacuum can be combined at a cost that can offer design engineers and heat treaters the best of both worlds.

This paper discusses the combination of these designs into a new furnace offering. The furnace, named the Super IQ™, is not revolutionary, it is evolutionary. It is not a hybrid design, but a re-imagining of existing atmosphere integral quench furnaces, while eliminating the need for endothermic carrier gas produced by a generator or supplied by a nitrogen-methanol system. Gone are the flames, smoke and dangers associated with atmosphere furnace processing. Gone are the dirty, old days, replaced by a clean, quiet operation. The quench uses the same familiar oil quench technology as its atmosphere furnace cousins. Results are repeatable with known distortion characteristics. Commercial heat treaters now have a flexible piece of equipment that is high quality, yet processes affordably. High-end processors and automotive industries have a work horse that can be run day after day with exceptional quality, and industrial machinery markets have a furnace that can be turned on and off, as needed, to fit shifts and work days.

Brief Overview/History of the Technologies

Atmosphere Designs

Atmosphere integral (sealed) quench furnaces, either in a batch in-out or continuous straight-through configuration, were introduced to industry following World War II, and what has followed over the past 70 years has been a series of design upgrades/improvements, but with few real technological changes. They are the de-facto standard in the general heat treatment industry (Figure 1). A typical atmosphere integral quench furnace has four main components.

1. A heating chamber with either gas-fired radiant tubes or electrical heating elements and an atmosphere recirculating fan
2. An oil quench with oil agitation system,
3. Instrumentation and controls, often including a limited form of HMI (Human Machine Interface)
4. Gas composition sensors and instruments (often in the form of oxygen (carbon) probes, three-gas analyzers or dew point instruments and shim ports to make manual and/or automatic adjustment of hydrocarbon (natural gas or propane), air and (optionally) ammonia additions.

In addition, an entry vestibule (above the oil in the case of a batch in-out design) is often provided as is a so-called “top cool” located above the vestibule for processes requiring a slower gas cool.
**Vacuum Designs**

Low-pressure carburizing furnaces, either in a batch in-out or continuous straight-through configuration, were introduced in the mid 1990’s based on designs originally developed some 30 years earlier. They are the *de-facto* standard in the Aerospace Industry (Figure 2) and today are seeing more widespread adaptation in other industries. A typical vacuum integral quench furnace also has four main components.

1. A vacuum heating chamber with electric heating elements and a cold-wall design. Typical vacuum level being in the order of 10⁻³ Torr.
2. Either a vacuum oil quench or high-pressure gas quench chamber.
3. Instrumentation and controls, including comprehensive vacuum programmable controller and extended HMI (Human Machine Interface);
4. Process simulation and development software (case depth, carbon profile, surface carbon content based on part geometry and surface area and optional hardness prediction based on heat transfer characteristics).

In the batch in-out configuration an entry vestibule (above the oil) is provided, in the case of the straight through design, a pre-heat chamber shortens the heating/carburizing cycle (Figure 3).

**Design Considerations for the Future Furnace.**

**Low-Pressure Carburizing**

Carburizing in the future should be performed in low pressure. Hardening in the future should also be under vacuum. Controlling de-carb in hardening is unmatched in vacuum. For this reason, the future furnace design has to have a vacuum heating chamber. The functions requiring a vacuum chamber makes the future furnace a bit different than standard atmosphere gas carburizers. In a standard atmosphere carburizer the load is transferred over the oil through the Endothermic atmosphere to the heating chamber.

With a vacuum heating chamber, and operating without vacuum in the oil quench vestibule, and assuming the heating chamber is already at an elevated temperature, the load must first be placed into the loading vestibule, which contains a Nitrogen atmosphere. The loading door is closed and Nitrogen is further introduced, to reduce any oxygen levels. Shortly after, the hot door to the heating chamber opens, which also contains nitrogen and is at an elevated “pre-heat” temperature (very similar to a standard gas atmosphere carburizing furnace. After transfer to the heating chamber, convection can be turned on or
a vacuum can be rapidly pulled on the heating chamber as the load is heating. Once the entire part load has reached and is uniformly at temperature, a hydrocarbon gas source is added. Typical gas mixtures are 100% acetylene. Diluting the acetylene can assist in process control by limiting the process reaction.

From this point forward, operation is per common low pressure carburizing method:. Introduction of the gas/mixture directly into the hot zone initiates a catalytic decomposition of the acetylene gas on the surface of the parts to be processes according to the reaction (1).

\[
C_3H_2 \rightarrow 2C + H_2
\]

Once this reaction has taken place, carbon absorbed at the surface of the steel will start to diffuse into the material according to Fick’s second law of diffusion. Because the nature of the carbon saturation is virtually instantaneous, control of carbide formation is performed by injecting the gas in short duration pulses of a few seconds to a few minutes, alternating with intervals either in vacuum or in an inert gas such as nitrogen or argon introduced into the hot zone. During these intervals, the carbon diffuses further into the part. This is called a “boost/diffuse” process cycle, which is repeated until achieving the proper case depth and carbon profile predetermined by the recipe inputted or obtained via the process simulator (SimVac™).

When using acetylene in a low-pressure carburizing process a pressure range of 3 to 10 Torr is typical and vacuum pumps are used to remove the gaseous by-products of the reaction from the system. Both the case uniformity and repeatability of the process can be assured through proper control of the carburizing process.

**Efficiency of the Process**

It is well proven that the availability of carbon in the furnace atmosphere drives the effectiveness of the process, as well as the adsorption of carbon at the part surface and the rate of carbon diffusion into the part under partial pressure.

An example using conventional Endothermic gas carburizing can be seen by looking at the carbon flux (carbon potential) generated from carbon monoxide (CO) and supplemental gases in Endothermic atmospheres, usually methane or propane, which is controlled by a rate function according to Equation (2). Depending on the formation process used, the CO content is in the range of 20-30% of the atmosphere; subsequently the supplemental gases make up about 10% of the total flowrate. Due to the nature of the atmospheric process of carburizing, it must be noted that effective utilization of all of the carbon will not occur.

\[
J = - \beta (C_o - C_s)
\]

In this example, J represents the carbon flux portion of the process atmosphere. It relates proportionally to the carbon transfer coefficient (β) and the difference between both the atmospheric carbon potential and the concentration difference at the surface carbon (C_o – C_s). Typically, the force driving this reaction is in the range of about 0.2% - 1.2%. Knowing this allows one to determine that an Endothermic gas carburizing process will provide up to about 3 g/m³ of carbon into the process.

By comparison, low-pressure vacuum carburizing mixtures offer approximately 60-90% (i.e. not 0.2% - 1.2%) of the available carbon (mass). This promotes a higher potential adsorption force on the surface of the part and is almost 100 times higher than displayed by Endothermic gas! Therefore, depending on the hydrocarbon mixture used in the vacuum carburizing process, about 600 – 900 g/m³ are available, giving an approximate 100 times increase in process efficiency.

**Influence of Time**

An important process parameter is time at carburizing temperature for carbon to adsorb into the alloy and is somewhat dependent on the material being run. The case depth will be determined by the carbon diffusion process (3). The rate of carbon diffusion (J, carbon flux) is dependent upon the different carbon concentrations C_1 – C_2 (at the unit distance dx) with D being the diffusion coefficient. Limiting this difference between carbon concentrations is the solubility of the carbon into austenite and the carbide formation limitation, which normally should not exceed a maximum value of 1.73% C at 1040ºC (1900°F).

\[
J = D \frac{(C_1 - C_2)}{dx}
\]

Another process parameter is temperature, the controlling factor of diffusion coefficient D, which is roughly 50% higher at 950ºC (1740°F) than at 925ºC (1700°F), 100% higher at 980ºC (1800°F) and 200% higher at 1020ºC (1875°F). Therefore, increasing the temperature of the process, significantly reduces the time needed to reach the desired case depth.

This same phenomenon holds for atmospheric pressures and partial pressures, but it is well established that running atmosphere furnaces at elevated temperatures will dramatically decrease the radiant tube and fan life and increase the maintenance needed to keep them operational. The same limitation cannot be said in regard to vacuum carburizing furnaces whose materials of construction are typically designed for a 1315ºC (2400°F) maximum operating temperature (well above the carburizing temperature). It is due to this factor that using the boost/diffuse method at temperatures over 1000ºC (1825°F) can offer tremendous reductions in process time (Table 1), up to 4 – 5 fold as compared to a carburizing processing temperature of 925ºC (1700°F).
Table 1: Vacuum carburizing time as a function of case depth

<table>
<thead>
<tr>
<th>Effective case depth b mm (inches)</th>
<th>Carburizing time (hours:minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>925°C 1700°F</td>
</tr>
<tr>
<td>0.50 (0.020)</td>
<td>1:23</td>
</tr>
<tr>
<td>1.00 (0.040)</td>
<td>5:30</td>
</tr>
<tr>
<td>2.00 (0.060)</td>
<td>22:00</td>
</tr>
</tbody>
</table>

1. Notes:
   a. SAE 5120 (16MnCr5) steel
   b. Effective case depth at 0.35% carbon

Load Transfer to the Quench

The furnace design is similar, but not identical, to that of a traditional atmosphere integral quench furnace. Heated and hardened load transfer in an atmosphere integral quench furnace running under Endothermic or a nitrogen/methanol atmosphere begins when the inner door between the heating chamber and the quench can be opened and the parts are transferred to the quench, all under Endothermic atmosphere. In the furnace, the inner door is opened only after the additional step of backfilling (with Nitrogen) the heating chamber to atmospheric pressure at the holding temperature (as with the atmosphere variant, temperature can be dropped from carburizing temperature to hardening temperature). The influence of this backfill step was one the focal points in the R&D investigation. In the end, the research and trials found any negatives to be insignificant with respect to part contamination in the form of surface oxidation (results of which are summarized below). As well, the research aided in the final design configuration of the system.

Load Transfer Speeds

A load transfer speed of 20-30 seconds - total time from inner door open to the load submerged into the oil, was chosen as the design parameter that most closely mirrors the transfer time in most atmosphere integral quench furnaces. In many applications, parts are heated in the austenitic range and either direct oil quenched (in hardening, for example), or the temperature is lowered to a final hardening temperature (in case hardening, for example) prior to oil quenching. In these processes, a proper transfer speed is critical in order to avoid the nose of the material’s transformation curve.

Oil Selection

Special vacuum quench oil is not required in the furnace, with a standard atmosphere integral quench oil being selected for the trials that were conducted. In other words, the oil is not required to have special additives to minimize gas pickup and allow for vacuum “degassing” of the oil. Oil degradation was considered to be identical to that found in a typical atmosphere integral quench furnace and confirmed at the oil company’s testing laboratory. As well, the difference between always having vacuum over the oil each cycle, compared to the oils used in the atmospheric process trials presented no detectable oxidation variations.

Test Scope

A detailed and comprehensive test plan was established by the Research & Development team and that portion of their work pertaining to the evaluation of part surface contamination will be reported here. Field verification testing on a unit built to the new design and operating at a commercial heat treater was part of the Scope of Work but is not summarized in the work below. Some of the confirmation tests involved:

- Measurement of the oxygen and water vapor levels in the incoming gas supply (in the equipment) immediately before testing;
- Visual and stereographic examination of the test coupon surfaces for evidence of discoloration (Fig. 3);
- Metallurgical examination of part microstructures (unetched and etched);
- Microscopic analysis of oxygen pickup on part surfaces (via SEM/EDS analysis).
- Evaluation/measurement of surface carbon concentration (i.e. carbon pickup).
- Surface/core hardness (after quenching) including microhardness traverses;

Figure 3: Typical appearance of test coupons evaluated for surface oxidation and mechanical/metallurgical property comparison
Test Unit

Tests were performed in a SECO/WARWICK two chamber vacuum integral oil quench furnace (Figure 4) equipped with low-pressure carburizing (LPC) technology located at the Silesian University of Technology in Gliwice, Poland. The furnace heating chamber was modified with the addition of a nitrogen (fast) backfill capability and the quench tank saw the incorporation of a special probe to measure trace (ppm) oxygen levels. One additional feature incorporated was the ability to intentionally add small amounts of air into the quench atmosphere to simulate “worst case” conditions.

Figure 4: Modified test furnace (located at a University in Poland)

Process Confirmation Testing

Three steel grades were selected for process comparison with known atmosphere and vacuum carburizing results:

- SAE 5120 (20MnCr5), a popular carburizing grade used in the automotive and transportation industry;
- X4317H (18CrNiMo7-6), a carburizing grade used in the energy segment
- SAE 9310 (14NiCrMo13-4), a carburizing grade by the Aerospace, Industrial and Automotive industries.

Various process variables were fixed and common to all testing including:

- Loads were charged into the vacuum heating chamber at ambient temperature;
- Pump down heating chamber to the 10\(^3\) Torr range
  - Heat in vacuum
  - Carburize with acetylene at 980°C (1800°F) to a case depth of 0.80 – 1.00 mm (0.032 – 0.040”) effective case depth measured at 550HV(52.5 HRC)
- Drop and hold temperature from the carburizing temperature to the hardening temperature;
- Immediately before quenching, backfill the heating chamber with nitrogen to atmospheric pressure (while measuring the oxygen content)
  - Transfer the load onto the quench elevator
  - Quench into 60°C (140°F) agitated oil;
- Quench for 30 minutes in the oil, raise the elevator and allow the load to drain prior to unloading.
- Wash and temper all samples (at appropriate tempering temperatures).

Test Results – Absence of Part Contamination During Load Transfer

Intergranular oxidation (IGO) and intergranular attack (IGA) are common and tolerated consequences of conventional atmosphere carburizing. Whereas IGO/IGA are completely avoidable in low-pressure vacuum carburizing. In the past designers have compensated for IGO/IGA (via stock allowance, deeper carburized cases and post grinding, etc.), but these added costs can now be avoided.

The phase of testing reported here focuses on the main design change, namely moving from a total vacuum furnace and vacuum transfer to vacuum over the oil quench to a modified design with a non-vacuum oil quench chamber and in particular the transfer of the parts that occurs through the nitrogen atmosphere. Cryogenic nitrogen from a bulk tank was the focus of the testing.

Test Results – Hardness and Case Depth Confirmation

In addition to a comparison of atmosphere and vacuum carburizing results, the effect of potential oxygen pickup from the nitrogen atmosphere was evaluated for the test steels involved. The end result after extensive metallurgical examination using optical and scanning electron microscopy and hardness verification (Figures 5 – 7) showed that part oxidation was not significant enough to be a factor in part performance. These conclusions should be verified on a case-by-case basis.

Figure 5: Verification of process under varying levels of oxygen contamination (3 – 350 ppm \(O_2\)). SAE 5120 or 20MnCr5 steel grade. Surface hardness was greater than 62 HRC on all samples.
Figure 6: Verification of process under varying levels of oxygen contamination (3 – 350 ppm \(O_2\)). X4317H or 18CrNiMo7-6 steel grade (DIN 1.6587). Surface hardness was greater than 62 HRC on all samples.

Figure 7: Verification of process under varying levels of oxygen contamination (3 – 350 ppm \(O_2\)). SAE 9310 or 14NiCrMo13-4 steel grade (DIN 1.6657). Surface hardness was greater than 62 HRC on all samples.

Test Results – Microstructural Analysis

Metallurgical analysis (Figure 8) of all samples analysed showed fully transformed microstructures consisting of tempered martensite, finely dispersed carbides and some retained austenite. Subsequent analysis under the Scanning Electron Microscope revealed decarburization to a depth of approximately 0.005 mm (0.0002") values similar to what is observed when processing under full vacuum. Carbon concentration profiles show a high conformity with the simulation and a very high conformity between tests with no impact on either surface hardness or micro-hardness values.

Figure 8: Representative photomicrographs from a test with 350 ppm of oxygen present in the quench tank nitrogen atmosphere. No surface decarburization nor the presence of any IGO/IGA. Material is SAE 5120 (20MnCr5).

The Design Process

The design team looked at the gas atmosphere version of the sealed quench furnace being sold for many years and also at the vacuum LPC with vacuum oil quench version of the equipment, which also has been built and sold for many years. It was determined that the greatest cost savings could be accomplished by using the standard, simpler oil quench. Thus, the standard atmosphere oil quench, adapted for use with nitrogen rather than Endothermic gas, was chosen to interface with the vacuum heating chamber. This necessitated the different operating procedures of the vacuum furnace, meaning it had to be both a vacuum heating chamber (both for LPC and for through hardening) and an atmosphere chamber for transfer and quenching. The final design accomplished this. Figure 9 shows the 3D model of this design. Figure 10 shows the actual furnace as built.

An additional goal was the need to use already existing material handling systems customers might have at their plants. The engineering team reviewed some of the more popular loading systems and incorporated the ability to load the new furnace with other manufacturers’ loading systems.
As in any furnace design, the engineers must make compromises between features, size, and cost factors. One of the design requirements was to create a furnace that not only competes with the quality of gas carburizing by beating it, but the furnace also needed to be a realistically priced option to those buying a standard atmosphere integral quench furnace and its required endothermic atmosphere peripheral equipment. Heretofore, most LPC or Vacuum Carburizing equipment has been much more expensive than standard atmosphere variants and as such, the design team knew that they must create a top quality piece of equipment, but it must compete in the realm of the standard gas atmosphere integral quench field.

Another design consideration was unit size. In the past, designers always wanted to offer a range of sizes. The team here decided to offer only one size – the popular 36” x 36” x 48” deep. Those using smaller sizes can turn their trays around and double the production. Those using the most popular size are ready to go. The end result is the equipment design shown in Figures 9 and 10. This equipment is now already in the marketing stages and has been fully prepared for commercialization. As well, a full-size, final design furnace has already been installed in the plant of a US-based heat treater next to older, standard atmosphere gas integral quench furnaces utilizing the loading system of the older equipment and the washing and temper system which was already in existence.

One final factor for the design team was to create a furnace design that exhibited high productivity and through-put, much like the atmosphere units. This meant fast transfer into a pre-heated chamber, ability to carburize at higher temperatures and a unit that can perform other processes such as fast through hardening, annealing, normalizing, etc. The design team accomplished all these goals.

Conclusions

A very flexible furnace for through-hardening, case hardening and other thermal vacuum processes (i.e. annealing) under controlled conditions is the result of this development effort. Low-pressure carburizing and vacuum atmosphere has been proven superior to its atmosphere counterpart[8]-[28]. The furnace eliminates the need for endothermic gas and can easily be turned on and off. Compatibility (with most existing gas furnace designs) ensures the Super IQ™ can be integrated with existing material handling and washing and tempering equipment. Its operation is now confirmed and understood, and the cost is very competitive. The bottom line: performance without compromise.

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References


