

Development Experience in Low-Torr Range Vacuum Carburizing

Virginia M. Osterman, Solar Atmospheres Inc., Souderton, Pa.

Vacuum carburizing has become one of the hottest topics in the heat treat industry and the development of low-torr range carburization is an important advancement for metallurgical applications.

The Carburizing Research Team at Solar has developed a vacuum carburizing process that uses a carburizing gas in conjunction with hydrogen as a pretreatment and processing gas to produce high quality carburized parts (a patent application has been filed). The goal was to provide a method that eliminates a highly programmed cycle without losing the benefit of a sequential boost/diffuse process. In conjunction with process development, a special carburizing furnace was developed, which uses an integrated gas-quenching technology, thereby making the entire process available in a single self-contained chamber.

Low-pressure carburizing background

Low-pressure carburization (LPC) is a non-equilibrium process in which small amounts of the carburizing gas are continuously pumped in and out of the furnace at a rate that allows the carbon on the surface of the steel to reach the austenite solubility limit [1]. Four distinct stages of the process are:

1. Heating the parts under vacuum to carburizing temperature and soaking at temperature
2. Introducing carburizing gas to absorb carbon on the surface
3. Removing the reactive gas and allowing the surface carbon to diffuse below the surface
4. Quenching parts to produce the desired microstructure

The benefits of vacuum carburizing over traditional atmospheric carburizing have been the driving force for investigative

studies. The use of moderately higher carburizing temperatures over shorter carburizing times provides a more uniform carburized oxide-free case depth, cleaner parts and less part distortion and eliminates the need for post-process machining. Over the years, vacuum carburizing has become cost effective over traditional atmosphere carburization [2].

Research in the area of vacuum carburizing has been ongoing for over 40 years. Its feasibility as a heat-treating process has become more accepted owing to the use of reactive hydrocarbons as a carbon source [3,4]. In recent years researchers have looked at various hydrocarbon gases [5-9] and liquids [10] with

hopes to find an alternative to soot forming carburizing gases. The main goal of all LPC studies is to provide an oxide free uniform case on the surface and within all recesses while minimizing any soot on parts and in the furnace.

Early work in LPC relied on the hydrocarbon gases such as methane and propane. Methane is not easily cracked in the vacuum furnace and has received limited interest for low-pressure carburization without plasma assistance. The use of propane as a carburizing source is common although there can be a high degree of soot generation owing to excessive decomposition in the furnace. Uniform carbon pick-up and deep hole penetration were not easily achieved with



Carburizing development starts out small in lab-scale retort furnace and finishes big (in production scale furnace; see Fig. 6)

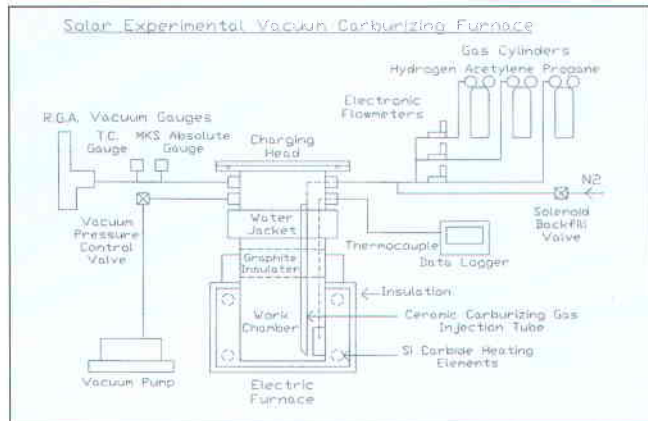


Fig. 1. Schematic of the carburizing system used in Solar's carburizing process development effort

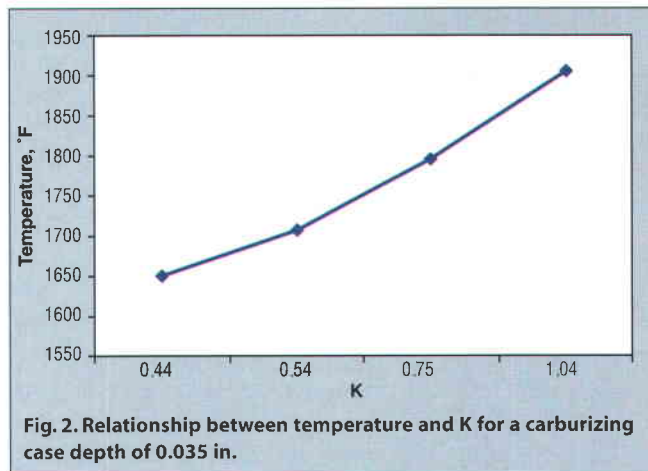


Fig. 2. Relationship between temperature and K for a carburizing case depth of 0.035 in.

propane at pressures below 15 torr [11]. However, in recent years propane has been used successfully with low-pressure techniques in the range of 3 to 10 torr.

The need to develop a cost effective process that was soot free led to the study of unsaturated hydrocarbons such as ethylene [9] and acetylene [3, 5]. Interest in the use of unsaturated hydrocarbons as carburizing agents was not unique to vacuum carburizing. An extensive study [4] of reactive hydrocarbons including propane, ethylene and acetylene looked at the rates of carburization at near-atmospheric pressure. Suggestions for improving rates and decreasing soot were fully outlined.

As early as 1977, the use of acetylene as the carburizing gas for LPC was illustrated [3]. Uniquely, acetylene has an affinity for iron and reacts by forming an iron-acetylene complex, which leads to a metal acetylide [12]. Therefore, theory predicts that acetylene will crack catalytically at the metal surface as opposed to thermally throughout the hot zone. Thus, under the proper conditions, predominant cracking of acetylene on the hot surface should lead to the cleanest possible product, hot zone and cold walls.

One major obstacle in the first acetylene vacuum carburization was soot build up in the furnace and on the parts. Whereas Russian scientists started at 7.5 torr and increased the pressure up to 750 torr acetylene in the carburizing cycle [3], JH Corp.[5] found that carburizing below 7.5 torr produced good case depths and decreased soot significantly [9]. Since these

discoveries, several researchers have shown that LPC with acetylene provides good carburizing case depths on parts with various geometries [13-15].

Solar's carburizing development

The Solar Carburizing Team conducted research for a unique carburizing process in a lab scale retort furnace built on site (see lead photo). A schematic of the system is shown in Fig. 1. Certain requirements are critical for vacuum carburizing. For instance, all gases used in the process must be of the highest purity (99.9%), because impurities found in lower grade gases contribute to soot formation and product contamination. Before each run, it is essential to bubble test all gas feed lines to ensure they are leak free. In addition, vacuum furnace integrity must have a leak rate of 5 microns (Hg) or less per hour.

Using established data [16] based on a solution of *Fick's Law of Diffusion* (Eq 1, Fig. 2) and the known ratio R, which relates diffuse time to boost time (Eq 2, Fig. 3), carburizing cycles were developed for a carburizing temperature of 1750°F (955°C). All cycles were designed to give case depths in the range of 0.035 in. (~1 mm), a surface carbon content of approximately 0.8% and Rockwell hardness values in the low to mid 60s, with a total carburizing time of two hours or less.

$$D = K\sqrt{t} \quad (\text{Eq 1})$$

where D is carburized case depth, K is a constant that depends on temperature and t is total process time (boost + diffuse)

$$R = t_d/t_b \quad (\text{Eq 2})$$

where t_d is diffusion time and t_b is boost time.

Several studies of propane, ethylene and acetylene as carburizing gases were carefully evaluated. Although propane could be used successfully at pressures as low as 3 torr, acetylene gave better overall results. Given previous documentation on the use of acetylene in LPC, new directions were sought, which would allow its use in this process development study.

In earlier studies, only previously sand-blasted parts gave uniform carburization. Since a machining step would not be cost effective, an alternative precleaning step using hydrogen was investigated. It is well known that hydrogen reduction of oxide impurities provides a cleaner, more open surface for carburization [17]. The data in Table 1 shows that the pretreatment with hydrogen at a partial pressure (4.5 torr) reduces heavy oxides and prepares the surface for carbon pick-up. (Note that preoxidation was used as a test and is not being advocated as part of the process.)

The low carburizing gas pressures required to prevent soot issues were insufficient and led to nonuniform case depths. Increasing the pressure in the furnace without changing the pressure of the carburizing gas was achieved by adding hydrogen or nitrogen during the boost. The preferred method involved a hydrogenated (4.5 torr H_2) pre-heat, a boost cycle using a gas mixture of approximately 12.5% acetylene and 87.5% of a carrier gas (N_2 or H_2) for a total pressure



Fig. 5. Production furnace refitted with carburizing controls for vacuum carburization

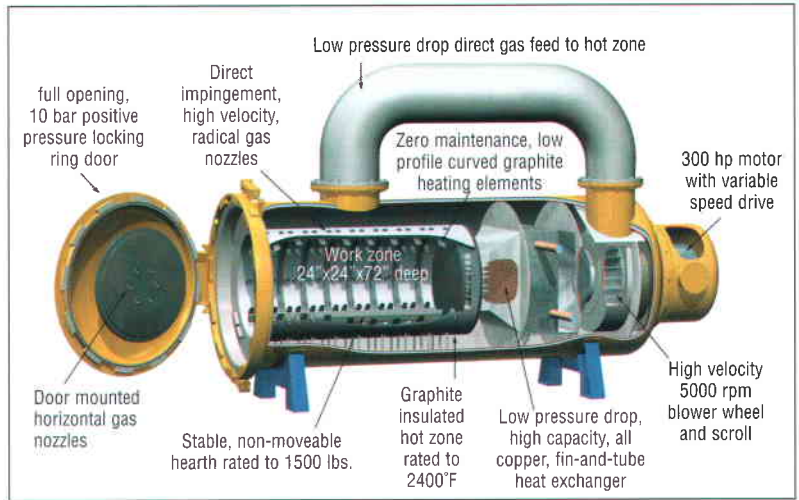


Fig. 6. Details of the Solar low-torr range vacuum carburizing chamber and high-speed gas quench system

How to choose gas flow and type of cycle

The knowledge of established data provided a framework for a basic process with respect to temperature, time and case depth. Pressures were chosen based on cleanliness.

However, a method for calculating the flow rate of the carburizing gas for a predetermined carbon level was unavailable. A relationship was developed between the surface area of the parts to be carburized and the flow rate of the carburizing gas

[19]. Using data obtained from a residual gas analyzer (RGA) collected during the carburizing runs (Fig. 4) and taking into account surface area, change in carbon content of the parts, boost time, the density of iron and the amount of carbon available from acety-

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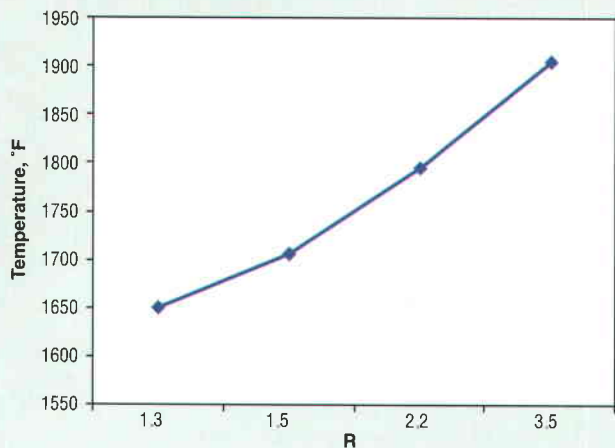


Fig. 3. Relationship between temperature and R value for a carburizing case depth of 0.035 in.

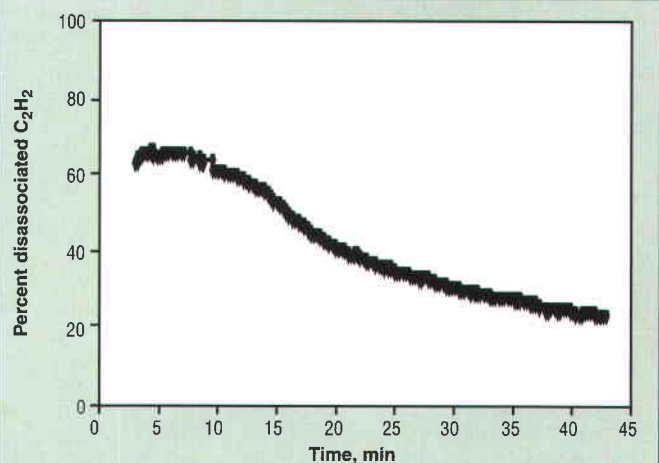


Fig. 4. Relationship between fraction of acetylene dissociated and time

of 8 torr, followed by diffusion in a partial pressure of 10 torr nitrogen. Each experiment was designed as a continuous cycle with $R = 2$, temperature = 1750 °F and $t_{total} = 2$ hours. The results are listed in Table 2.

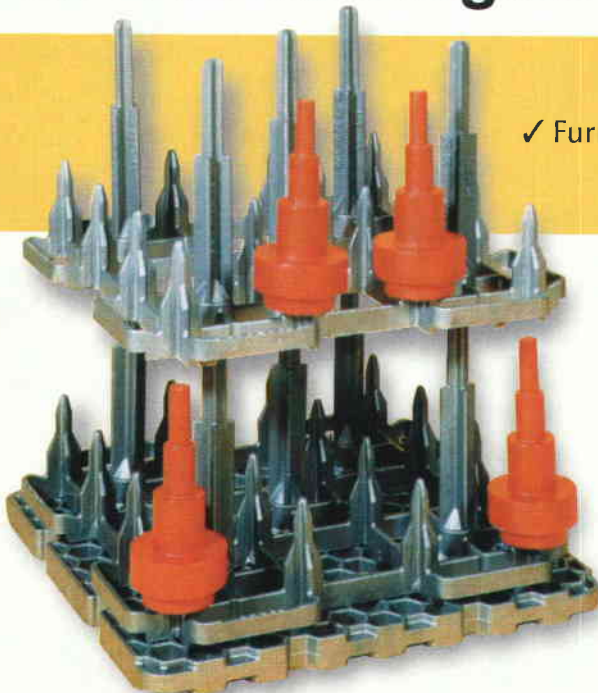
Although conceived here independently, others recently have reported the use of gas mixtures to improve vacuum carburizing with acetylene [13,14,18]. The combination of a hydrogen pretreatment, followed by a hydro-

gen or a nitrogen mixed carburizing boost has not been previously reported. The only pre-treat study involving LPC actually suggested that hydrogen in the boost would be detrimental to the carburizing process [15].

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Table 1 Beneficial effect of hydrogen pretreatment on oxidized material

Specimen	Oxidation temp., °F (°C)	Surface case depth (0.8%C), in.	Hole case depth, in.
A100	No preoxidation	0.030	0.026
A300	800 (430)	0.030	0.025
A310	600 (315)	0.031	0.022
A320	400 (200)	0.028	0.024

Table 2 Comparison of hydrogen and nitrogen in the boost

Specimen	Diluting gas	Surface case depth (0.8%C), in.	Hole case depth, in.
A330	H ₂	0.030	0.027
A340	H ₂	0.032	0.026
A350	H ₂	0.030	0.027
A360	N ₂	0.030	0.022
A370	N ₂	0.030	0.018
A380	N ₂	0.030	0.026

lene over time, a theoretical flow rate was calculated using the following equation.

$$\text{Flow rate}_{\text{theor.}} = \text{carbon required}/t_{\text{boost}} \times (0.0011g_{\text{carbon}}/cc_{\text{acetylene}}) \quad (\text{Eq 3})$$

The theoretical calculation assumes complete dissociation of acetylene. Since complete dissociation does not occur, a correction for the fraction that does dissociate must be included. This dissociation value must be determined for each furnace system. The theoretical calculation can be used to provide a minimum flow rate required to achieve a given case depth and carbon content.

Most of the current carburizing techniques favor a sequential boost/diffuse cycling [20] process. The sequential method involves a series of short cycles in which the first step is a carburizing step and the second is a soaking step, which allows the adsorbed carbon to diffuse into the metal in the absence of a high-carbon atmosphere. The reasoning behind several short successive boost/diffuse cycles stems from the need to provide effective case depths and ensure the carburization of deep recesses. It is believed that the successive cycles provide a means to give laminar flow into blind holes thereby improving carburization [21].

The carburizing process developed in this study is accomplished using a continuous cycle involving only one boost and one diffusion step. With the application of the flow rate calculation (Eq 3) the amount of carburizing gas can be controlled based on the size of the work. An alternative process combines a continuous boost cycle in which the flow rate of the carburizing gas is decreased

in a stepwise process throughout the boost. RGA data for this method suggests that the dissociation factor of acetylene actually increased as the flow rate decreased.

Scaling up to a production stage

To gain a greater understanding of how a production furnace will perform, carburizing controls were incorporated on an existing furnace. The production furnace shown in Fig. 5 was refitted with a bypass vacuum pumping line, mass flow controllers, absolute vacuum gauge and nozzles for introduction of hydrocarbon gases. This furnace provides a scale up of 20:1 from the lab retort furnace used to develop a carburizing process. Adjustments were needed in nozzle design and size to ensure maximum gas flow and appropriate gas distribution throughout the furnace.

Advantages of using this furnace for vacuum carburizing development are twofold. The first is to repeat the success of lab-scale runs at production levels, and the second is to introduce an approach to carburize parts in situ; that is, carburizing and quenching parts in a single chamber. This is a unique capability since most current vacuum carburizing furnaces require multiple chambers to process parts. The simplified engineering reduces processing time and enhances furnace integrity since fewer moving parts offer a more durable and smoother operating system.

The ultimate goal of the development project is the introduction of a new Solar vacuum carburizing chamber (Fig. 6) fitted with the latest control systems to regulate a unique low-torr range process. In addition the furnace will have the capa-

bility to include 10 bar gas quenching at high gas velocities superior to existing furnaces. **IH**

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The Solar Carburizing Research Team included Trevor Jones, Don Jordan, Harry W. Antes, Ph.D., William R. Jones, Robert Hill and Virginia M. Osterman, Ph.D.

For more information: Virginia Osterman, is Technical Director, Solar Atmospheres Inc., 1969 Clearview Rd., Souderton, PA 18964-0476; tel: 215-721-1502; fax: 215-723-6460; e-mail: vmo@solaratm.com; Internet: www.solaratm.com

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