

# Copper Evaporation During Low Pressure Carburizing

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## Abstract

Low pressure carburizing (LPC) in a vacuum furnace is increasingly the preferred method of case hardening for aerospace gears, and acetylene is often one of the gases used in the process. Selective case hardening is common with gears, where certain sections of a part are “stopped off” or “masked” to prevent carburization at those locations. For aerospace parts, the masking used is typically copper electroplating. The low pressures and high temperatures used in LPC lead to copper evaporation, which contaminates the vacuum furnace hot zone and components. In a worst-case scenario, deposited copper can lead to short-circuiting of power feedthroughs. This study looks at the effect of vacuum and partial pressure gases on copper evaporation and its application in production processes.

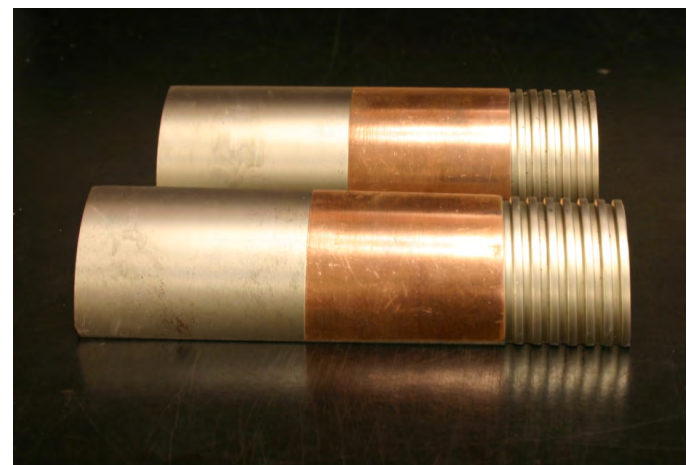
## Introduction

Low pressure carburizing (LPC) in a vacuum furnace is increasingly the preferred method of case hardening aerospace gears, with some alloys like Ferrium C61 and C64 have been designed specifically for LPC. Several industry processes use acetylene as the principal carburizing gas. Each LPC cycle has a series of carburizing boost steps, immediately followed by an equal number of diffusion steps. A computerized controlled process using gas mass flow controllers allows for rapid buildup of carbon at the surface of parts during the boost stage. The time for each diffusion step increases throughout the cycle, as a uniform case is formed, free of large blocky and networked carbides. The length and number of each step is determined by the material type, temperature, and case depth requirement.

Heating to the carburizing temperature with LPC can be performed in vacuum or using a partial pressure gas. The pressures used in LPC processing typically ranges from 0.1 Torr (13.3 Pa) to 11 Torr (1.5kPa) (1). With some of the high alloy steels specified for aerospace parts, hydrogen is often the preferred partial pressure gas to use during heat-up. Hydrogen reduces passive oxide films at the surface, readily activating it for efficient and consistent carburizing (no preoxidation treatment is required). In comparison to the boost stages that use patented or proprietary acetylene/carrier gas mixtures for short periods, the total time for the diffusion stages is

relatively long. Like the heat-up stage, the diffusion stage can be done in vacuum or using a partial pressure gas.

Selective case hardening is common with gears, where certain sections of a part are “stopped off” or “masked” to prevent carburization at those locations. The intent is to have the masked areas remain the same carbon weight percentage as the core base metal (Figure 1). For aerospace parts, the masking used is typically copper electroplating, which in instances covers much of the surface area of a part. The low pressures and high temperatures used in LPC can lead to copper evaporation during the diffusion steps. Copper vapor then condenses on colder components in the hot zone, such as gas nozzles and ceramic insulators. In a worst-case scenario, copper deposits have caused arcing and melting of power feedthroughs damaging the furnace and in some instances the parts (Figure 2).



*Figure 1: Copper plating shields the section from case hardening*



Figure 2: Example of copper and soot-like acetylene by-product build up on power feedthroughs

The use of partial pressure gas helps suppress evaporation is well-established in the heat treating industry (2,3). This present study investigates the effect of temperature, pressure, and carrier gas species on the amount of copper evaporation that occurs from copper foil test samples.

### Experimental Details

Oxygen-free high conductivity copper foil, 0.002" thick, was cut into 2 inch square coupons. Three coupons were prepared for each test process. Just prior to placing the coupons into the heat treat furnace, they were briefly immersed in a solution of Almco 2510™ (citric acid solution) to remove surface oxidation and contaminants. They were rinsed in distilled

water, methanol, and dried with compressed nitrogen gas. Each sample was weighed using an analytical balance that records up to four decimal points in grams. Upon removing coupons from the heat treat furnace they were immediately reweighed using the same scale. The measured weight loss indicates the capability of the gas/partial pressure combination to suppress copper evaporation.

A graphite insulated vacuum furnace with a cylindrical, vertical hot zone, 10" diameter x 18" high, was used for this study. The three coupons were hung on separate stainless steel wires attached to the lid of the furnace. All cycles began after an initial pump down to  $1 \times 10^{-4}$  Torr ( $1.3 \times 10^{-2}$  Pa) to assure vacuum integrity. Three temperatures were chosen for testing: 1575°F (857°C), 1700°F (927°C), and 1825°F (996°C), to cover the common LPC temperature range. Four atmosphere types were used for processing: vacuum at  $<1 \times 10^{-3}$  Torr ( $<13.3 \times 10^{-2}$  Pa), partial pressure nitrogen, argon, and hydrogen, at 0.25 Torr (33.3 Pa), 2.5 Torr (333.3 Pa), and 10 Torr (1.3 kPa). The partial pressure was set prior to heating and maintained during the 6-hour hold time at temperature. All samples were cooled in nitrogen at 625 Torr (83.3kPa)

A temperature uniformity survey (TUS) was performed in a horizontal graphite insulated LPC production furnace with a hot zone 24" (0.6 m) x 24" (0.6 m) x 72" (1.8 m). Following the AMS2750E protocol, a nine point TUS was performed for each gas and pressure to compare with the baseline data at pressures less than  $1 \times 10^{-3}$  Torr ( $<13.3 \times 10^{-2}$  Pa).

| Table 1: Evaporation rate (gram/cm <sup>2</sup> minute) vs Pressure |                |          |                |                |          |                |                |          |                |
|---|----------------|----------|----------------|----------------|----------|----------------|----------------|----------|----------------|
| Pressure  | 1575°F         |          |                | 1700°F         |          |                | 1825°F         |          |                |
| Torr  | N <sub>2</sub> | Ar       | H <sub>2</sub> | N <sub>2</sub> | Ar       | H <sub>2</sub> | N <sub>2</sub> | Ar       | H <sub>2</sub> |
| 0.001   | 4.95E-07       | 4.95E-07 | 4.95E-07       | 3.79E-06       | 3.79E-06 | 3.79E-06       | 1.93E-05       | 1.93E-05 | 1.93E-05       |
| 0.250   | 1.47E-07       | 1.31E-07 | 2.28E-07       | 1.14E-06       | 8.68E-07 | 3.06E-06       | 5.26E-06       | 4.43E-06 | 1.70E-05       |
| 2.5   | 3.58E-08       | 4.30E-08 | 1.22E-07       | 1.36E-07       | 1.51E-07 | 8.29E-07       | 7.31E-07       | 6.93E-07 | 4.70E-06       |
| 10  | 2.51E-08       | 1.43E-08 | 5.70E-08       | 2.50E-08       | 5.38E-08 | 3.19E-07       | 2.79E-07       | 1.99E-07 | 1.61E-06       |

Table 1: Evaporation Rate vs. Pressure

## Results

The average rate of copper evaporation for each temperature, gas species, and pressure is listed in Table 1. The evaporation rates versus pressure curves in Figure 3 are representative of all three temperatures studied. Copper evaporation drops off sharply in nitrogen and argon, even at pressures as low as 0.25 Torr (33.3 Pa). Argon is slightly more suppressive than nitrogen; however, hydrogen is considerably less effective at reducing metal vapor losses at the temperatures studied. The data reveals that copper evaporation decreases with a decrease in temperature and an increase in pressure. Figure 3 also shows a flattening of the curve as pressure increases indicating that suppression slows down with continued increase in pressure.

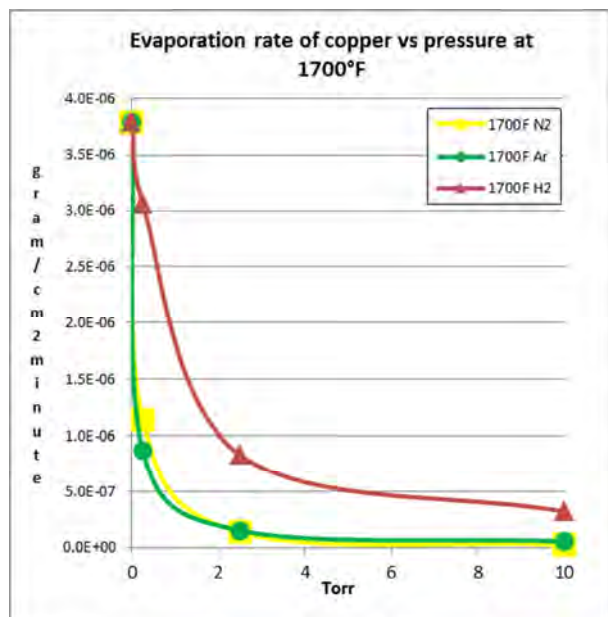


Figure 3: Evaporation rate of Copper vs pressure at 1700 F

Temperature data reveals that the uniformity of the furnace degrades as temperature increases (figure 4). Compared to high vacuum,  $<1 \times 10^{-3}$  Torr ( $13.3 \times 10^{-2}$  Pa), temperature uniformity improved with all gases at 0.250 Torr (33.3 Pa) or 2.5 Torr (333.3 Pa). However, at 10 Torr (1.3 kPa), for all gases, uniformity was inferior to high vacuum. Generally, the work thermocouple temperatures decreased as the pressure increased in the furnace. This is not unexpected as the gas flow rates and convective losses are greater as the pressure increases.

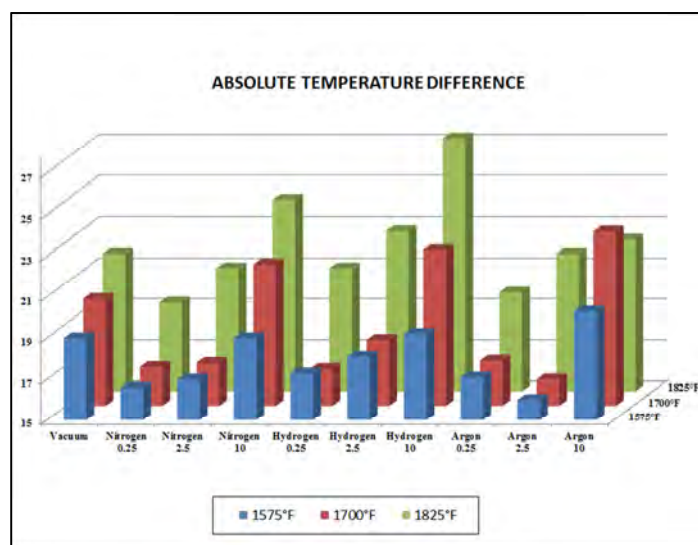


Figure 4: Temperature Uniformity Study

## Conclusions

The stringent temperature uniformity requirements of the aerospace industry play an important role in selecting the partial pressure gas to minimize evaporation. Nitrogen at 2.5 Torr is the most economic and effective gas for minimizing copper evaporation. Argon, was marginally more effective at suppressing evaporation compared to nitrogen, however, nitrogen enhanced the temperature uniformity compared to argon. The use of nitrogen gas is increasingly important to use during longer time diffusion steps as the LPC temperature increases to minimize copper evaporation and copper contamination of furnace components.

## References

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- [2] Herring, Daniel, Using Partial Pressure in Vacuum, Industrial Heating; November 2005
- [3] Jones, William, Partial Pressure Vacuum Processing – Part I and II, Industrial Heating, September and October 1997