Gassing Up To Get The Right Atmosphere

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ABSTRACT
In a continuous sintering furnace, one of the most important variables in achieving a successful process is control of the atmosphere. From the initial installation and start-up of the furnace to its maintenance throughout its life cycle, the process atmosphere demands an attentive eye in order to produce powder metal parts that are well sintered, meet the specification and have no defects. Starting with the source of the atmosphere gases and working through the components of the furnace, the atmosphere integrity must be verified before any parts can be processed. As the furnace ages, issues will arise that affect the integrity of the atmosphere and ultimately the quality of the product. Continual preventative maintenance and troubleshooting techniques will maintain the integrity of the process atmosphere. An understanding of the causes, diagnostic techniques and solutions to the many atmosphere problems that may compromise the atmosphere integrity, will enable a furnace operator to run an efficient and profitable process.

This paper focuses on the important parameters of the furnace and its atmosphere that affect the integrity of the sintering process, and gives guidance on how to approach and solve the problems they may cause.

INTRODUCTION
The quality of the furnace atmosphere control will determine the quality of the sintering process. There are many parameters in a sintering furnace that could affect the process but some of them may be impossible to control. This makes the sintering process quite complicated in terms of process control and troubleshooting. In this complicated environment, process issues are quite likely to be encountered. Many problems occur during the sintering process but the most important and most frequent are associated with improper or incomplete removal of lubrication, in other words delubing.

In order to achieve the best quality from a sintering process, the furnace and the furnace atmosphere have to supply crucial conditions so that the process produces a good bonding between metal particles. There should be minimum decarburisation in the core, minimum oxide content, round pores between particles, tight control of surface carbon, good dimensional control and consistent quality.

The following furnace parameters play a vital role in process quality and need to be closely controlled by the furnace operator in order to obtain the best product quality. These are: furnace temperature profile, belt speed, belt loading capacity, furnace door opening height, furnace cooling rate, furnace curtain integrity, furnace atmosphere volume and composition, atmosphere location and direction, P/M part composition and external influences such as environmental effects.
FURNACE ATMOSPHERE FUNCTION

The first step in establishing and maintaining the process atmosphere in a powder metal sintering furnace is to understand the function of the furnace atmosphere. The composition of a sintering process atmosphere may have to vary through the furnace to be able to execute different metallurgical functions. Therefore furnace atmosphere optimisation becomes a very important aspect of achieving components of the highest quality. Atmosphere zoning techniques have been designed and developed to meet this purpose for many years. It is possible to reduce or optimise the atmosphere cost in a sintering process by proper control of the functions, including flow control and control of the mixture to meet different thermal and chemical requirements. A typical sintering process requires a number of basic functions to be able to produce sintered components. Figure-1 illustrates a typical sintering furnace and the furnace atmosphere distribution.

![Figure – 1 Sketch of a typical sintering furnace and the atmosphere profile in furnace zones](image)

a. *Pre-Heat Zone*: This section is designed to prepare the P/M parts for the high heat sintering zone by pre-heating and removing lubricant. Therefore the furnace atmosphere should produce oxidising conditions for delubrication. This simply requires an atmosphere with a high dew point and enough flow to remove burnt-out lubricant. As Figure – 1 shows, this zone’s high dew point gives it a high moisture content, helping the delubrication process.

b. *Hot Zone*: The sintering process requires a strongly reducing atmosphere to remove oxides and help the powder metal particles to bond. The moisture content or dew point must be low enough to reduce metal oxides through the powerful reducing effect of hydrogen gas. Since this zone is almost moisture free, it is essential to remove lubricants prior to the sintering operation in the high heat zone. Any hydrocarbon remaining in this zone will always decompose and the carbon will be deposited as soot on metal surfaces, the furnace components and/or P/M parts.

c. *Cooling Zone*: The main function of the atmosphere in the cooling zone is to cool down the components in a protective medium. Protective cooling requires a slightly reducing
atmosphere, especially in the pre-cooling section where the sintered components are still at a very high temperature. This zone is also used for sinter hardening processes where forced cooling is applied for rapid cooling. Figure – 1 shows the temperature profile of the cooling zone which suggests that pre-cooling would require a higher dew point while the rest of the cooling zone will require only a nitrogen atmosphere to maintain the furnace atmosphere distribution and integrity.

**FURNACE ATMOSPHERE PROFILING**

Atmosphere profiling is an important method to establish furnace data as well as understand possible problems. This method helps furnace operators to collect baseline data and most importantly gives them a starting point for troubleshooting. In order to run an atmosphere profile, some basic equipment is necessary. It includes a recording device, thermocouple fixtures and profile thermocouples in adequate lengths, as sometimes they need to be run through the furnace.

The detailed procedure is not within the scope of this paper but the basic steps are outlined below.

a. Calibrating of the furnace controllers
b. Setting the proper conditions for the furnace:
   i. belt speed
   ii. temperature settings
   iii. atmosphere flows
   iv. furnace load.
c. Marking off the furnace and the profile thermocouples
d. Starting the atmosphere profiling
e. Recording the information.

It is possible to achieve two important categories of information by means of Atmosphere Profiling. The first is the baseline data, consisting of baseline conditions for all furnaces including old and new ones. This information can be used to compare different production schemes and different furnace conditions. It enables the furnace operator to check the dew point and oxygen content of all incoming gases. Temperature profile data will be available to control and maintain furnace atmosphere functions throughout the furnace.

Secondly, the trouble-shooter will be able to analyse the furnace atmosphere conditions fully and these two types of information can be linked together to reveal a complete picture of the process, the furnace and the furnace atmosphere as a whole.

Figure 2 show a picture of a Sample Cart used by Abbott Furnace in their Furnace Atmosphere Profiling. The main functions of the unit are:

a. Temperature profiling
b. Atmosphere balancing
c. Atmosphere composition
d. Atmosphere cost optimisation.
Some of the equipment in this unit is:

a. Temperature measurement  
b. Hydrogen analyzer  
c. Oxygen analyzer  
d. Dew Point analyzer  
e. Independent read-outs  
f. Data logging.

**PROBLEMS DURING SINTERING**

As described earlier, the sintering process requires a highly complex furnace atmosphere in order to carry out different functions required, which can lead to many issues during sintering. The following problems are the most common encountered during the process and could be solved by understanding the possible reasons described below.

1. *Frosted parts*: Frosted parts will have a dull and matte finish on their previously shiny surfaces as can be seen in Figure - 3. This surface contamination also decreases the hardness. This problem is the one of many problems related to lubricant and the pre-heat zone. Possible reasons are:
   a. The parts are oxidised in the pre-heat zone and the additional oxide layer is then reduced in the high-heat zone  
   b. The oxygen level might be too high in the pre-heat zone  
   c. Air might be entering from the front end  
   d. There might be cracks or holes in the pre-heat muffle  
   e. A downdraft on the front stack may be causing the atmosphere to float unstably and push the flow out of the furnace.
2. **Sooting**: This is almost always caused by incorrect or incomplete delubrication. Sooting may occur when hydrocarbon vapours from the lubricant thermally decompose. There are different types of soot, with different causes and solutions; these are:

   a. **Staining**: This appears as black stain, as seen in Figure - 4, and occurs on all surfaces of the work piece. It cannot be removed easily. It happens when the dew point is too low in the pre-heat zone, or if the pre-heat zone is too short for the temperature and speed settings. Incorrect temperature and belt settings may lead to staining, which may also occur if the belt load is too heavy.

   b. **Black spot**: Black Spot looks like black snow, and occurs on the top surface as can be seen in Figure – 5 below. It occurs if the preheat dew point is too low and/or the forward flow of the furnace atmosphere and its velocity are insufficient. Drafts or improper design of the stack may make the atmosphere unstable. The belt speed may be too fast, not allowing enough time for delubrication.
c. **Blistering/Rippling:** This appears as blisters or ripples on the part. Sharp edges can crumble. This is the most extreme case of sooting and it is commonly seen in nickel-containing and high-density parts. The reasons may be too rapid heating up in the reheat, or the belt speed may be too fast or the belt load too heavy.

3. **Oxidation:** This is a common problem in all heat treatment process applications and is caused by air, moisture, or carbon dioxide. The problem is sometimes caused by one or more different contaminants in the furnace atmosphere. Heavy oxidation sometimes occurs in sintering applications when oxygen or water enters the high heat zone making the part black and possibly with a scale. The colour of the oxidation indicates the temperature the part is exposed to in the oxidising atmosphere. The following Figure – 6 shows the colour scales and associated temperatures.

![Figure – 6 Oxidation Colours – temperature is in Degrees Celsius](image)

Figure – 5 Black Spot
4. **Surface Oxidation**: This occurs in cooling zones if air enters from the exit end. Air can also enter from loose flanges between various cooling zones. This type of oxidation is generally just a surface effect and does not affect the internal properties. The colour of the oxidation will also vary with the part weight. This oxidation may occur on just one side or on the leading edge.

5. **Belt Oxidation**: Most sintering furnaces have stainless steel belts that contain chromium. Oxidation may occur in the pre-heat zone. The belt looks dark and the parts bright.

6. **Decarburisation**: Decarburisation may be simply described as loss of carbon from the surface of the part. During sintering, decarburisation may also occur in the core. Both these reduce hardness. Possible reasons may be air infiltration into the preheat and / or high heat zones. The dew point or CO₂ might be too high in the hot zone or CO may be too low.

7. **Inadequate sintering**: This can be caused by many factors, such as too low a sintering temperature, or insufficient hydrogen levels to achieve enough reducing power. The dew point might be too high in the high heat zone, or the belt speed too fast.

**DELUBRATION EQUIPMENT**

The above description shows clearly that most of the problems described in this paper are caused by improper or incomplete delubration. Different types of equipment and solutions to improve the delubration process are commercially available. The trade names and basic information about the systems are given below, however more detailed information can be obtained from the manufacturer*. Some of these systems are:

1. **LBT** – **Lubricant Burner Technology**: The equipment is added to front of the furnace. Air is injected to combust the unreacted combustibles in the furnace atmosphere.
2. **Zone 0**: This is also added to the front of the furnace. Heat coming from the furnace is actually used to preheat the parts.
3. **Bubblers**: This system is essentially just humidifying the nitrogen gas by passing it through bubblers or in other words through a specially designed water container to increase its dew point. It is also called “Wet Nitrogen” and the high dew point assists in burning off the lubricants.
4. **QDP**: This is the abbreviation and trade name for “Quality Delube Process”
5. **ADS**: This is the abbreviation and trade name for “Accelerated Delube System”

**DIAGNOSING SINTERING PROBLEMS**

Troubleshooting of a sintering process requires experience and relies mostly on knowledge of previous practice. However there are some simple methods that can be easily applied by furnace operators to discover the basic sources of the problem. Two of these methods are briefly described below.

1. **Copper Infiltration Test.**
   a. Place a piece of copper on a green part and another on a previously sintered part.
   b. Send both parts through the furnace under the same conditions that caused the problem.
   c. The results will indicate the following problems:
      i. If the copper does not melt, the sintering temperature is too low.
      ii. If the copper melts but flows only into the sintered part, there are delubing problems in the pre-heat zone.
      iii. If the copper melts but does not infiltrate either part, then the reducing power of the atmosphere in high heat zone is insufficient.
      iv. If the copper infiltrates both parts but they both appear oxidised, then there may be a leak in the cooling zone.
2. **Copper / Steel / Stainless Steel Test**: This test determines whether oxidation occurring in cooling section is caused by an air or water leak. The procedure is as follows:

   a. Reduce the furnace temperature to 1900 °F (1030 °C)
   b. Place clean strips of copper, steel and stainless steel on the belt
   c. Run them through the furnace
   d. The results will indicate the following problems:
      i. If copper and steel are both oxidised, then there is an air leak
      ii. If only the steel is oxidised, then there is a water leak
      e. Stainless steel reveals smaller leaks since it is more sensitive to an air leak than steel.

**CONCLUSION**

Establishing good baseline conditions that include atmosphere and temperature profiles helps to maintain sintering furnace conditions. It is also advisable to perform routine checks of the profiles to help maintain furnace integrity. When a problem does occur, the furnace operator should try to isolate it with some of the techniques described in this paper.

Sintering determines the final mechanical properties of P/M parts so it is a very important step in the production. Quality and costs are affected by fast troubleshooting, and a consistent level of quality is only possible by controlling the furnace atmosphere closely and maintaining the furnace integrity. Furnace atmosphere management and furnace troubleshooting experience is very important in maintaining both quality and productivity. The companies of both authors have significant resources available to support their customers worldwide.

**REFERENCES**

5. ASM Source Book on Powder Metallurgy, 1979