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ATMOSPHERE CONTROL SYSTEMS - FOR HIGHER QUALITY AND CONSISTENCY[1]

Pierre Forêt[2]
Christoph Laumen[3]
Sylvano Bigio[4]

Abstract

Atmosphere control in heat treatment furnaces is one of the most important variables in a successful heat treatment process. This statement becomes even more weight when the range of the materials and parts treated is varying so that relying on experiences can become a bold venture. Today the consumers expect goods of good value with a stable and long lasting controlled quality. In heat treatment that has been attempted by strict control of the ingoing materials and keeping the parameters like temperature, atmosphere composition and flow on a predefined level. However there are influencing variables that cannot be scoped with by that approach. Here the on-line atmosphere control can be used for a wide range of annealing, carburizing, nitriding and nitrocarburising processes as well as for sintering to assure a uniform result without surprises. The presented paper shows tailor-made concepts of atmosphere control systems for a variation of different HT processes and illustrates the technological results and cost benefits that can be achieved.

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2 *The Linde Group, Unterschleissheim, Germany. pierre.foret@linde-gas.com.*
3 *The Linde Group, Unterschleissheim, Germany. christoph.laumen@linde-gas.com.*
4 *Linde Gas, Sao Paulo, Brazil. Sylvano.Bigio@br.aga.com.*

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INTRODUCTION

The inexorable pressure, particularly from the automotive industry, for higher quality and consistency in the high volume production of parts has inevitably led to increasingly sophisticated control systems for every heat treatment process.

Since the 1970s, when industrial gas suppliers entered the heat-treat market with the aim to replace gas generators, they have increasingly served not only as suppliers of gases but also technologies, services and equipment. They also are increasingly partnering with others suppliers including furnace equipment and control equipment manufacturers. Figure 1 illustrates the atmosphere system with gas supply, atmosphere control, remote supervision and service assistance.

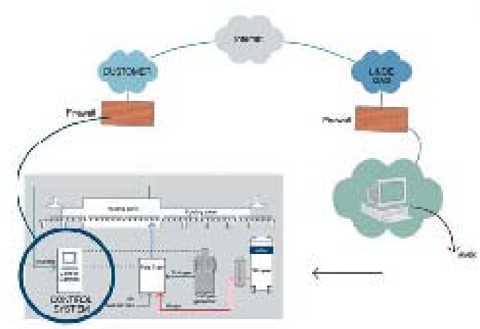

Figure 1. Atmosphere system.

Using advanced atmosphere control as a tool in surface engineering has made it possible to obtain dramatic improvements in both product value and process efficiency, and such improvements will be further realized in the future. Environmental sustainability, product quality, and process efficiency will be future development drivers.

The quality and cost improvement due to atmosphere control system can be explained with Figure 2. The gas flow in a furnace without atmosphere control system does not take into account parameters like part dimension and part quality requirements (top graphic). An atmosphere control system will adapt the gas flow to the production intensity and parts parameters (bottom graphic). This results into gas savings and quality control.

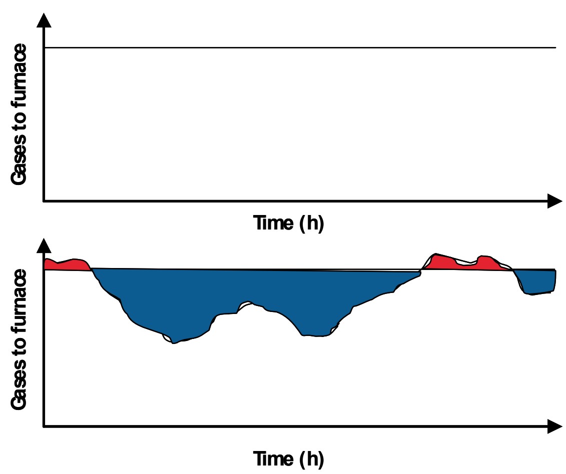

Figure 2. Achievable gas savings by applying Atmosphere Control Systems (ACS).

Following are some examples of developments in the controlled atmosphere area that will meet requirements like environmental sustainability, product quality, and process efficiency (Table 1).

Table 1. The impact of proper use of controlled atmosphere

Part	**Heat treat process**	**Way of optimizing**	**Impact**
Wind screen wiper	Nitrocarburising	Replacing multi component design	62% weight reduction Elimination of production process steps
Gear	Carburizing	Optimizing carburizing recipe	Reduced carburizing time and improved quality
Carbon steel tube	Annealing	Ensure freedom from decarburizing	Increased strength and improved quality
Steel strip coil	Annealing	Hydrogen as atmosphere	Clean surfaces Process time reduction up to 50%
Aluminium heat exchanger	Brazing	Atmosphere control	Reduced flux consumption Joint quality

Atmosphere Carbon Activity and Carbon Potential

A key to atmosphere control system is the carbon activity or carbon potential.
Atmosphere carbon potential is preferably controlled by oxygen probe or CO_2 infrared gas analysis. This is based on the assumption of gas equilibrium in the water gas reaction:

$$CO + H_2O \Longleftrightarrow CO_2 + H_2$$

The carbon activities (a_C) in the furnace gas can be expressed in the following ways where K is the equilibrium constant and P the partial pressure of the gas:

$$a_C = K_x \, . \, P^2_{CO}/P_{CO2}$$

$$a_C = K_y \, . \, P_{CO}/P^{1/2}_{O2}$$

The carbon activity of the gas can be controlled by controlling the CO_2 content or the O_2 content, provided that P_{CO} is known. CO_2 control with infrared (IR) gas analyzer and O2 control with an oxygen probe are practical ways to do this.
In practice, the concept of “carbon potential” is used instead of carbon activity. The carbon potential of a furnace atmosphere is equal to the carbon content that pure iron would have in equilibrium with the gas.

Annealing

Introduction and basic principles

Annealing is a heat treatment for steels and non-ferrous materials in order to change the microstructure and the properties of the material. Some examples of annealing processes are:

- Recrystallization, for tubes, plates, strips and wires in order to form new undeformed crystal grains and increase ductility

- Stress relieving, for welded, cold deformed or machined parts to relieve stresses in order to avoid warping or distortions when in use or at later production stages
- Solution annealing and precipitation hardening (PH), on Cr/Ni-steels to bring alloying element into solution to increase corrosion resistance or on PH and maraging steels to increase strength and toughness
- Isothermal annealing for low alloy steels to produce a ferritic/pearlitic microstructure for good machinability
- Soft Annealing for high carbon steels in order to form round carbides more easy to machine
- Normalizing of tubes and strips to improve machinability by refining and uniformising the grains size

In a typical annealing process, the metal is heated above a critical temperature then slowly cooled to room temperature to obtain a softer and less distorted material structure. Unwanted reactions with residual lubricants can occur between the surface and the surrounding atmosphere. Proper selection and control of the annealing atmosphere is critical to produce a bright, high quality surface.

The main requirement on the furnace atmosphere is that it should be neutral with respect to the metal. Neutrality implies that the atmosphere composition must be controlled so that there are no reactions between the metal surface and the three elements oxygen (O), to avoid oxidation, carbon (C) to eliminate decarburization of carbon steels, and nitrogen (N), to eliminate nitrogen pick up in stainless steels

Atmospheres Systems

CARBOFLEX® and HYDROFLEX® are the name of two families of atmosphere systems developed by Linde Gas with the capability of controlling carbon and /or oxygen potential in annealing processes. An advantage of this atmosphere control system is the flexibility to mix the incoming gas streams into widely varying ratios to produce atmospheres specifically tailored to the alloy, product mix and location within the furnace.

CARBOFLEX® is designed for annealing of carbon steel tubes and carbon containing atmospheres based on nitrogen (N_2), see Figure 3.

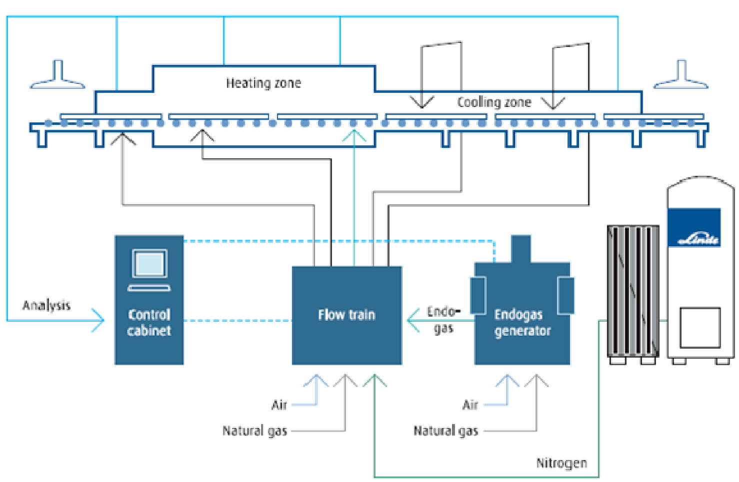

Figure 3. The CARBOFLEX® gas system with nitrogen and external endogas supply.

HYDROFLEX® is designed for annealing of stainless steel and copper tubes with atmospheres based on nitrogen (N_2) and containing hydrogen (H_2), see Figure 4.

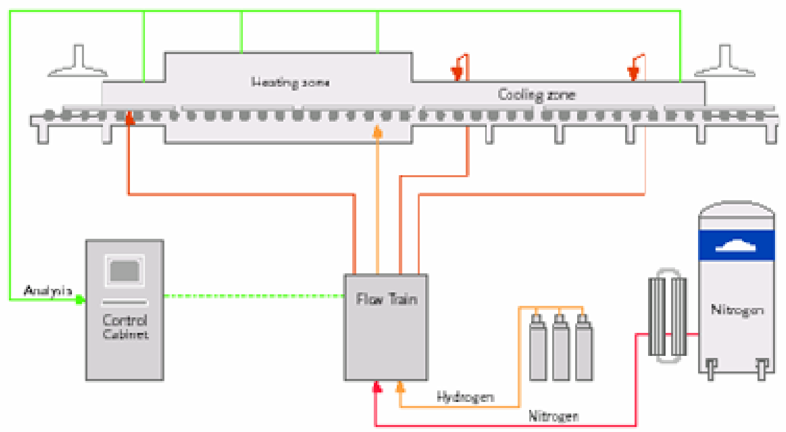

Figure 4. HYDROFLEX® nitrogen/hydrogen system for bright annealing.

Decarburization

Decarburization is usually caused by a reaction between the carbon dissolved in the steel (C_{Fe}) and oxygen or an oxidizing species in the surrounding atmosphere. The following reactions are the main causes to explain decarburization:

$$C_{Fe} + O_2 \rightarrow 2CO$$

$$C_{Fe} + H_2O \rightarrow CO + H_2$$

$$C_{Fe} + CO_2 \rightarrow 2CO$$

Carbon control enables to minimize decarburization when oxidizing species are present. A control system, such as the CARBOFLEX® system, can balance the carbon potential of the atmosphere with the carbon in the steel and eliminate decarburization.

Carburizing

Introduction and basic principles

The principle of carburizing is to apply a high-carbon gas on a low-carbon material at a temperature where the steel is austenitic, typically in the temperature range of 820°C-950°C. The outer surface or case will have higher carbon content than the original material. The iron or steel is cooled rapidly by quenching in order to keep the higher carbon content on the surface. The outer surface becomes hard, while the core remains soft and tough.

A carburizing atmosphere must be able to transfer carbon to the steel surface to provide the required surface hardness. The furnace atmosphere can principally be divided into a neutral gas and an active gas.

The neutral gas is generally nitrogen. Nitrogen dilutes the concentrations of the active and flammable gases to minimize flames, risk of soot and ensures safety.

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The active gas plays the role of carbon source. This could be carbon monoxide (CO), hydrogen (H_2), a hydrocarbon (C_nH_m), an alcohol or any other liquid carbon source.

There are three main options to produce an atmosphere for carburizing:

- Endogas – Fuel and air are mixed and combusted in a special endothermic gas generator in accordance with one of the following reactions:
  - $C_3H_8 + 7.2\ \text{air} \rightarrow 5.7\ N_2 + 3CO + 4H_2$
  - $CH_4 + 2.4\ \text{air} \rightarrow 1.9\ N_2 + CO + 2H_2$

- Nitrogen/Methanol Atmospheres – Introducing nitrogen and methanol directly into the furnace chamber by a temperature above 700-800°C is a common way to create a carburizing atmosphere. Methanol cracks to form carbon monoxide:
  - $CH_3OH \rightarrow CO + 2H_2$

- 50%CO/50%H_2 Atmosphere – Oxidizing methane with CO_2 creates an atmosphere consisting of equal parts of CO and H_2, according to the reaction:
  - $CH_4 + CO_2 \rightarrow 2CO + 2H_2$

Atmosphere Control Systems

Atmosphere control for carburizing processes can be automatic, semiautomatic or manual. In 100% automatic control, the flows of different media are automatically adjusted to ensure that the set points for the atmosphere carbon potential and composition are maintained. This is achieved by connecting gas sampling, gas analysis and flow control to the control cabinet that contains the required software algorithms, analyzers and controllers as shown for the example of nitrogen/methanol system for a pusher furnace in Figure 5.

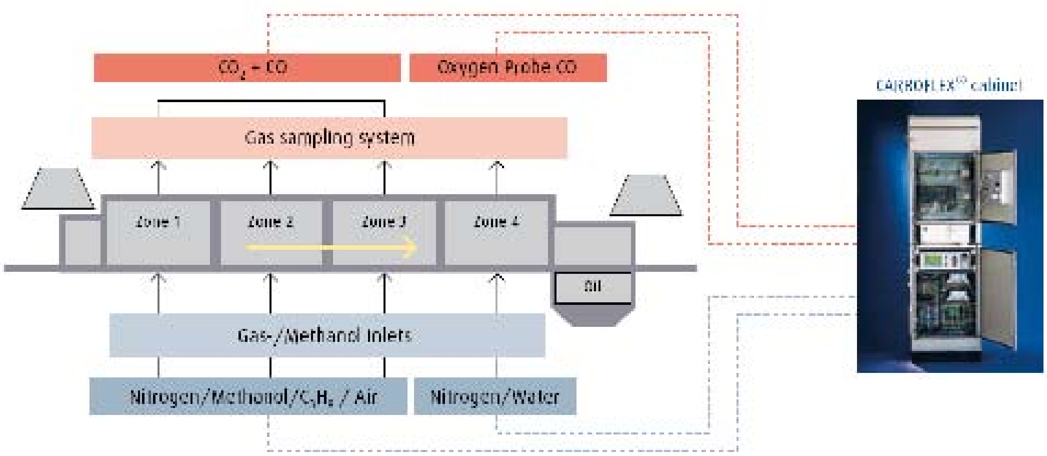

Figure 5. Example of a closed loop atmosphere control system.

As a safety precaution, all media except nitrogen should have safety shut-off devices. The most common method is to allow all additions only to be made above a given temperature. The additions should also be stopped at a given minimum flow or nitrogen pressure.

Solution for faster carburizing in continuous furnaces

In order to have a short carburizing time, the atmosphere in the furnace should have a high carbon potential in the beginning and a lower carbon potential at the end of the process cycle. In case of a continuous furnace without sealed doors, it is not possible to separate different atmospheres.

A process has been developed, consisting of adding water in the diffusion zone. In order to restrict the effect of water to this specific zone, a new flow direction in the furnace has been defined. Figure 6 shows the atmosphere control system.

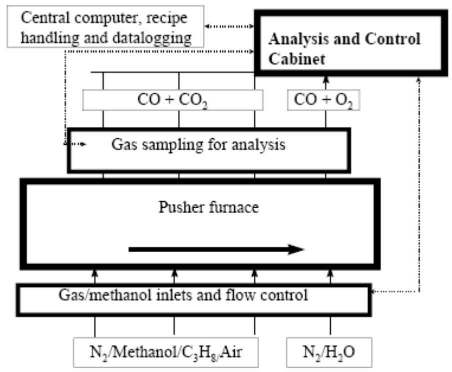

Figure 6. The principal design of the atmosphere control system.

Figure 7 and 8 shows the improvement in the process due to this new system. A time saving of 15% can be reached. There is although a potential to run with higher carbon potentials without the risk of sooting and too produce parts with a lower amount of retained austenite by reducing the surface carbon concentration.

If there is no need for higher production rate, it is possible to utilise the control system to lower the furnace's temperature and maintain the same productivity.

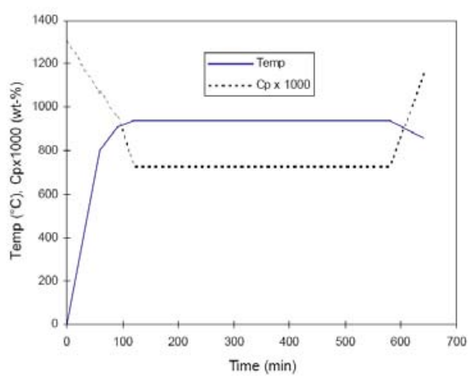

Figure 7. Carbon potential Cp, temperature and time needed with the normal carburizing cycle.

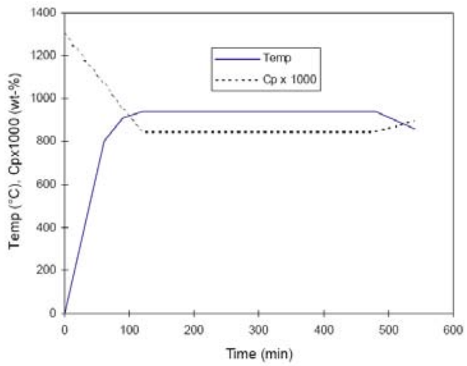

Figure 8. Carbon potential Cp, temperature and time needed with the new atmosphere control system.

Nitriding and Nitrocarburising

Introduction and basic principles

Nitriding and nitrocarburising are thermochemical processes where nitrogen and/or carbon are transferred from the process medium, normally gas, to the surface of the treated steel und a low temperature (500-700°C). Both processes give unique improvement in wear and corrosion as well as a reduced distortion of the treated parts. Figure 9 shows the hardness after Nitriding and nitrocarburising of different steels.

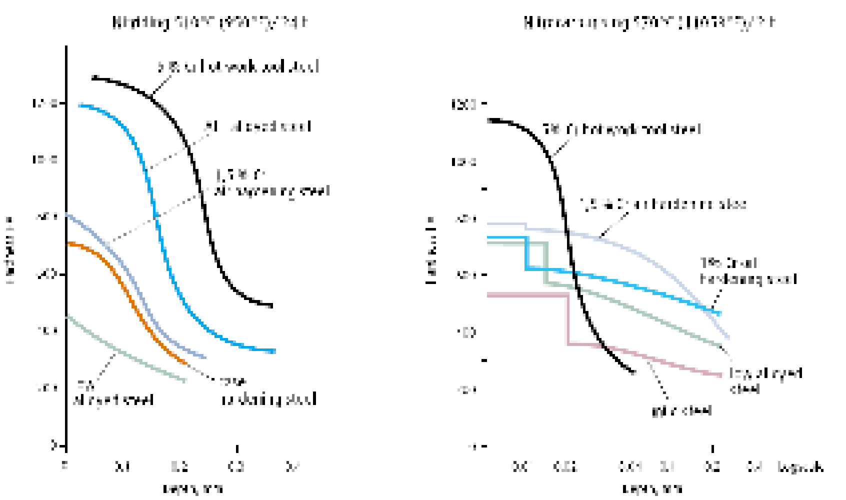

Figure 9. Typical hardness after nitriding and nitrocarburising.

During Nitriding, ammonia (NH3) decomposes into hydrogen and nitrogen at the surface, enabling nitrogen atoms to be absorbed at the steel surface and to be diffuse further into the steel. In nitrocarburising, it is additionally necessary to have a carbonaceous gas transferring carbon to the steel surface.

Atmosphere Control Systems

Atmosphere composition and control are of crucial importance for the nitriding and nitrocarburising results with respect to final properties such as wear and fatigue resistance.

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The nitriding potential is determined by gas analysis. In nitrocarburising the gas analysis aims to determine both the nitriding and the carbon potentials. The analysis of nitrocarburising atmosphere is difficult due to the complex gas composition (NH3, H2, CO, CO2 and H2O) with fairly high water vapour content, which may result in the precipitation of ammonium carbonate, which clogs the analyser and analysing sample gas pipes.

Linde Gas developed an atmosphere control system for nitriding and nitrocarburising named NITROFLEX®. This atmosphere system has the inherent advantages of flow rate and mixing flexibility typical of synthetic in situ generated atmospheres. It provides an opportunity to optimise the gas composition in relation to the type of furnace, steel and properties required. If used efficiently, these advantages lead to a minimized costs and a higher quality. Safety and the potential for increased productivity are additional benefits.

One feature of NITROFLEX® atmospheres is a carbon activity that is much lower than that in systems using endogas together with a higher oxygen activity. Due to this fact, practice has shown that a faster growth rate of the compound layer can be obtained. It has been demonstrated that alloyed steels such as hot work tool steels obtain thicker and more even compound layer (see Figure 10). The balanced carbon activity means that the driving force for soot deposits in furnaces is low.

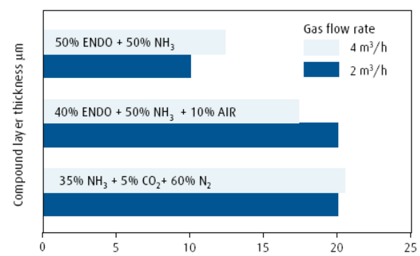

Figure 65. Compound layer thickness for different atmospheres showing faster growth for the NITROFLEX® system [13].

Figure 10. Compound layer thickness for different atmospheres showing faster growth for the NITROFLEX® system.

Sintering

Introduction

The term sintering describes a heat treatment during which a powder mass is densified and adopts the desired composition. Due to their porous structure, pressed powder components react more readily with the surrounding atmosphere than fully dense materials. Therefore, the sintering atmosphere and its control are very important.

The protective atmosphere can be based on hydrogen, nitrogen, dissociated ammonia, endogas, exogas or vacuum.

Atmosphere Control Systems

The quality of any production is directly related to control of process parameters. The metallurgical properties and especially the microstructural changes in a sintering component can only be controlled by controlling the surrounding furnace atmosphere accurately, however the carbon control of the sintering process has always been incomplete and somewhat out of control. A successful control of carbon potential

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during a sintering process helps to produce higher quality products and at the same time facilitate a lower cost production.

Chromium has become more popular as an alloying element in the sintering industry in recent years because of its advantages in increasing hardenability and resistance to softening during tempering. Nickel is a more common alloying element but it is starting to be substituted by chromium because of its significantly higher price. One of the main reasons why this shift is slow is that chromium has properties that affect its performance in the sintering process, particularly its high affinity for oxygen. Surface decarburization is the most common problem associated with the use of present furnace atmospheres for chromium alloys such as Astaloy CrM. This decarburization increases the sensitivity to fatigue cracks.

In order to solve this problem, Linde Gas and Höganäs AB developed a new C-Potential Control Technology for Sintering Furnaces named SINTERFLEX^{TM}. The system uses the simple gas sampling principle of the sintering furnace in such a way that the newly designed oxygen probe and internal analysis furnace arrangement would give exact C-Potential readings of the sintering zone of the furnace. The system controls the gas composition, particularly its carbon enrichment, to maintain a healthy C-Potential to produce sintered products of chromium alloys such as Astaloy CrM that are free of decarburization.

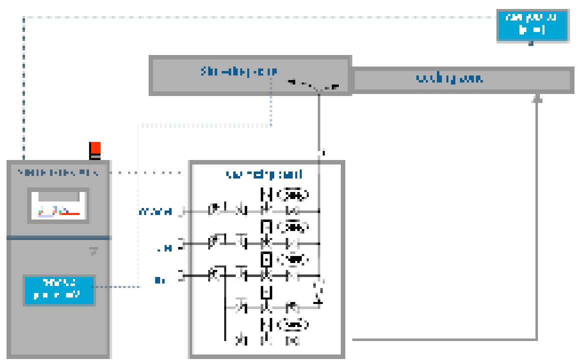
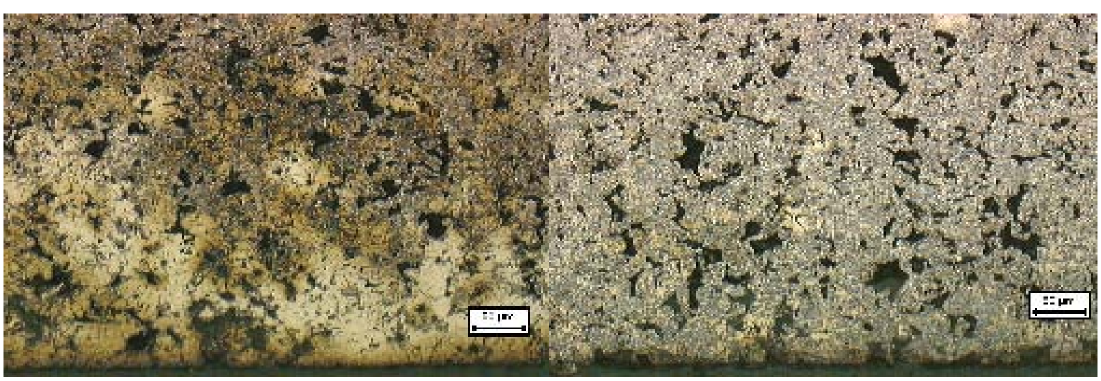


Figure 11. SINTERFLEX.

Figure 12 (left side) shows an Astaloy CrM treated with an atmosphere containing N2 - 2% H2- 2% CO and additions of C3H8. Goal was to carburize the surface to 0.45% carbon where the carbon content in the centre is 0.38%. The results showed a positive carburizing gradient approximately ~150µm thick for CrM material. The same material however showed no carbon profile in a dry atmosphere of N2 – 10% H2 (Figure 12 right side).



Astaloy CrM treated with CO atmosphere *Astaloy CrM treated with N2/H2 Atmosphere*

Figure 12. Comparison of CrM Microstructures in different atmosphere conditions.

The SINTERFLEX™ enables to process with carbon neutral or carburizing atmospheres to avoid surface decarburization giving higher quality parts and increased product performance. It can although be used to replace a subsequent case hardening operation by means of tailored surface carbon gradients during sintering, leading to lower total cost and better dimensional tolerances.

CONCLUSIONS

The demand for higher quality and cost improvement in the high volume production of parts has inevitably led to increasingly sophisticated control systems for every heat treatment process.

Keeping parameters like temperature, atmosphere composition and flow on a predefined level is not sufficient to reach these requirements.

Using advanced atmosphere control as a tool in surface engineering has made it possible to obtain dramatic improvements in both product value and process efficiency, and such improvements will be further realized in the future.

An example of positive results obtained with an atmosphere control system is:

- Precise carbon control to avoid decarburization and meet strong quality requirements.
- Drastically reduced total gas consumption compared to generator systems. The flow rate is minimized at anytime, based on the atmosphere composition required to maintain the different set points in each moment.
- Optimized recipes with different flows are at hand to adapt flow and atmosphere composition to part dimension and part quality requirements.
- Special idling programs are used to minimize gas consumption when there is no production.
- The system eliminates substantial costs and manpower for rework of oxidized, decarburized and discoloured parts.
- Process visualization, recipe handling, historical data collection and alarms are appreciated functions.

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