RETROFITTED ATMOSPHERE FURNACE PROVIDES HIGH QUALITY FNC PARTS

Automated control of process temperature, gas composition, and nitriding potential allows precise, repeatable control of white layer on FNC processes, significantly improving quality over that of non-controlled processes.

John W. Gottschalk
Surface Combustion Inc.
Maumee, Ohio

Dave Nelson
Metal Improvement Inc.
Columbus, Ohio

Advanced furnace control systems and processing software allow for fully automated control of the ferritic nitrocarburizing (FNC) process for a wide variety of steel, powder metallurgy, and ductile iron components. The automated system improves process control and reduces cycle times over non-controlled or alternative technologies, such as fluidized bed treatment.

FNC processes have been successfully run in a variety of furnace designs over the history of the process. Conventional atmosphere and fluidized bed furnaces have been used for many years with a variety of atmosphere blends to achieve a nitrided case on steel and ductile iron components. The process has typically been performed in an open-loop control system utilizing predetermined gas flows to create the required atmosphere in the furnace. Empirical results achieved after processing are then used to determine the required gas flows for the next cycle. Cycle repeatability can be challenging with this system based on fluctuations in utility gas supply, furnace maintenance, and load size and part configurations. New process specifications requiring closed-loop control and general requirements for tight metallurgical repeatability require improved process controls.

Metal Improvement Co., Columbus, Ohio, was previously processing FNC work in fluidized bed furnaces within its organization. Metal Improvement's requirements for a more controlled, flexible process led the company to a controlled FNC process that was retrofitted onto an existing two-chamber atmosphere furnace design. The furnace selected for this retrofit was a 36 in. × 48 in. × 36 in. Surface Combustion Allcase® batch furnace with integral atmosphere cooling chamber (Fig. 1). The atmosphere cooling chamber allows cooling the parts under atmosphere to near ambient temperatures providing a "clean" nonoxidized appearance to the parts. In furnaces with oil quench chambers in addition to atmosphere cooling, it is possible to provide improved corrosion resistance by quenching the parts at the completion of the cycle.

The furnace had previously been used in the motor-lamination industry and required a complete conversion of controls and atmosphere for the new process. The conversion consisted of new atmosphere controls (flow meters, shut off valves, safety components, etc.), atmosphere probes and process control PLC (provided by Stange Elektronik GmbH, Gummersbach, Germany) to supplement the existing material-handling PLC. To fit the many potential processes that would be run in the furnace, a variety of atmosphere gasses were provided. Endothermic gas (RX®), ammonia, nitrogen, and nat-

Retrofitted Surface Combustion Allcase furnace at Metal Improvement for running FNC process.

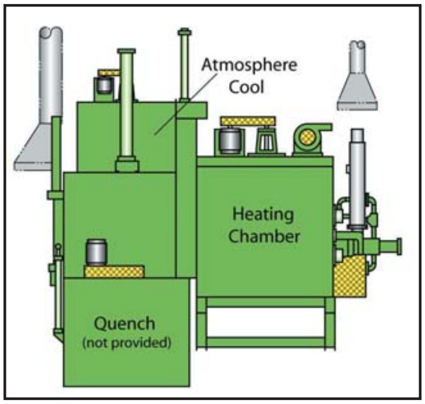

Fig. 1 — Basic configuration of Surface Combustion Allcase batch furnace with integral atmosphere cooling chamber.

ural gas (added for use with Triniding™ cycles) were provided. A critical component of the retrofit was monitoring/control of process gases, so flow measurement and control components were provided for all process gases.

When operating with the gas mixtures used for FNC processes, equilibrium of both carbon and nitrogen in the material must be considered to achieve the desired metallurgical characteristics. To control the process at the appropriate process conditions, both the nitriding and carbon levels must be considered. As the carbon level and nitriding potential are linked by the gas flow rates, only one of them must be controlled to maintain a constant atmosphere potential. The nitriding potential was selected as it is much easier to measure than the carburizing potential at the reduced process temperatures associated with the FNC process. The formula for the nitriding potential is given by:

$$K_n = pNH_3/pH_2^{1.5}$$

where pNH_3 = partial pressure of ammonia and pH_2 = partial pressure hydrogen.

K_n was calculated using a special probe for measuring hydrogen concentration of the furnace atmosphere. The probe has been widely used in Europe on both classical gas nitriding and FNC furnaces (Fig. 2). Using this information with the known flow rates of the process gases makes it possible to calculate the nitriding potential throughout the cycle. This probe design eliminates sample pumps, piping, analyzers, and filters generally used to monitor ammonia bearing gases. Additionally, the design eliminates precipitation of solids caused by the reaction of ammonia and carbon dioxide at low temperatures. The measured K_n value changes throughout the cycle as the dissociation rate of ammonia changes with the nitrided surface condition of the parts. This control mechanism allows the system to function independently of part surface area and condition as it tries to maintain a constant potential level through the process. If required, the process can be split into multiple segments of varying K_n settings to provide more precise control of white-layer thickness.

The nitriding potential calculation modules were provided by STANGE Elektronik and integrated into the furnace mechanical and atmosphere control systems by Surface Combustion.

To develop process recipes, an off-line calculation tool is available to determine process control parameters. Inputs and outputs to the calculation tool include process gas type, flow rates, operating temperature, and nitriding potential (K_n). Examples of the module are shown in Fig. 3 for two different process gas mixtures achieving the same equilibrium relationship to the material. The charts are single-temperature representations of the tertiary *Kunze* diagram for nitrogen and carbon equilibrium in the steel.

The benefit of this software model is shown by the atmosphere equilibrium with the epsilon ($Fe_{2\text{-}3}N$), gamma prime (Fe_4N) and iron carbide (Fe_3C) portions of the *Kunze* diagram embedded within the program and the ability to correlate this information back to the selected process gases.

The FNC process has been run in

Fig. 2 — Probe for measuring hydrogen concentration of the furnace atmosphere.

Process selection: Nitrocarburizing

Setpoint values:
585 Temperature [°C]
☐ 750 Temperature of probe [°C]
● 2 Kn
○ 13,6 Dissoziation [%]

Fresh gas (Part, %):
450 66.2 Ammonia
☐ 0 Dissoziated Ammonia
☑ 100 14.7 Nitrogen
☐ 0 Air
☐ 0 Nitrous Oxide
☐ 0 Water
☐ 0 Carbon dioxide
☐ 200 Endogas
☑ 90 13.2 Carbon monoxide
☐ 0 Exogas
☑ 40 5.9 Hydrogen
☐ 0 frei

Results:
Kn 2
Dissoziation [%] 22.5
Ko 0
Ko(W) 0
Kc(W) 5.53
Kc(B) 2.17
Q-probe [mV] 1157.98
H2-Sensor [%] 31.95

Process selection: Nitrocarburizing

Setpoint values:
585 Temperature [°C]
☐ 750 Temperature of probe [°C]
● 2 Kn
○ 13,6 Dissoziation [%]

Fresh gas (Part, %):
450 56.2 Ammonia
☐ 0 Dissoziated Ammonia
☑ 150 18.8 Nitrogen
☐ 0 Air
☐ 0 Nitrous Oxide
☐ 0 Water
☐ 0 Carbon dioxide
☑ 200 25 Endogas
☐ 40 Carbon monoxide
☐ 0 Exogas
☐ 80 Hydrogen
☐ 0 frei

Results:
Kn 2
Dissoziation [%] 17.4
Ko 0
Ko(W) 0
Kc(W) 5.53
Kc(B) 2.17
Q-probe [mV] 1192.34
H2-Sensor [%] 30.41

Fig. 3 — Modules for two different process gas mixtures achieving the same equilibrium relationship to the material. Charts are single-temperature representations of the tertiary *Kunze* diagram for nitrogen and carbon equilibrium in the steel. Inputs and outputs to the calculation tool include process gas type, flow rates, operating temperature, and nitriding potential (K_n).

Table 1 — Test cycle parameters for ferritic nitrocarburizing low and medium carbon steels

Operating parameter	Test 1 (AISI 1010 low carbon steel)	Test 2 (AISI 1018 low carbon steel)
Temperature, °F	1085	1085
Cycle time, h	2	2
K_n(a)	2	2
Operating gases	Ammonia Nitrogen endothermic (RX)	Ammonia Nitrogen endothermic (RX)
White layer thickness, in.	~0.0005	~0.0007
Microstructure	See Fig. 4	See Fig. 5
Hydrogen concentration, %	20-22	20-22

(a) Calculated K_n value. Initial test performed in manual mode of control.

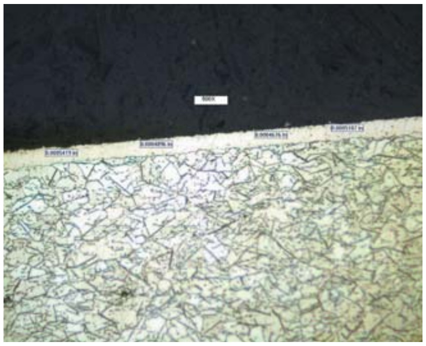

Fig. 4 — Very uniform white layer (0.0004676 to 0.0005419 in. thick) with limited porosity at the surface of the sample. 500×

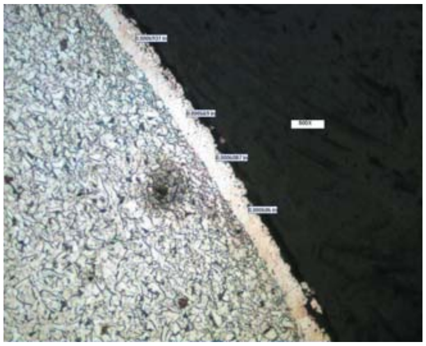

Fig. 5 — Substantially thicker white layer (0.000606 to 0.0006937 in. thick) with some developed porosity at the surface. 500×

the past with combinations of some or all of the following process gasses: ammonia, endothermic, exothermic, natural gas, nitrogen, and carbon dioxide. The calculation tool allows specific process gas flow rates to be compared among alternative gases to achieve the same process environment. This flexibility allows different facilities using the same or different processes to approximate cycle conditions without running test loads. The method of automating the process is by modulating one of the gas flows to maintain a constant K_n throughout the process cycle.

The furnace design selected for this process is a two-chamber design with the following typical process cycle:

- Furnace initially purged with nitrogen
- Load charged into furnace under nitrogen and/or endothermic (RX) atmosphere
- Ammonia introduced to heating chamber
- Process completed per required cycle time
- Load transferred under atmosphere to vestibule
- Load transferred into cooling chamber (nitrogen or RX gas used in cooling chamber)
- New load transferred into furnace chamber for processing

The flow rates are automatically adjusted throughout the process by preconfigured gas flow set points. This configuration allows conserving atmosphere during process segments (low flow condition) and allows for high flows during load/unload sequences, ensuring safe operation of the equipment.

Initial testing of the system was performed with low and medium carbon steel and ductile iron. Test cycle parameters for two of the materials are given in Table 1. Figure 5 show that the white layer is very uniform with limited porosity at the surface of the sample. Figure 6 shows the whiter layer is substantially thicker and has developed porosity at the surface. As the porosity is not generally desirable, several modifications could be made to the cycle to reduce this condition. Reduction in cycle time or operating temperature would both reduce the white layer condition providing a less porous condition.

Conclusions

Automated control of process temperature, gas composition, and nitriding potential allows precise, repeatable control of white layer on FNC processes, significantly improving quality over that of non-controlled processes. By combining the advanced control functionality with a multichamber furnace design, cycle times can be optimized over those of conventional single chamber batch furnace designs. System productivity may be doubled by elimination of purging, heat up, and cool down required in single-chamber designs. This increase in production leads to faster door-to-door times and reduces operating costs per pound of material processed. HTP

For more information: John W. Gottschalk is Director Special Products, Surface Combustion Inc., 1700 Indian Wood Circle, Maumee, Ohio 43537; tel: 419-891-7150, or 800-537-8980; e-mail: info@surfacecombustion.com; Web site: www.surfacecombustion.com.

Dave Nelson, Metal Improvement Inc., 1515 Universal Rd., Columbus, OH 43207; tel: 614-444-1181; fax: 614-444-0421; e-mail: columbus-heattreat@metalimprovement.com; Web site: www.metalimprovement.com.