Application Note

**MFCs for Gas Control In Case Hardening Processes**

**PROBLEM**
Surface hardening is a high temperature process that creates hard surface alloy layers that improve a metal’s wear resistance. The technique has been used for many years, however, precise control of the surface alloy composition is difficult since accurate control of the gas chemistry is challenging in these processes, which in turn makes it difficult to control the composition of the surface alloy. Poor control leads to high part rejection rates, inefficient use of furnace operating time, and higher than necessary ambient gas costs.

**BACKGROUND**

**Case Hardening**
System case hardening is a high temperature, batch furnace process in which gaseous hardening agents (a carbon monoxide/hydrogen mixture generated in an endothermic gas generator) are cracked on the steel surface to produce carbon that then diffuses into the microstructure of the steel, raising the carbon content (up to ~0.9% C) of a surface layer and hardening it relative to the tough, ductile core. The surface layer thickness (“case depth”) depends on process temperature, composition of the carbon source, time-at-temperature, and the carbon chemical potential at the surface during diffusion. The carrier gas is produced using a precisely metered mixture of methane and other hydrocarbons that are reacted with just enough oxygen to produce the desired carbon monoxide/hydrogen ratio. Alternatively, the need for endothermic gas generators can be avoided by direct injection of a nitrogen/methanol mixture into the furnace chamber. Both methods require precise control of the precursor gas concentrations in the furnace ambient. Nitridation is a lower temperature hardening process that cracks a mixture of ammonia, NH₃, and hydrogen, H₂, to form a hard nitride layer on steel. Different nitride phases can form, depending on the NH₃ and H₂ partial pressures in the furnace and ammonia must always be present in excess in the furnace ambient to ensure the proper nitride formulation on the steel surface. Excess ammonia in the furnace ambient is ensured using direct measurements of its concentration in the process exhaust and these measurements are used to control the ammonia flow rate into the furnace ambient using Mass Flow Controller (MFC) technology (Figure 1).

**Gas Delivery Requirements in Case Hardening Processes**
Endothermic gas generators in carburization processes require specific feed-gas ratios to ensure very low levels of by-product CO₂ and H₂O in the carrier gas since these contaminants produce out-of-spec case hardening. As well, if air/hydrocarbon ratios in the feed are too low, soot will deposit in both the gas generator and furnace, resulting in maintenance problems. Precise control of precursor gas ratios is critical for proper gas generator and furnace operation. Unfortunately, no matter how precise the precursor control, some small amount of CO₂ and H₂O will always be present in the carrier gas. These residues are removed using a controlled injection of methane (“hardening agent”) just prior to injection of carrier gas into the furnace. Process control is maintained by monitoring CO₂ and H₂O levels in the carrier gas. Then, this information is employed in feedback control of the input and injection gas flow rates by mass flow controller technology. O₂, CH₄, CO, and CO₂ levels in the carrier gas are monitored by oxygen probes and infrared gas analyzers adapted for hydrocarbon analyses. Feedback from these analytical tools and MFC control of feed-gas flows determine generator gas ratio and amount of hardening agent injected into the carrier gas. This ensures constant chemistry in the furnace ambient which in turn assures process accuracy and repeatability.

Carburization processes that inject methanol into the furnace ambient are complicated by the fact that direct injection of pure liquid methanol results in vaporization and pressure pulsing in the furnace ambient which disrupts steady state.
operation. This is avoided by injecting an entrained N₂/methanol gas with tightly controlled ratios that are maintained using MFC control.

Nitridation processes require a controlled excess of ammonia in the furnace ambient that is maintained by exhaust analyses and MFC control of feed-gases similar to that employed for carburization. As well, precision gas flow control in nitridation processes minimizes operational costs due to ammonia gas and any need for high temperature processing steps.

The integration of MKS Instruments’ digital MFC technology with sensor technologies in closed loop control ensures continuously adjustable gas flows and steady state conditions during case hardening processes. In multi-step nitridation processes with changing temperature profiles, MKS MFC’s permit controlled, time-dependent gas flow ramps tailored to furnace temperature transitions.

MKS Instruments offers a variety of standard industrial communication protocols and is continually expanding MFC options based on industry communication requirements for other digital protocols such as EtherCAT and Profinet. On board diagnostics, accessible via Ethernet TCP/IP communications protocols, provide customers with the ability to modify the setup of the MFC to control the flow of different gases and to change the scale of the flow measurement and control. The on-board diagnostics can also be used to troubleshoot the functionality of the device, providing plot functions for critical MFC variables.

CONCLUSION

The product quality and operational costs associated with case hardening heat treatment processes such as carburizing and nitriding are determined by the ambient gas atmosphere and temperature in the furnace. Significant improvements in throughput, reductions in gas costs, and improved run-to-run consistency can be achieved by coupling appropriate sensor technology with advanced MFC technology to achieve real-time, adjustable gas flow rate control that maintains a constant chemical composition in the furnace, which in turn maintains a constant concentration of the hardening agent at the gas/steel interface. MKS Instruments’ I-Series MFC family of IP66-rated Thermal Mass Flow Controllers and Mass Flow Meters for Industrial Environments have all the features necessary for successful integration into case hardening applications.

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