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Smart nitrogen-methanol injection lance for carburizing and hardening atmospheres, a case study

by **Maximus Akuh, Tim Nitsch, Guido Plicht, Felix Steinbach**

Air Products' innovative Smart Lance and Air Products Process Intelligence (APPI) system in combination is already being used in the field to achieve optimal atmosphere results, process monitoring and visualization including documentation and possible preventive maintenance. In the process, the following advantages were realized in real applications at customers: provision of a reliable protective atmosphere supply to produce components within a tight tolerance range, an increase in productivity due to fewer burnout cycles and shorter forming times, a reduction in rework, a reduction in the cost of the atmosphere, documentation of the atmosphere and process parameters also in line with CQI-9 standards, indication for carrying out preventive maintenance during planned shutdowns, reducing lost time for troubleshooting during furnace failures.

In the heat treatment process of carbon steels, the use of tailored furnace atmospheres for the realization of targeted material properties on the components treated is indispensable. There are two main reasons for the use of an atmosphere: on the one hand, it should have a reactive effect on the component to be treated, on the other hand, it can be protective or in a state of equilibrium with the component surface.

This study deals with carbon containing atmospheres for heat treatment of carbon steels, which can be produced by endogas generators or by a nitrogen-methanol direct injection.

The use of endogas generators to produce the atmosphere through a natural gas/air reaction is widely used in heat treatment processes such as carburizing, hardening, and neutral annealing of carbon steel components requiring a protective atmosphere.

In today's world, where sustainability is more and more in the public focus, combined with the fact that the composition of natural gas will be variable in the future due to enrichment with green H_2, this will also have an impact on the atmosphere composition from the endogas generator. In addition, in most cases an endogas generator is kept at temperature as a backup in case of failure of the running system, thus unnecessarily using energy that is basically not directly generated for production.

These and other factors are driving heat treatment companies to look for alternative solutions. One not far-fetched and proven alternative is the well-known use of methanol and nitrogen to produce the required carbon monoxide (CO), hydrogen (H_2) and nitrogen (N_2) atmosphere mixture.

Air Products has developed a new nitrogen-methanol lance which, on the one hand, achieves an ideal cracking of the methanol into CO and H_2 through an optimal atomization of the methanol into the furnace chamber and, on the other hand, is also monitored to achieve a higher process reliability and to reduce rejects and rework. The following study is based on an example to illustrate the advantages achieved in the practical operation of a chamber furnace carried out at Härtetechnik Steinbach GmbH & Co KG (Härtetechnik Steinbach).

THE NITROGEN-METHANOL PROCESS

The use of nitrogen and methanol in heat treatment to provide a protective atmosphere in a neutral annealing, hardening or carburizing process is well known and well

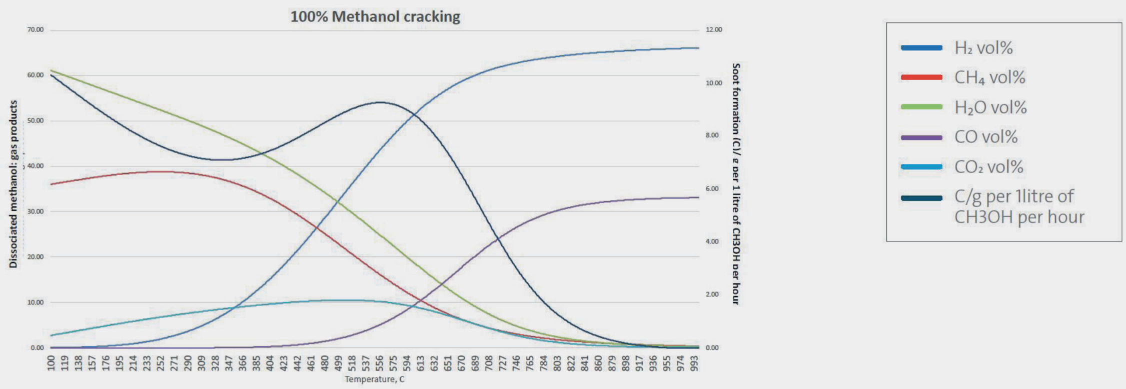

Diagram 1: FactSage simulation of methanol cracking

established. The atmosphere produced from nitrogen-methanol is a simple and practical alternative to that of an endogas generator and has a high degree of flexibility in application.

Nitrogen and methanol are stored on site in tanks. Alternatively, nitrogen can also be generated on site. Both media can then be withdrawn as needed for the process, allowing the CO content in the atmosphere to be adapted to the application to reduce the CO_2-footprint. If no reactive or protective atmosphere is required, no further energy needs to be used contrary to a standby endogas generator.

Theoretically, the atmosphere composition generated in the furnace from methanol cracking at temperatures above 800 °C can be comparable to that of endogas generators according to the cracking reaction:

$CH_3OH \rightarrow CO + 2\ H_2$ with minor components of CO_2 and H_2O (**Diagram 1**)

In practice, however, the optimal composition is only achieved if the methanol injection into the furnace is realized in such a way that the methanol cracking from the liquid phase occurs directly at high furnace temperature. Diagram 1 shows the variation of the composition of the methanol cracking at different temperatures from a factSage simulation. Slow heating of the methanol with evaporation at temperatures above 400 °C leads to a high soot content.

At temperatures above 850 °C the by-products such as CH_4, H_2O, CO_2 and soot are only present in very small proportions, while the majority is cracked into the desired CO and H_2.

Air Products' metals processing development team, with many years of experience in this field, had set itself the task of further optimizing the nitrogen-methanol injection and has developed an intelligent nitrogen-methanol lance for this purpose.

A sketch of the system concept for a complete installation including atmosphere control is shown in **Fig. 1**.

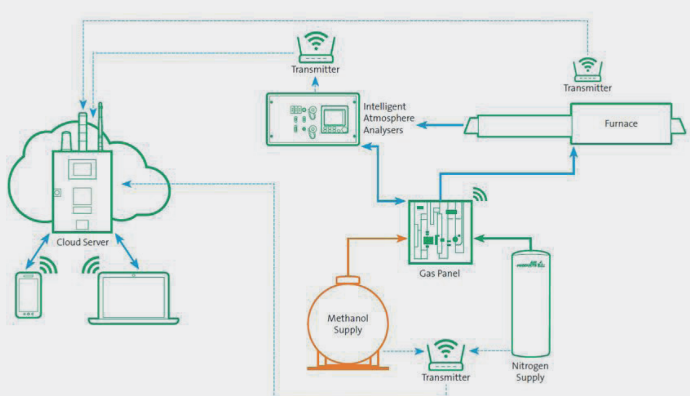

Fig. 1: Nitrogen-methanol installation

AIR PRODUCTS' INNOVATIVE NITROGEN-METHANOL SMART LANCE AND AIR PRODUCTS PROCESS INTELLIGENCE SYSTEM (APPI)

Based on practical applications in the industry and optimization work carried out, the following points have been identified as key to achieving the desired optimum methanol cracking results:

- The practice of dropping methanol through the lance onto a metal plate in a furnace causes it to evaporate slowly and often results in higher CO_2, H_2O and CH_4 levels in the generated atmosphere, which in turn leads to a higher addition of boosting gas and thus increased soot formation.
- Slow heating (vaporization) and cracking of methanol at low temperature inside the lance must be avoided. This

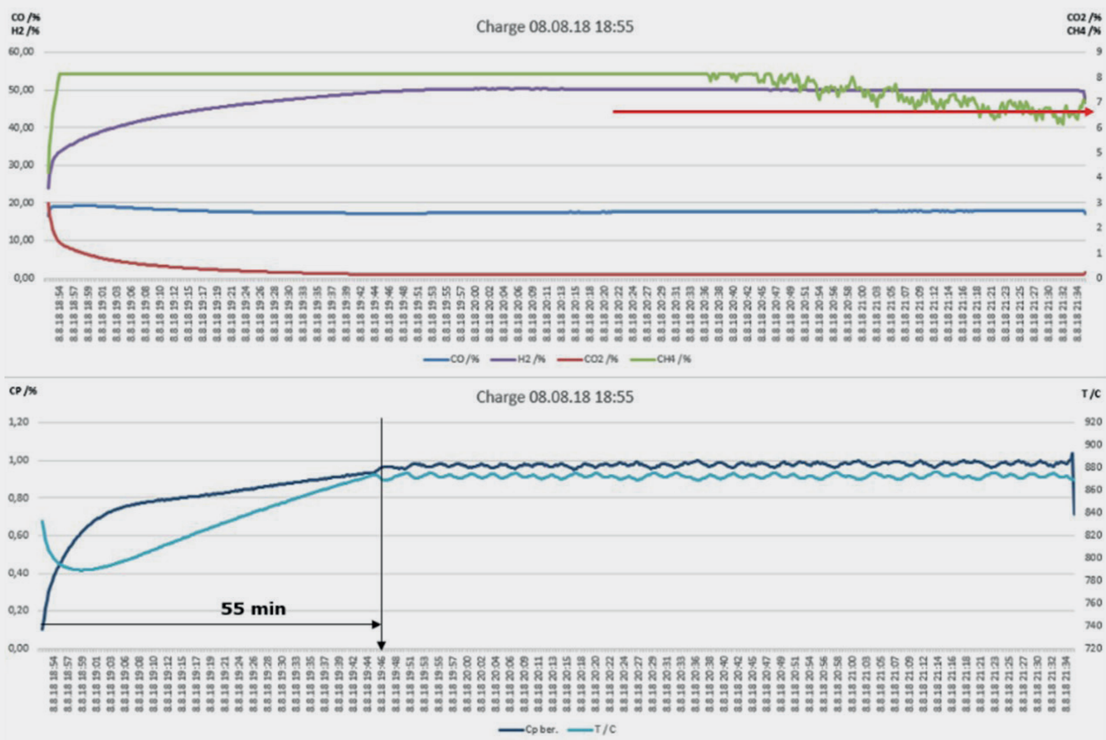

Diagram 2: An example of a batch analysis with the conventional lance

creates soot that can clog the lance and an increased CO_2 and H_2O content.

- A homogeneous distribution of a fine methanol aerosol (micrometer particle size) must be generated in the furnace, providing a large surface area to achieve maximum cracking into CO & H_2, thereby reducing the formation of undesired by-products such as CO_2 & H_2O. The generation of the aerosol means that the methanol must enter the furnace atmosphere still in liquid form.
- In the case of a bricked furnace, the methanol aerosol must not be sprayed onto the brickwork, which would lead to slow evaporation with the formation of CO_2 & H_2O.
- Temperature and pressure monitoring of both media in the lance can provide real-time information about its status. In this way, the lance can be positioned correctly and any deterioration in the furnace atmosphere can be detected and corrected timely, before any damage or failure occurs.

These requirements for achieving optimum performance were considered in the development of the new smart lance from Air Products. The results and advantages achieved at Härtetechnik Steinbach with the smart lance compared to an existing conventional lance are mentioned below.

THE SMART LANCE COMPARED TO A CONVENTIONAL NITROGEN-METHANOL INTRODUCTION

Before installing the new nitrogen-methanol lance, a representative measurement was carried out over a long period to record the existing atmosphere conditions of as many different heat-treated batches as possible. The data obtained from such an analysis of the furnace atmosphere forms the basis for a subsequent benchmarking with the smart lance and makes the direct short-term benefits visible. The long-term benefits, such as extension of burnout cycles or reduction of rejects through preventive maintenance, then become apparent over the course of the operating time.

The following study was carried out on a chamber furnace equipped with a conventional methanol/nitrogen lance installed near the blower, dropping the methanol on the fan. For later comparison, the atmosphere profiles of different carburizing and hardening batches were recorded. An example of a charge measurement shows in **Diagram 2**. In the above diagram, a batch was heat treated at a temperature of 870 °C and the holding time was one hour and 46 minutes. The conditioning of the furnace atmosphere took 55 minutes. This is the time from the completion of charging until the atmosphere reached the set carbon potential (CP). In this case, the carbon potential (CP) set point was 1.0%.

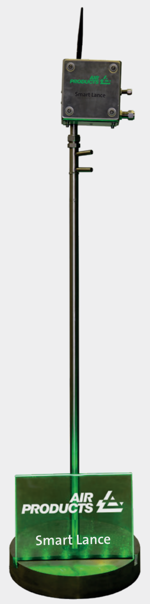

Fig. 2a): The smart methanol lance

Fig. 2b): The communication unit

The CP is influenced by various parameters such as temperature, composition of the atmosphere and input of oxidizing media. Nevertheless, the composition of the atmosphere also depends on the quality of the methanol cracking. As already explained, poor cracking results in higher amounts of H_2O and CO_2, which must be reduced by adding more hydrocarbons (boosting gas), which in turn can lead to greater soot formation. It also increases the conditioning time of the furnace atmosphere, the time needed to reach the desired CP set point.

The conditioning time, especially if it is longer than the batch heating time, can be considered as unproductive and resource-intensive time. Reducing this time reduces production costs and significantly increases productivity.

It is also worth mentioning that the charge surface area and the process temperature also influence the composition of the atmosphere.

Nevertheless, low residual CH_4 contents after reaching the CP setpoint in the atmosphere is an indication of a conditioned state of the required atmosphere in which excessive boosting gas (natural gas or propane) does not have to be added to compensate for external influences such as CO_2, H_2O, or air (O_2) ingress. This also indicates good methanol cracking and a well-adjusted addition of boosting gas to control the CP set point in the furnace. In the example above the residual CH_4-content in the conditioned stage is still very high, indicating sources of CO_2 and H_2O to be compensated by high propane addition.

INSTALLATION OF THE NITROGEN-METHANOL SMART LANCE

After analyzing the furnace atmosphere with the conventional methanol lance, the Air Products smart lance was installed on the same furnace and same injection point. The smart lance consists of two components:

- The methanol and nitrogen injection lance, usually installed through the furnace roof (**Fig. 2a**).
- The communication unit and data acquisition (**Fig. 2b**)

Both components communicate via a Wi-Fi connection. The communication unit collects real-time pressure and temperature data of the media methanol and nitrogen flowing through the lance into the furnace. It processes the data and provides immediate feedback on the status of the lance via green, yellow, and red status signal lights on the communication unit. The data is then uploaded to an internet cloud server for further processing, visualization, and archiving. The immediate feedback is as follows:

- Green signal light: the injection is working properly.
- Yellow light indicates that the process needs to be observed, but is still in a well-functioning window. The operator should check the process (lance and media supply). If there is no obvious supply problem, preventive maintenance should be carried out at the next production stop and the lance should be inspected.
- Red light means that either the temperature or pressure of methanol or nitrogen is out of range. To avoid the risk of an unplanned production stop, the media supply and methanol lance should be checked as soon as possible.

The effort required to install the smart lance is comparable to that of the conventional lance. After installing the lance in the furnace chamber, the furnace is ready for operation. The communication unit is then set up with alarm settings, paired with the lance via WLAN and connected to the cloud server.

In the study, CO, CO_2, H_2 and CH_4 levels were also measured using a 4-gas analyzer. The flow settings of the media were the same as for the conventional lance. Methanol and natural gas were released after reaching the safety temperature of 750 °C. The composition of the atmosphere was stabilized fairly quickly, and it was further observed for about one hour in stable conditions. The CO and H_2 concentrations were stable and higher than in the conventional lance with the same N_2 and CH_3OH flow rates. To obtain the standard values of 20% CO and 40%H_2 the flow rate of methanol was reduced. The first positive effect on cost reduction was realized.

Diagram 3 shows the results of a batch of 240 kg annealed at a temperature of 870 °C and held for 40 minutes. It took only 16 minutes for the atmosphere to be conditioned, a great contribution to the increase in productivity. The residual CH_4 levels in the atmosphere were also very low, less than 0.3% in the conditioned state, indicating good methanol cracking.

The comparison of the performance of both lances is summarized in **Diagram 4**. In both cases, the measurements were carried out during normal production with mixed batches. A total of 32 batches were analyzed for the conventional lance and 28 batches for the smart lance.

REALIZED BENEFITS WITH THE SMART LANCE

Methanol cracking, as shown in the table above, has been significantly improved with the smart lance, reflecting its high efficiency, with the following advantages obtained:

- Higher CO and H_2 contents in the atmosphere composition with the same methanol and nitrogen flow rates as with the conventional lance.
- Reduced H_2O, CO_2 and CH_4 concentrations from methanol cracking.
- To achieve the same atmosphere composition as with the conventional lance, a reduction in the methanol flow rate was necessary, thus savings in methanol were achieved.
- In addition, with the improved cracking, the conditioning time of the atmosphere has been shortened, resulting in a time gain and an increase in productivity.
- This was accompanied by a reduction in the amount of natural gas used to set the carbon potential.
- And due to the lower CH_4 levels in the atmosphere, less soot was formed in the furnace, reducing the number of burnout cycles, which also led to an increase in productivity.

The real-time status of the smart lance can be viewed on the communication unit through alarm messages and in detail on the display, which shows the temperature and pressure of both media in the lance in real time (**Fig. 3**). The temperature of the methanol in this example is 44 °C, which indicates that it is still in liquid form and has not evaporated or started to crack in the lance.

The pressure is 0.1 bar, indicating that there is no clogging at the nozzle. A pressure build-up here would indicate a possible clogging of the nozzle and requires operator attention.

In an observation period of over a year in operation, the lance has convinced with stable operating parameters.

THE AIR PRODUCTS PROCESS INTELLIGENCE SYSTEM (APPI)

In addition to the smart lance, Härtetechnik Steinbach is also taking advantage of the APPI system with the aim of increasing productivity and reducing costs. The system is a cloud-based system for retrieving and visualizing process data, as shown in **Fig. 4** and **Diagram 5**.

The advantages of the system are manifold, the most important ones are listed on the next page:

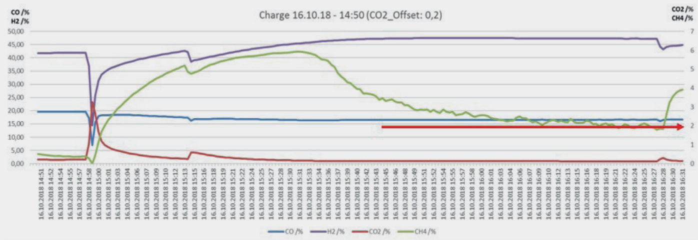

Diagram 3: Example of a batch analysis with the smart lance

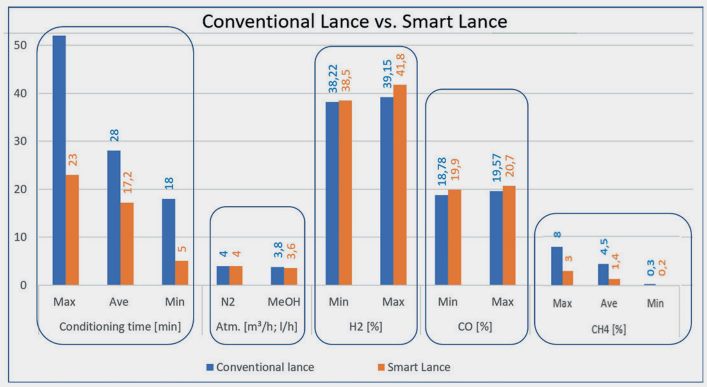

Diagram 4: Direct comparison between conventional lance and the smart lance

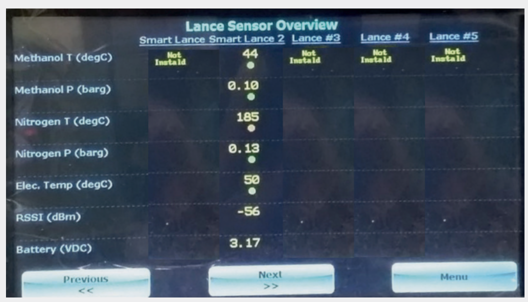

Fig. 3: Screen shot of the smart lance status

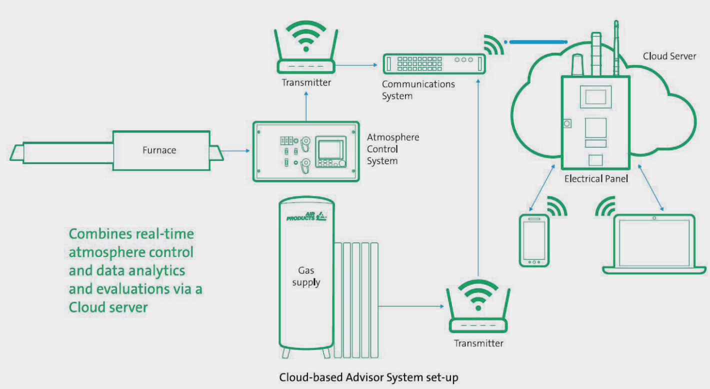

Fig. 4: APPI functional diagram

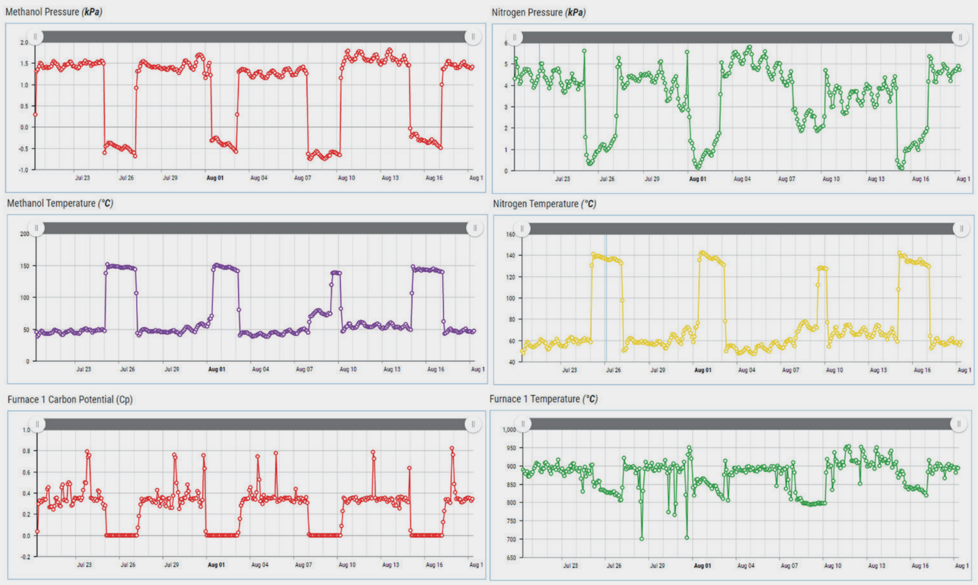

Diagram 5: Online visualization of APPI data

- Visualization and documentation of the process parameters and the atmosphere optionally, also supporting CQI-9 regulation.
- Historical trends and comparisons are carried out, which evaluate the operating parameters and enable process optimizations.
- Online remote monitoring of important process data, access from any Internet-enabled PC or telephone
- Alarms via e-mail or SMS.
- Building historical trends for critical performance indicators with presentation of anomalies.

SUMMARY

Air Products' innovative smart lance and APPI system in combination is already being used in the field to achieve optimal atmosphere results, process monitoring and visualization including documentation and possible preventive maintenance.

In the process, the following advantages were realized in real applications at Härtetechnik Steinbach:

- Provision of a reliable protective atmosphere supply to produce components within a tight tolerance range.
- An increase in productivity due to fewer burnout cycles and shorter forming times.
- A reduction in rework.
- A reduction in the cost of the atmosphere.
- Documentation of the atmosphere and process parameters also in line with CQI-9 standards.
- Indication for carrying out preventive maintenance during planned shutdowns, reducing lost time for troubleshooting during furnace failures.

AUTHORS

Maximus Akuh
Air Products GmbH
Hattingen, Germany
+49 (0) 2324 / 689-300
akuhm@airproducts.com

Tim Nitsch
Air Products GmbH
Hattingen, Germany
+49 (0) 2324 / 689-206
nitscht@airproduct.com

Guido Plicht
Air Products GmbH
Hattingen, Germany
+49 (0) 2324 / 689-300
plichtg@airproduct.com

Felix Steinbach
Härtetechnik Steinbach GmbH & Co KG
Wuppertal, Germany
+49 (0) 0202 / 87053-049
f.steinbach@haertetechnik-steinbach.de