Air Products Smart Technology for Sintering Atmosphere Optimization

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Abstract

Continuous sintering of metal parts under a nitrogen/hydrogen atmosphere is one of the most complicated heat treatment processes. Although the industry has established standard processing methods, the technical challenges still remain to minimize part defects, rework, and the excessive use of energy.

Sustainability is one of our social responsibilities, requiring continuous improvement to engineer technologies and processes to reduce waste and emissions, and minimize our use of resources.

Air Products, Bleistahl and K2 have partnered to develop a cloud-based process optimization system dedicated to sintering. This Industry 4.0 tool enables collection and analysis of the primary process parameters, linked to operational results, facilitating process optimization and product quality improvement. Air Products Smart Technology is integrated with thermodynamic models, field experience, and documentation features. This tool provides process engineers with extended capabilities to manage the production, and support process troubleshooting, audits, and CQI-9 compliance. In this article, results of our development work on this project will be presented and discussed.

Introduction

In recent years, the reduction of CO_2 footprint has become one of the main drivers of continuous process improvement across the heat treatment industry, calling for more sustainable approaches to manufacturing with the ultimate goal of transitioning into carbon-neutral factories. Today, it is widely recognized that environmental responsibility is becoming a new norm and an integral part of product quality on the competitive global market[1,2]. On the other hand, it is of ever-growing importance, to ensure that production facilities remain competitive by keeping their costs down and entering new markets.

The first steps on the path towards achieving this goal do not necessarily equal significant expenditure. Improving the efficiency of the manufacturing operations and optimizing the process, can result in less production waste (i.e., rejects), saved manufacturing time (i.e., to avoid rework), reduced use of resources and lowered energy consumption. All these aspects can provide significant savings and at the same time contribute to reduction of CO_2 footprint/kg and lead to improved quality of the final product[3,4].

The transitioning to green manufacturing via process optimization requires heat treaters to look at the long-term operation performance to identify bottlenecks, areas for improvement or perform troubleshooting. As a result, more attention is given to real time process monitoring, data acquisition and analytics.

Sintering furnaces and atmosphere

The selection of the type of sintering furnace used in production is dictated by a few main (aside of the economics) aspects that include the characteristics of materials and parts to be treated in the furnace, type of protective atmosphere used, and temperature required[5].

In automotive industry, where the great mechanical properties need to be achieved within tight tolerances, the frequent furnace choice is a continuous mesh belt type with an internal muffle. The mesh belt type allows not only for achieving sufficiently good parts' quality to meet the stringent requirements of automotive standards, but also high throughput and for more consistent results as compared to i.e., batch type production. One of the most common materials used for the belt in continuous sintering furnace is austenitic stainless steel AISI 314 (**see Table 1**)[5, 6, 12].

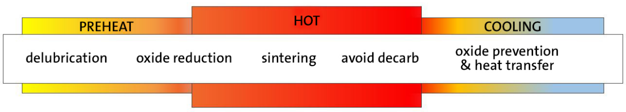
Table 1 – Chemical composition of the AISI314 stainless steel used for sintering furnace belt[12]

Element	C	Mn	P	S	Si	Cr	Ni	Fe
wt.%	0.25	2.00	0.045	0.030	1.50 – 3.00	23.00 – 26.00	19.00 – 22.00	Bal.

When selecting the atmosphere for continuous sintering, a few criteria must be considered. Most of the powders require some level of protection against in-process high temperature oxidation, as well as removing the oxides already present on powder particles prior to sintering, that could negatively impact diffusion bonding and infiltration. Additionally, the right atmosphere in preheat zone should ensure removal of lubricants and binders added to maintain the green part's post-pressing shape and control the carbon activity of the atmosphere to avoid sooting. If required, the atmosphere needs to be adjusted to avoid and minimize risk of partial carburization or decarburization[4, 5, 7, 9].

As a result of continuous operation and dynamics of the numerous reactions, the atmosphere in the sintering furnace may be quite unique and its function will depend on the location inside the furnace. Three main zones can be distinguished: preheat, hot and cooling (**see Figure 1**) where the local "micro-environments" are created. Each of the sections is critical to the successful sintering and high-quality final components, and therefore should be strictly controlled[4, 9]. The most popular choice for sintering atmosphere is blend of varying concentrations of nitrogen and hydrogen, which may involve zone-specific modifications tailored to the process and material requirements[3, 4, 10, 11].

Figure 1 – Continuous sintering furnace diagram with the three zones distinguished and main atmosphere functions associated with local micro-environments



Sintering process monitoring

Several variables, i.e., powder particle morphology and size, chemical composition, green part density, temperature-time profiles, and heating rates, have strong influence on the part quality and productivity. Most of them can be specified at an early stage of the design process. The atmosphere as another process variable is often overlooked, despite its key influence on the final quality of the components and benefits that it can bring when properly controlled. It is important to highlight that due to the nature of reactions taking place during sintering (delubrication, oxide reduction, decarburizing or carbon restoration) and contaminations carried in with the parts, the composition of the atmosphere is dynamically changing within the local microenvironments and along the furnace length. As a result, the gas blend introduced into the furnace is rarely the same as a process gas composition sampled from furnace during production[4, 5, 6].

To realize the benefits associated with correct process atmosphere, it is imperative to continuously monitor the furnace atmosphere composition in respect to its KPIs for oxidation- and carburizing reactions like H_2O/H_2, pO_2, CO_2/CO and carbon activity.

Including other additional parameters into furnace monitoring system could result in holistic approach and even better process understanding. Among the parameters, the following should be considered[4, 7]:

- Atmosphere flow rates
- Inlet gas composition
- Atmosphere pressure distribution over the length of the furnace
- Flow distribution from introduction point into furnace zones
- Atmosphere exit and entrance velocity
- Furnace loading
- Exit part temperature
- Belt speed
- Furnace design features (curtains) and door opening level
- Ambient conditions

In most cases, if monitored, the sintering atmosphere quality indication relies on dew point or oxygen partial pressure measurements only, assuming fixed blends and stable conditions inside the furnace. As already indicated, one parameter or single point measurement approach does not reflect the actual dynamics of the process atmosphere and therefore more atmosphere and furnace parameters should be monitored to assess the furnace and atmosphere efficient and sustainable deployment[7].

Digitalized heat treatment and Industry 4.0 tools

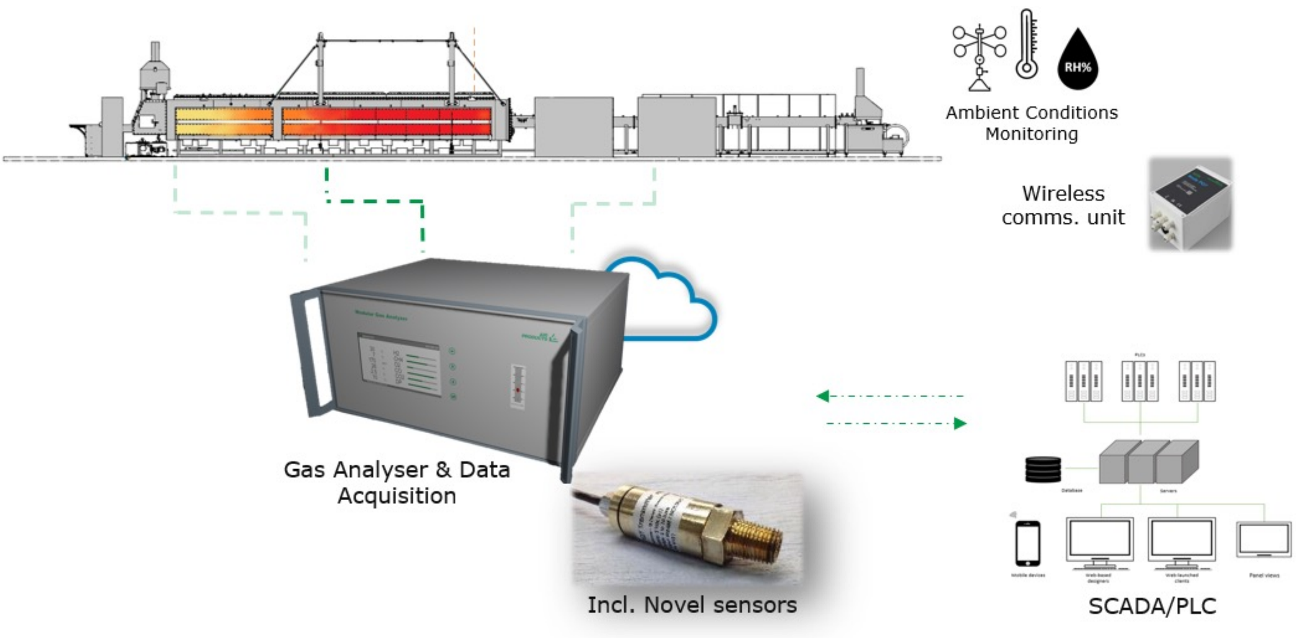
Air Products, Bleistahl and K2 Digital Transformation have partnered to design and test an Industry 4.0 optimization tool dedicated to sintering furnaces. It is a cloud-based system for continuous monitoring of the furnace condition including (but not limited to) process atmosphere, treated products, furnace loading and external conditions. The system consists of multiple atmosphere monitoring stations located in different zones of the furnace, as well as measurements in various points along its length. The atmosphere monitoring stations are connected (both, wired and wireless) to the data acquisition unit which sends the collected process data to the cloud server at the pre-set time intervals.

The data acquisition unit can communicate with the plant or furnace SCADA system, which collects and stores the furnace parameters and production information, under pre-defined communication protocol (MODBUS, OPC) (**Figure 2**). At Bleistahl, more than 70 parameters are logged from the SCADA system on a continuous basis to help create a comprehensive database and ensure traceability of parts in respect to the quality of the atmosphere and process conditions.

Cloud server computing capabilities allow integration of calculations involving multiple measured parameters for more accurate process monitoring and quicker optimization. The system installed at Bleistahl includes various software packages designed to perform real-time calculations of the Atmosphere Key Performance Indication. Some of them include the atmosphere redox potential from critical furnace zones, where the relevant reactions could take place or the model analyzing relevant process parameters to calculate distribution of the gas flow and its impact on process quality.

An important aspect in process troubleshooting and optimization is to not only monitor or calculate the instant process parameters, but also record and study the long-term behavior of the system and deviations from desired process conditions. It is especially relevant to issues that could develop and worsen gradually over time i.e., muffle cracks and leaks or belt damage through oxidation (**Figure 6**).

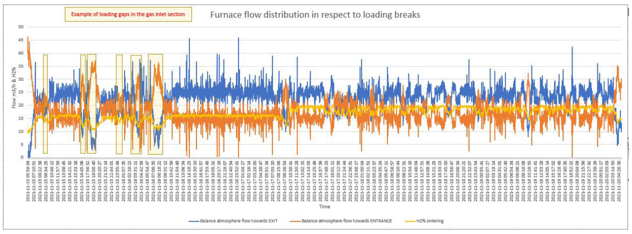
Figure 2 – Cloud based sytem layout integrated with local SCADA system and optional atmosphere control unit



Case study 1 – Evaluation of furnace flow distribution

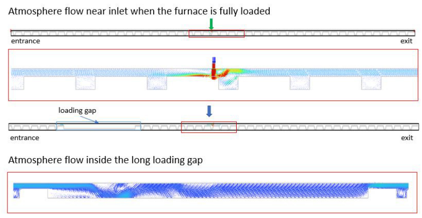
With this new tool and collected process data, a digital model was developed to calculate dynamic flow distribution of the furnace atmosphere. It was observed that atmosphere distribution not only depends on the furnace design features, but also is largely driven by furnace loading conditions. An example of the change in flow distribution is shown in **Figure 3**. The yellow rectangles marked on the flow graph represent loading gaps that have reached the gas inlet position in the furnace. It can be noted that shifts in gas flow distribution are associated with lack of parts and flow restrictions that are created under different loading conditions. The longer the break, the more significant impact it has on the changes in furnace atmosphere flow distribution.

Figure 3 – Calculated gas flow distribution from gas inlet position towards entrance (sintering zone) and exit (cooling zone) in respect to loading gaps present at gas inlet



The analytical model predicts the changes of furnace atmosphere flow distribution based on loading patterns, atmosphere condition and hydrogen concentrations in different furnace sections. The driving principles and flow dynamics were also confirmed with dedicated CFD modelling (**Figure 4**). The simplified 2D model does not encompass all the details of gas injection, flow setup or ventilation, but it shows that in the presence of loading gaps in the hot zone of the furnace, the flow towards entrance increases. It can be associated with a volume of gas contained within the gap (proportional to the gap size) and fewer restrictions in that area that encourage the flow in that direction.

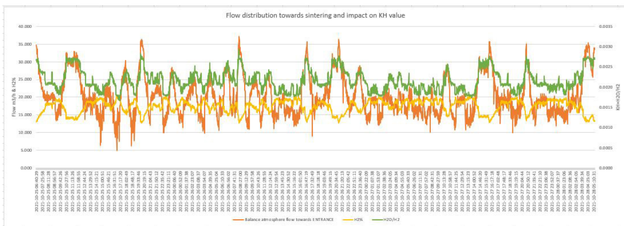
Figure 4 – Simplified CFD model of different furnace loading conditions and impact on gas flow distribution



Flow rate (kg/s)	Towards entrance	Inlet	Towards exit
Full production load	0.02536 (51%)	0.04955	0.02419 (49%)
With loading gap	0.02799 (57%)	0.04955	0.02157 (43%)

Moreover, it was observed that re-distribution of furnace atmosphere and presence of the loading gap, change the dynamic of the flow (velocity) and level of mixing between gas introduced from two supply lines available in this furnace, nitrogen/hydrogen blend injected towards sintering zone and pure nitrogen injected towards cooling zone. As a result, the amount of hydrogen varies in the range of 11-18.5 vol% in sintering zone and 1.5-8 vol% in the cooling zone and leads to fluctuations in the redox potentials of respective furnace sections and increased ratio of water to hydrogen by up to 10 times its regular value (**Figure 5**).

Figure 5 – Impact of the change in flow distribution and gas mixing on the H_2% and atmosphere redox potential (KH) of furnace sintering zone



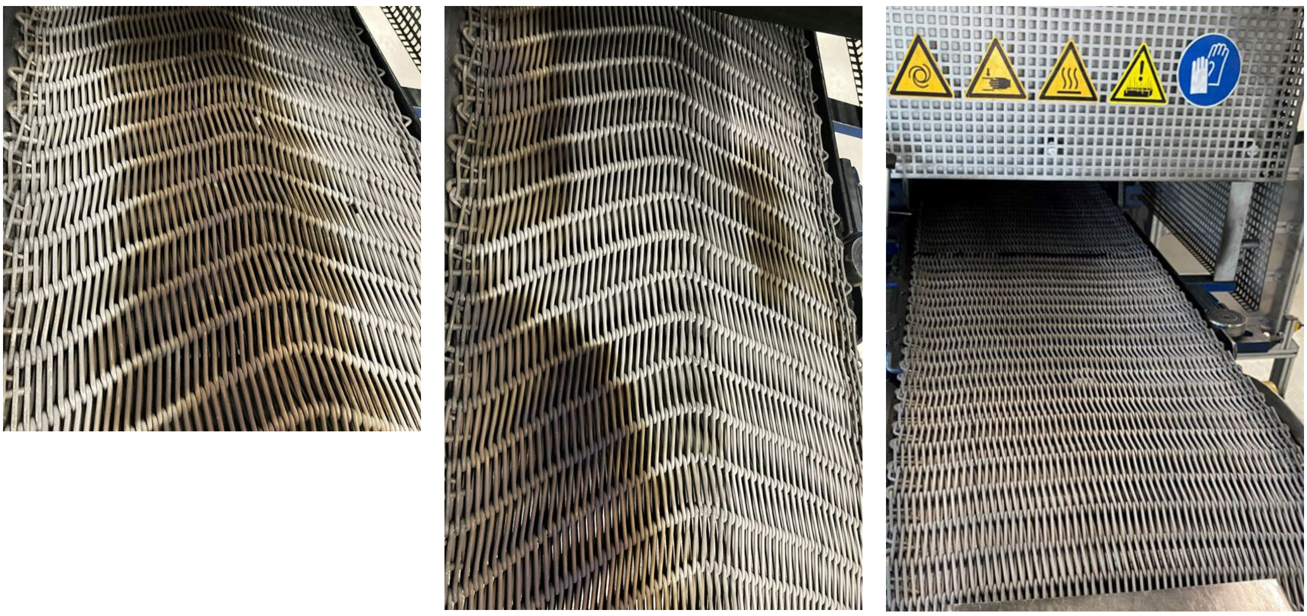
Case study 2 – Belt life extension

Stainless steels are protected from corrosion and friction damage thanks to passive layer on its surface. Typically, this is done by creating uniform chromium oxide (Cr_2O_3) layer and maintaining it throughout the sintering cycles. However, in a continuous sintering furnace, often due to lack of belt conditioning, changes in atmosphere redox potential and variations in gas mixing and distribution, the passive layer can be destroyed, and belt undergoes cyclic oxidation and reduction. As a result, depth of chromium-depleted zones and areas of internal oxidation increase, and therefore reduce the material strength and shorten lifetime of the belt. One of the alterations used for furnaces with nitrogen-hydrogen atmospheres is the addition of moisture into furnace hot zone. Increasing the hot zone dew point helps to maintain a protective layer of chromium oxide on the surface of the stainless steel belt throughout the full cycle, and results in an extension of its service life[12]. Any of the implemented modifications should be monitored and controlled in respect to the part material composition and atmosphere requirements to prevent a detrimental effect on the product quality.

An impact of the increased oxidizing potential of the atmosphere in hot and cooling zone of the furnace was also clearly visible on the surface of the stainless steel belt (**Figure 6**). This belt was not conditioned prior being installed so the passive layer available on its surface at the beginning of its service life was reduced over time in hot zone under dry atmosphere. As a result, the belt surface became gradually exposed to possible reactions: oxidizing (by water), carburizing (by delubrication products) and nitriding (nitrogen in highest temperature zones). As previously indicated by Wehr-Aukland and Bowe[12], such chemical reactions happening on the stainless steel belt surface could affect the long-term mechanical properties and longevity of the belt, and put an additional strain on use of resources and production costs.

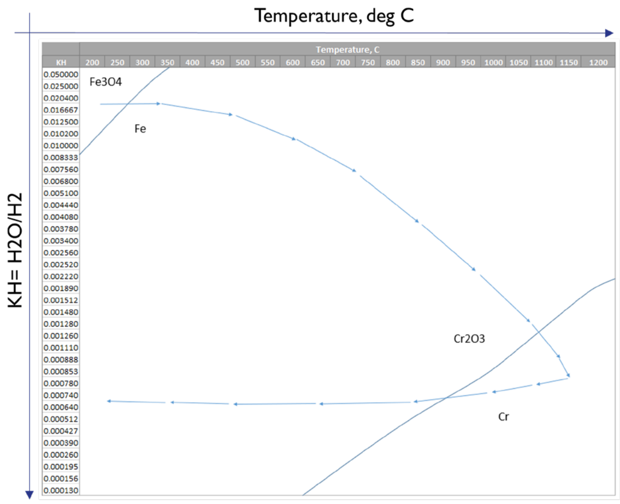
Figure 6 – Belt surface at the exit of the sintering furnace, a) & b) different patterns of oxide layer resulting from flow dynamics and changes in atmosphere oxidizing potential c) oxide free surface (fully reduced oxide layer)

a) **b)** **c)**



The oxide layer created during sintering cycles is very irregular and the oxidized areas on the belt surface change from cycle to cycle (**Figure 6**). Thanks to the atmosphere and process data collected through Air Products Smart, the changes were tracked to variations in gas distribution and periodically increasing oxidation potential in sintering and cooling zones of the furnace. Calculated ratios of water to hydrogen available from process data were verified with FactSage thermodynamic software. The lower water to hydrogen ratio is observed during the production when the furnace is in regular full loading mode. Such furnace atmosphere has a strongly reducing potential to remove oxides at the belt surface. It also indicates that increased water to hydrogen ratio in the hot and cooling zone could result in maintaining the oxides created in previous cycles or even further buildup of the scale area (or thickness) leading to uneven layers of oxides present on the belt surface at the end of sintering cycle (**Figure 7**).

Figure 7 – FeNiCr-Cr_2O_3 oxidation- reduction curve as function of temperature and H_2O/H_2 ratio. The arrows show an example of changing H_2O/H_2 ratio upon heating that the belt material could experience during a full sintering cycle



FactSage simulation results for given stainless steel composition and varying H2O/H2 ratios in N2/H2 atmosphere.

No detrimental effect on part quality was associated with the variations in the water to hydrogen ratios of the hot and cooling zones due to the nature of material composition. It was also proven that simply reducing the hydrogen flow into the furnace in order to increase water to hydrogen ratio is not sufficient to overcome the variation in flow distribution and belt oxidation issues, and could result in reduced part quality. Consequently, the furnace hydrogen flow is set at levels higher than required to cover "the worst-case scenarios" and to provide sufficiently high hydrogen concentrations (and low water to hydrogen ratio) to ensure high part quality for all alloys processed in the furnace.

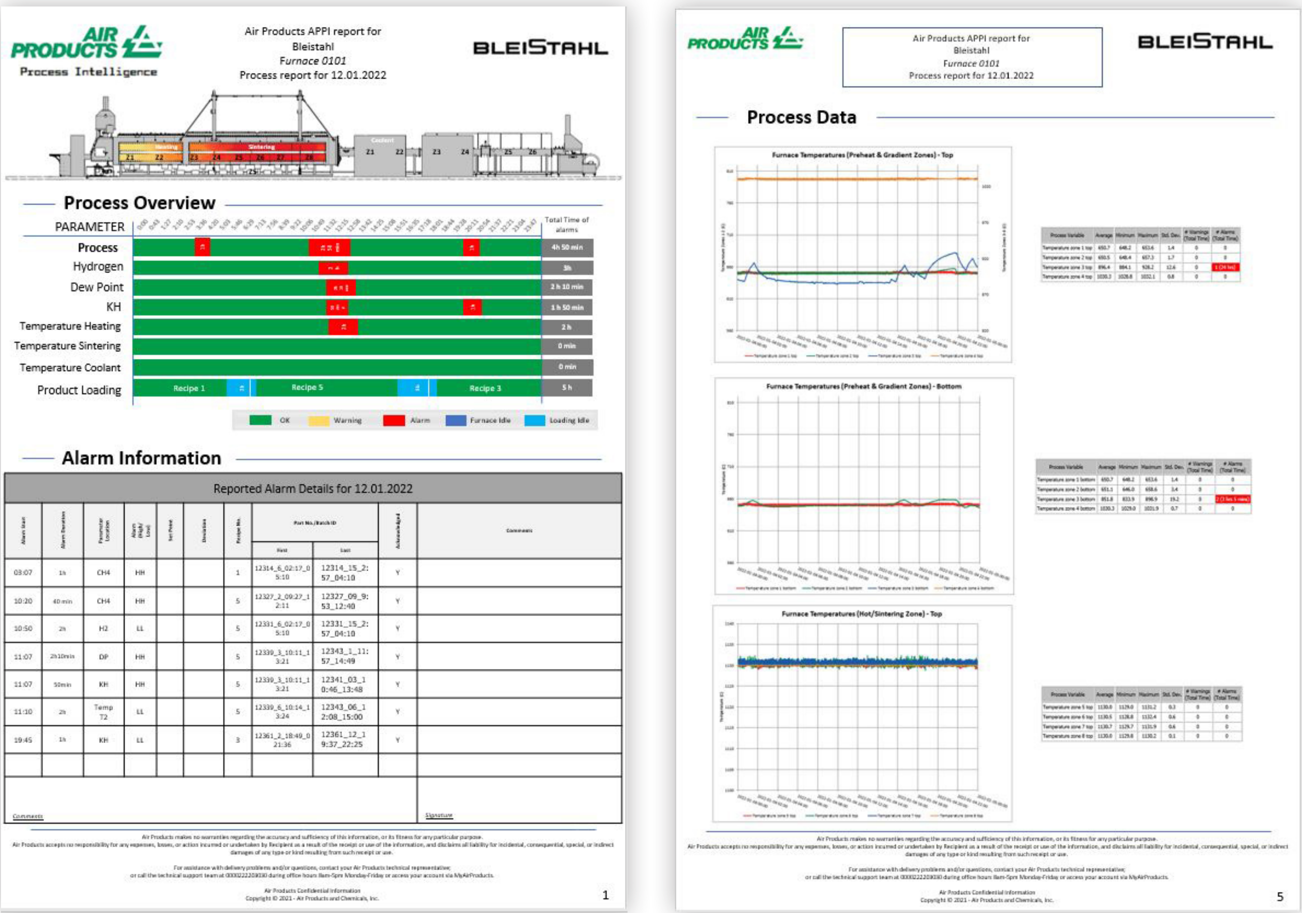
Based on preliminary study, it is expected that implementation of local, active control into the gas supply system based on the predictive flow distribution models could bring benefits in the form of improved part quality, more efficient use of protective atmosphere, and even possible savings associated with reduced, but actively controlled and distributed hydrogen concentrations.

Implementation of conditioning of the belt and humidification system to extend the lifetime of the belt could bring additional savings by reducing the frequency of an expensive belt replacement.

Case study 3 – System generated process documentation

In addition to real time process monitoring, the Air Products Smart Technology system provides process documentation compliant with CQI-9 audit requirements. The pdf report generated daily (**Figure 8**), tracks all the alarms established for process and atmosphere parameters within tight tolerances to ensure that any out-of-range conditions are captured by the system. It also creates an indication of all parts that could have been affected by these alarms in respect to the value set points established for the recipe with which the parts were treated, and their position in the furnace at the time of alarm. Other sections of the report include summary of all products treated in a day, recipes used in a furnace, production summary, utility and supply system monitoring, and graph section for selected parameters.

Figure 8 – Example of reporting



Summary

Continuous measurement, real time modelling, control, documentation and process analytics of sintering furnace atmospheres is becoming increasingly important to reduce reject rates, increase productivity and enable taking the first steps on the path towards “green factory“ and carbon neutral production. Equipping the furnace with a cloud-based monitoring system such as Air Products Smart Technology, permits not only for monitoring of the instant production conditions, but also long-term trends and changes from normal conditions that speed up troubleshooting, enable root-cause analysis and decrease the risk of repetitive failures resulting in production shut-downs or out-of-spec parts.

Data collection and analytics also allow for generating process specific algorithms like the newly developed flow distribution model for continuous mesh belt sintering furnaces. Implementation of such models indicates which parts could be exposed to lowered quality-atmosphere or when the atmosphere gas is used inefficiently due to i.e., uneven distribution inside the furnace, and therefore allows for further development of control models that could prevent this from happening, enhancing process predictive maintenance.

Addition of the furnace and production data via SCADA or PLC integration facilitate taking the optimization a step further and creating a direct link between atmosphere conditions, operational results, and energy consumption. Sintering atmosphere and process data can be successfully used to optimize the process, decrease the operating costs by reducing the number of production rejects and therefore reduce energy and resource demand/kg (i.e., hydrogen) of the product and result in lowered CO_2 footprint/kg parts.

Speak to one of Air Products representative to learn more how Air Products Smart Technology can help you optimize your process and generate a cleaner future.

Note: This paper was originally presented and published as part of World PM2022 Congress proceedings.

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