# SIEMENS

# ANALYTICAL PRODUCTS Optimize Heat Treatment Process

Continuous measurement for improved and consistent steel quality

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Annealing furnaces require heat and specific operating atmospheres to ensure desired carbon content in steel. The specific atmosphere depends on the type of heat treatment application. The continuous measurement and control of specifically CO, CO<sub>2</sub> and methane is of importance to achieve the desired quality. The Ultramat 23 applies NDIR to measure the concentration of those components on-line and continuously. Because the sample does not come in contact with the measuring sensor, even corrosive furnace gas can be analyzed without consequences. Simple calibration with ambient air, long term repeatability and low cost of ownership enables consistent steel quality.

Heat treating are processes in which metallic workpieces are subjected to temperature changes in order to manipulate certain material characteristics, in particular surface characteristics. The composition of the surrounding atmosphere is of prime importance for these applications and must be controlled and monitored on a continuous basis using gas analyzers in order to achieve the designed material quality and consistency.

There are two main types of heat treating processes, both of which require a chemically controlled atmosphere:

- Processes in which heat treating is required to create a certain crystalline structure (tempering, hardening) and to prevent surface oxidation (O<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O). This is generally achieved in a controlled, neutral atmosphere.
- Processes in which the specific hardness and adhesion properties of the surface are manipulated during the heat treating process by using a defined atmosphere.

Application examples include treatment of parts for the automotive industry, aerospace technology, and the manufacture of tools and fittings to mention just a few. A general distinction is made between flow-type and batch furnaces. Depending on the size of the parts and the required throughput, heat treating furnaces can be rather large. Test pieces are checked regularly in laboratories by metallurgists. Though there is a wide range of information available on heat treating in different atmospheres, the optimum temperature, cycle times, and atmospheric conditions still have to be determined for each workpiece specification. These conditions must be controlled as accurately as possible and monitored continuously during the process. After the heating process, the parts generally (except washing for example) do not need to be reworked.

# Carburizing

Carburizing is the standard method used to increase the surface hardness of steel components by enriching the carbon contained in the surface at high temperatures.

The "carbon potential" is a measure of the ability of the atmosphere in the furnace to impregnate the surface of the workpiece with carbon and is proportional to the CO/CO<sub>2</sub> ratio in the furnace atmosphere. Typical carburizing furnaces use a primary mixture of 40% nitrogen and 60% methanol. The methanol content dissociates at 1100 °C (1380 °F) to a suitable atmosphere comprising 20% CO, 40% H<sub>2</sub>, and 40% N<sub>2</sub>. Impurities of oxygen (e.g. caused by leakages in the furnace or impurities in the nitrogen supply) lead to the formation of CO<sub>2</sub> and H<sub>2</sub>O, which alter the ideal carbon potential and thus affect the quality of the treated material. This however, can be compensated for by adding methane to the process.

# Decarburizing

Decarburizing leads to a reduction in surface hardness by removing carbon from the surface in an atmosphere with a CO/  $CO_2$  ratio opposite to that required for carbonization.

There are various heat treating processes to achieve specific objects. Table 1 lists typical examples.

Process	Measuring Component	Typical Range	Analyzer	Objective
Brazing	со	0-20%	Ultramat 23	Control
	CO <sub>2</sub>	0-10%	Ultramat 23	
	CH₄	0-5000ppm	Ultramat 23	
	H <sub>2</sub> O	0-1000ppm 0-10,000ppm	Ultramat 6	Bluing
	H <sub>2</sub>	0-20%	Calomat 6	
Carburizing	со	0-35%	Ultramat 23	Carbon Potential
	CO <sub>2</sub>	0-4%	Ultramat 23	
	CH4	0-10%	Ultramat 23	
	H <sub>2</sub> O	0-1000ppm 0-10,000ppm	Ultramat 6	
	H <sub>2</sub>	0-20%	Calomat 6	
Ferrous Annealing	со	0-10%	Ultramat 23	Carbon Potential
	CO <sub>2</sub>	0-1%	Ultramat 23	
	CH4	0-5000ppm	Ultramat 23	
	H <sub>2</sub> O	0-1000ppm 0-10,000ppm	Ultramat 6	
	H <sub>2</sub>	0-20%	Calomat 6	
Nonferrous Annealing	H <sub>2</sub>	0-10/100%	Calomat 6	Control
	H <sub>2</sub> O	0-1000ppm 0-10,000ppm	Ultramat 6	
Malleableizing	со	0-20%	Ultramat 23	Control
	CO <sub>2</sub>	0-20%	Ultramat 23	
Nitriding	NH₃	0-10/100%	Ultramat 6	Dissociation
	H <sub>2</sub>	0-100%	Calomat 6	Control
Sintering	со	0-20%	Ultramat 23	Control
	CO <sub>2</sub>	0-10%	Ultramat 23	
	CH4	0-5000ppm	Ultramat 23	
	H <sub>2</sub> O	0-1000ppm 0-10,000ppm	Ultramat 6	
	H <sub>2</sub>	0-20%	Calomat 6	
Tin Annealing	со		Ultramat 23	Control

Table 1: Typical Heat Treating processes, Measurements and Analyzers applied.



Figure 1: Typical flow-type heat treating furnace

# Nitriding

Nitriding is a process in which a surface is impregnated with both carbon and nitrogen to further enhance the surface hardness. Ammonia (NH<sub>3</sub>), in this case, is the additional component in the furnace atmosphere.

# **Measuring Task**

The measuring task is to continuously measure the gas concentrations in the furnace atmosphere as accurately as possible (Figure 1). Thus the required gas composition can be achieved and controlled for high and consistent quality of the treated parts. Increasing knowledge of the processes involved and significant improvements in analyzer technology have expanded the measuring tasks considerably. In the past, a combined oxygen/humidity measurement using an in-situ zirconium probe (also known as carbon probe) was the most common method used, since it provides a direct correlation between the probe signal and the carbon potential. The probes have great advantages, however, their measuring principle leads to difficulties which can affect the accuracy of the measuring values (specifically contamination due to free carbon and catalytic reactions on the electrode surface where CH<sub>4</sub> is reduced to CO and H<sub>2</sub>). Furthermore, oxygen probes require a reference gas which may be contaminated with the result that the measured values may differ from the actual values.

In the 70's, the importance of additional CO and  $CO_2$ measurements was recognized in order to monitor the overall atmosphere in the furnace more accurately and to correct the values of the oxygen probe. Initially NDIR analyzer often required extensive maintenance and manual calibration and were seldom equipped with self-monitoring functions.

It was only in the last decade that methane was recognized as the most important component for carburization, resulting in the need of an additional measurement channel for



Figure 2: Carburization as function of Methane concentration

concentrations >10%. Figure 2 shows the function of methane concentration on the carbon profile in the surface layers of the treated material. Thus, a multicomponent analyzer for simultaneously measuring CO,  $CO_2$ , and  $CH_4$  has become standard.

# **Measuring Solution**

The ULTRAMAT 23 non dispersive infra-red (NDIR) multicomponent gas analyzer is ideal for heat treatment applications, as it enables the three relevant gas components CO, CO<sub>2</sub>, and CH<sub>4</sub> to be measured economically with just one device. Its physical design, which operates according to the single-beam principle using a dual or triple layer detector, ensures a high level of selectivity and accuracy. Thanks to its auto calibration principle using ambient air, it guarantees an extraordinarily high long-term stability of both the zero point and measuring value without the need for expensive reference gases. Calibration using reference gases is only required once or twice a year. The economical fuel cell used to measure the oxygen content has an optimum service life that is specifically designed to withstand the challenges for measuring oxygen in combustion gases. The integrated IO controller, automatic maintenance signal, and remote diagnosis option enable the ULTRAMAT 23 to be easily integrated into automation schemes.

In may be useful to install both a Zirconia probe and an extractive NDIR analyzer to combine the advantages of both methods. Several sampling points along the furnace improve the information about the process and allow better process optimization. Sampling point switching units (e.g. up to 12 sampling points) connected to just one analyzer offer a very economical solution.

Siemens is not just a product supplier for end users and system integrators but provides turn-key measurement solutions. In this case, Siemens takes on full responsibility from engineering, system integration and installation to after sales support.

# Benefits for the user

#### • Economic operation

Only one analyzer for all relevant gas components, minimum analyzer test gas consumption, very low analyzer maintenance requirements. Cost saving by control of the optimum reaction gas flow.

#### Reliability

With its multi-layer detector and the AUTOCAL calibration method, the ULTRAMAT 23 is much less sensitive to interferences.

## Product guality

The use of high performance NDIR analyzers will, according to published experiences, reduce the part waste rate by a factor of two compared to former measuring equipment.

### • System concept

Planning, delivery, installation and after sales support is the responsibility of a single partner.



ULTRAMAT 23 Features	ULTRAMAT 23 Benefits	
Single beam measuring principle together with AUTOCAL ambient air calibration and multilayer NDIR detector technology.	High level of selectivity and accuracy. Long-term stability. Very low consumption of test gas.	
Modular design with 1-3 IR channels and additional oxygen measurement using an electrochemical cell or a dumbbell.	Economical investment and operation.	
Easy cleaning of gas cells. Long lifetime of electrochemical cell.	Minimum maintenance requirements.	
SIPROM GA software for remote control and maintenance. Interface for PROFIBUS PA (Option).	Easy integration into automation systems.	

Table 2: ULTRAMAT 23 Features and Benefits

#### For more information, please contact:

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