Oxygen Doping or Closed Loop Controlled Nitrogen in Reflow Oven.

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ABSTRACT

An inert atmosphere opens the reflow process window. Reflow ovens that have a Nitrogen environment may have better yields because of the lack of oxidation during the process. The wetting properties are improved due to the absence of Oxygen. The drawback of a low Oxygen level is that there is a potential risk for tombstoning. Due to the surface tension this defect is more likely to happen in a Nitrogen oven than in air. To overcome this risk a controlled Oxygen level of 500 – 1000 PPM is preferred. It is not the objective of this paper to define the most efficient Oxygen level, but rather, the method to keep this level consistent during different process conditions. Two concepts are used in the market to keep the Oxygen level in the reflow process constant. One method purges Oxygen into a Nitrogen environment. Depending on the Oxygen PPM level, more or less Oxygen will be doped into the system. The second concept controls the Nitrogen supply to maintain the Oxygen level required. If the Oxygen level goes up, more Nitrogen will be purged into the oven. When there are no boards entering the oven the Nitrogen supply can be reduced and still maintain the Oxygen level. Pro and cons of both concepts are compared.

Key words: reflow, Nitrogen, controls.

INTRODUCTION

Nitrogen is a chemical element with the symbol N and has the atomic number 7. Nitrogen gas is transparent, odorless and is diatomic. Diatomic molecules contain two atoms; for this reason Nitrogen gas is also called N_2.

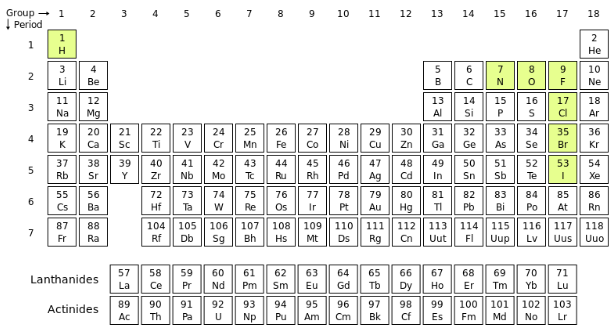

Figure 1. Showing the periodic table with elements that exist as diatomic molecules under laboratory conditions.

If reflow soldering takes place in air there will be by volume 78.09% Nitrogen, 20.95% Oxygen, 0.93% Argon and 0.039% Carbon Dioxide in the process compartment. In addition there is water vapor (approximately 1%) in air. The problem of soldering in air is the 20.95% of Oxygen that may oxidize metal surfaces that need to be soldered. Oxidized metal will not wet and thus solder joints will be insufficient or have no reliable intermetallic contact.

Nitrogen purge can bring the Oxygen level down. The reason why Nitrogen is used:

- It is the cheapest gas
- Nitrogen doesn't react with metal surfaces

Nitrogen doesn't clean the board or remove the oxides. This is the function of the flux in the solder paste. The nuclear weight of Nitrogen is similar to Oxygen and therefore heat transfer properties are identical.

There are three different methods to reflow soldering using Nitrogen:

1. Traditional method: continuously purge a fixed amount of Nitrogen in the oven without any control. The Oxygen level may change when there is more or less production.
2. Purge a fixed amount of Nitrogen and maintain a certain Oxygen level by purging (small) amounts of Oxygen. The Oxygen level is controlled by doping of Oxygen.
3. Purge an amount of Nitrogen into the oven that depends on the Oxygen level. The Oxygen level is controlled by the supplied amount of Nitrogen.

WHAT PPM OXYGEN LEVEL?

The Oxygen level needs to be low enough to prevent or slow down the oxidation of metal surfaces during heating. An Oxygen level that is too low generates the risk of defects. Which level this is depends on several parameters and varies from board to board. The parameters that have effect are:

- Pad dimensions
- Pad patterns
- Solder paste thickness
- Solder paste properties like tackiness
- Reflow profile

There may even be more. For this reason it is clear that the Oxygen PPM level can change from one assembly to the other. This makes the Oxygen PPM level a recipe parameter and not a machine parameter, since it is defined by the assembly.

Studies show that the spreading improves at lower Oxygen levels. For the majority of the solder pastes there is no more spreading when levels are below 1000 PPM. At lower PPM levels tombstoning becomes the critical

factor. Tombstoning will however disappear at a higher PPM level. So the process window will be defined by solder defects (Lower Specification Limit) and spreading properties of the solder paste (Upper Specification Limit).
In order to meet these requirements the supplied Nitrogen should have a ROL (Residual Oxygen Level) of 20 PPM or lower.

HOW TO MEASURE OXYGEN PPM LEVELS

In an inert reflow process the Oxygen levels are not equal in all areas of the oven. Some ovens have multiple sampling points in the different process zones. A switch is used to measure a sample from preheat, peak, cooling and/or cleaning unit part. Due to internal bias of the gas, Nitrogen injection points, and turbulent gas flows, there are differences from one point to the other as well as from zone to zone.
The Oxygen levels are measured using a zirconia electrolytic cell. When voltage is applied to a zirconia electrolyte cell, Oxygen is pumped through the zirconia disc from the cathode side to the anode side because the carriers of the current flowing through the zirconia electrolyte are Oxygen ions. By attaching a cap with a pinhole on the cathode side of the cell and by increasing the voltage over the cell the current shows saturation due to the rate limiting step in the transfer of Oxygen to the cathode. This saturation current is called limiting current and is nearly proportional to the ambient Oxygen concentration. The value is displayed as an analog current signal.

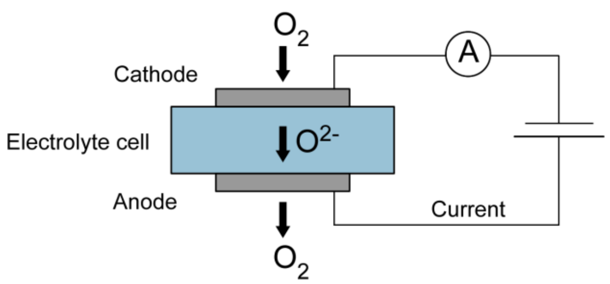

Figure 2. Schematic schema of an Oxygen sensor

A special measurement device was designed to define the Oxygen PPM levels through the complete reflow oven. The device contained a ZrO_2 sensor that was connected to a data logger. Just like a temperature reflow profiler the device was run through the oven measuring the PPM level every second. The accuracy of the measurement was 5%.
For this customer the requirements were an Oxygen level of <750 PPM in the reflow zone. For the soak and peak zone the upper specification limit was 1250 PPM and the lower, 50 PPM. The curve was made for a Nitrogen oven without closed loop control. Oxygen level was measured after a certain amount of Nitrogen was purged. The oven had 9 heating zones and 3 cooling zones. The graph shows the Oxygen level [PPM] in black and the reflow temperature [ºC] in red as measured by the thermocouple mounted on the data logger (measuring the gas temperature). This oven had a dual lane conveyor and the amount of Nitrogen during the measurement was 20 m³/h.

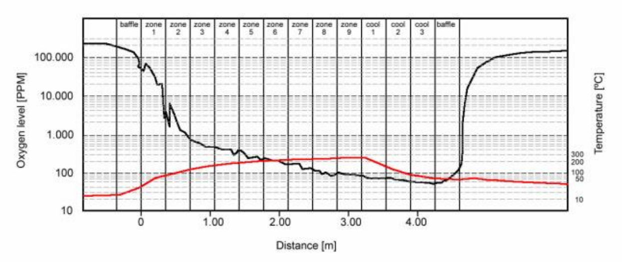

Figure 3. The Oxygen level in the complete oven at board level measured with a profiler.

Nitrogen was purged in the preheating, cooling zone and in the baffles. Although the response time of the sensor is fast, there is a trend when staying longer in the oven. Other measurements with multiple analyzers showed that due to purging and flow, the PPM level in cooling is lower than in the preheat. The target however is to have a low Oxygen level in soak and peak zone as well as in the first cooling zone. In this part of the process, the solder is liquid and oxidation risks are the highest.

BENEFITS OF A CATALYST ZONE CLEANING

One of the main concerns using an analyzer is the damage due to the chemistry of the solder paste. For this reason there is a filter installed before the sensor. A long sample line and a filter make the response time of the analyzer slow and it makes it very difficult to design a closed loop controlled Nitrogen supply.
To overcome the slow response a lambda sensor is a better application for a closed loop system. This sensor is well known in the car industry where it measures the exhaust gas concentration of Oxygen.

Figure 4. The lambda sensor mounted after the catalyst.
The sensor is installed in the last peak zone. To protect the lambda sensor a catalyst is placed in front of the sensor. Both sensor and catalyst are placed outside the zone. Due

to the over pressure in the reflow chamber 16-20 m³/h of gas is flowing through the catalyst and sensor.
So the principle is slightly different. Instead of a fixed sample position the gas is blown out of the zone along the sensor. The gas is continuously cleaned by the catalyst and returned into the zone after the sensor measures the Oxygen level.
The response time of the lambda sensor is very fast and makes it very suitable for a closed loop unit.

HOW TO GET A LOW OXYGEN LEVEL IN A REFLOW OVEN

Before designing a Nitrogen closed loop control for an inert (low Oxygen) reflow process, one should understand what parameters affect the Oxygen level the most and where the Nitrogen should be purged into the oven.
The best way to get a knowledge about the behavior of the oven is to do a Design of Experiment. For this study a ½ Fraction DoE with 4 factors at 2 levels were selected. The oven that was used was a reflow oven with 9 heating and 3 cooling zones. This oven had a dual lane conveyor. These factors were considered:

- Frequency of the blowers (first and last zone): 15 and 25 Hz.
- Baffle zones Nitrogen injection: 4 and 8 m³/h.
- Nitrogen injection in the preheating zone: 5 and 10 m³/h.
- Nitrogen injection in the cooling zone: 10 and 15 m³/h.

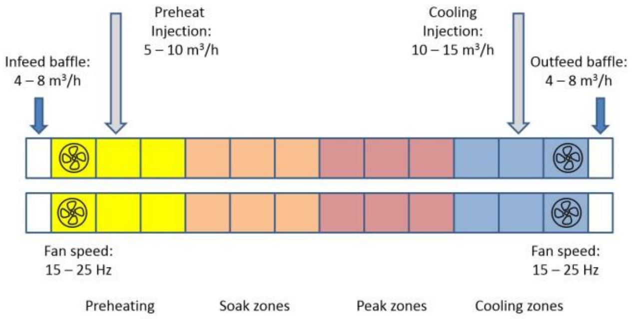

Figure 5. Schematic view of the oven and settings.

A half fraction DoE was selected to have a limited number of runs. Before each run the Nitrogen was purged into the oven for 10 minutes.
The Nitrogen flow was measured with a mass flow meter. The Oxygen levels were measured with a traditional Zr-Ox analyzer. Values were logged after15 minutes. During the experiment the oven was idle. Loading tests were not part of this experiment, but were executed later.

Table 1. Oven settings during experiment.

	Temperature [ºC]		Fan speed
	Top side	**Bottom side**	**[Hz]**
Zone 1	160	160	variable
Zone 2	170	170	40
Zone 3	180	180	40
Zone 4	190	190	40
Zone 5	200	200	40
Zone 6	210	210	40
Zone 7	260	260	60
Zone 8	245	245	60
Zone 9	240	240	60
Cool 1	90	90	40
Cool 2	60	60	40
Cool 3	40	40	variable

The experiment required a total of eight runs. The settings and scores are listed in the next table.

Table 2. Raw data Design of Experiment.

	Freqency [Hz]	**Baffle [m³/h]**	**Preheat [m³/h]**	**Cooling [m³/h]**	**Oxygen [PPM]**	**Total N_2 [m³/h]**
Run 1	25	8	10	15	55	41
Run 2	15	4	5	10	3125	23
Run 3	25	4	10	10	60	28
Run 4	15	4	10	15	40	33
Run 5	15	8	5	15	45	36
Run 6	25	8	5	10	65	31
Run 7	15	8	10	10	55	36
Run 8	25	4	5	15	2750	28

The data shows interaction between the baffle and preheat injection of Nitrogen. A lower Nitrogen supply in the baffle together with a low Nitrogen injection in the preheating makes the Oxygen level drift.

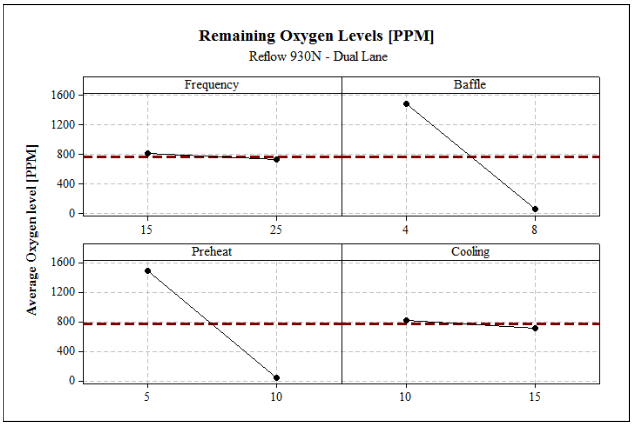

Figure 6. Showing the average Oxygen levels for the different settings.

The frequency of the fans in the first and last zone did not have a significant impact on the Oxygen level in the peak zone. However, it has a major impact on keeping the oven inert. Since there was no difference between 15 and 25 Hz, the set-point was kept at 20 Hz which is a typical value for a Nitrogen configuration.

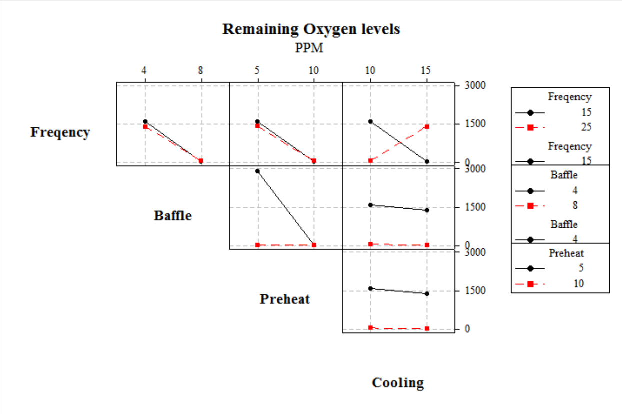

Figure 7. This figure shows the poor performance when the baffle and preheat both have low settings.

The main conclusions from this experiment is that a significant amount of Nitrogen should be purged into the entrance and exit of the oven in order to keep low Oxygen levels in the peak zone. The Nitrogen in the baffle zone acts like a gas curtain preventing outside air from entering the oven.

REQUIREMENTS FOR NITROGEN CLOSED LOOP CONTROL SYSTEM

What Oxygen PPM levels are of interest for the different assemblies has already been discussed. For the specification of a closed loop control system it doesn't make sense to define very tight tolerances. The Nitrogen controlled loop system has to generate a robust and consistent Oxygen PPM level using the lowest Nitrogen amount.
A 20% tolerance is a reasonable value for a controlled application.

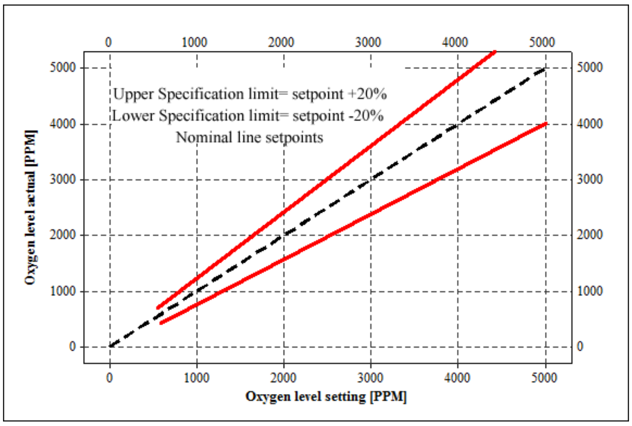

Figure 8. Upper and lower specifications for a closed loop Nitrogen control application.

OXYGEN DOPING APPLICATION

The first closed loop applications in the market are so-called Oxygen doping systems. Nitrogen is purged in the oven to get a low Oxygen level. After purging, the Oxygen level is low and it is controlled by adding Oxygen into the peak zone when the PPM level is too low.
For a better understanding of the amount of Oxygen to be supplied into a 930N oven:

- Ping-Pong ball equals 18 PPM
- Tennis ball is 82 PPM
- Football is 3500 PPM

The amounts are relatively small and for this reason a small proportional needle valve is used.

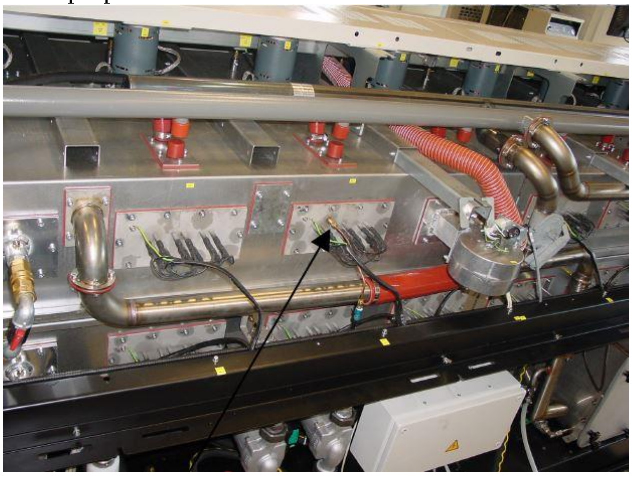

Figure 9. This pictures shows the air doping in the peak zone (arrow).

PURGE CYCLE

Before production start and after opening the hood of the oven there is a Nitrogen purge cycle to get the oven at lower Oxygen levels as fast as possible.
During the cycle the maximum Nitrogen amount is approximately 50 m³/h.

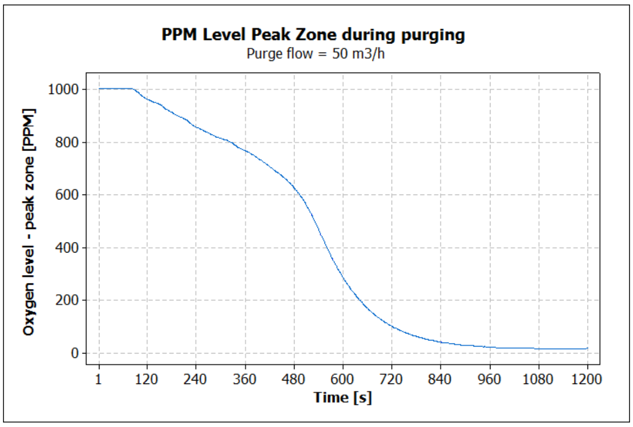

Figure 10. The Oxygen level during a purge cycle of a 930N reflow oven measured with a lambda sensor in the peak zone.

The purge cycle can be defined by the user. The amount of Nitrogen as well as the purge time can be adjusted. For this oven it takes approximately 12 minutes to get the Oxygen level below 100 PPM.
The purge time can be critical, but most likely it takes longer for the zones to achieve the set temperature than to achieve the PPM set-point.

NITROGEN CLOSED LOOP CONTROL

The experiences from the DoE are used for the final design of a closed loop system. The closed loop system should help to control the Oxygen PPM level over time. It should automatically correct any deviations of the Oxygen PPM number.
Essential in the design is that there should be enough flow at the baffles (entrance and exit). In order to minimize the Nitrogen consumption the Nitrogen is not fresh, but recycled out of the oven. At the entrance side the over

pressure in zone 2 and 3 brings the Nitrogen to the baffle and is injected in the top and bottom side. After the Nitrogen (± 16 m³/h) flows out of the zones it is cleaned by a catalyst, otherwise the flux residues would condensate in the baffles and contaminate the oven.
The Nitrogen for the exit baffle comes out of the peak zone. Between the last peak zone and first cool zone the gas is sucked out of the oven by a venture. This so called slot exhaust takes the contaminate gas and guides it through a catalyst before it is injected back into the exit baffle.

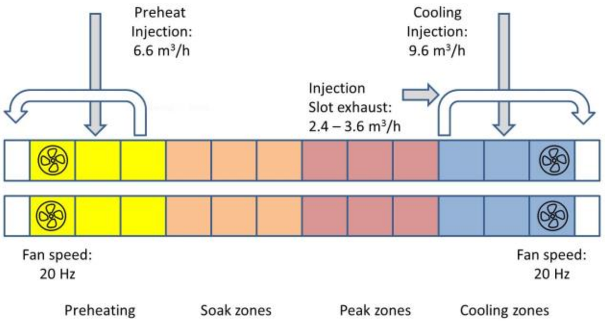

Figure 11. The schematic of the new design with the recycled Nitrogen for entrance and exit baffle.

The first experiments are done manually and are necessary to investigate the impact of the different features. The Nitrogen supply in preheat (6.6 m³/h) and cooling (9.6 m³/h) are kept constant and the amount of Nitrogen blown into the venture is changed from 2.4 to 3.6 m³/h to investigate the impact of this parameter.

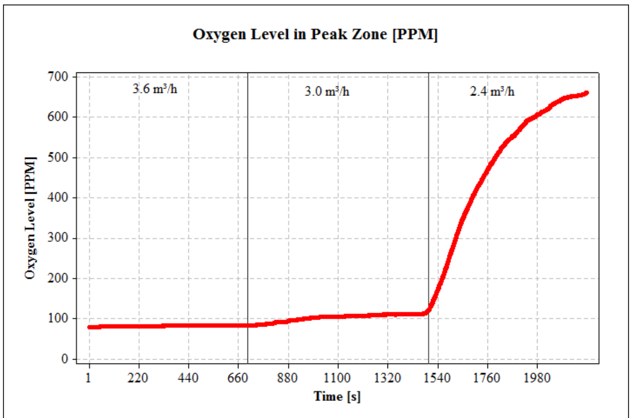

Figure 12. The PPM level changes when the venture injection is changed.

This graph shows that in order to keep the PPM level Oxygen low in the oven at least 3.6 m³/h of Nitrogen has to be purged into the venture.

Table 3. Slot exhaust flow versus Oxygen PPM level in peak zone.

Flow [m³/h]			**PPM**	**ΔPPM**		
Preh.	**Slot exhaust**	**Cooling**	**Start**	**1 min**	**5 min**	**10 min**
6.6	3.6	9.6	80	1	2	4
6.6	3.0	9.6	84	2	19	26
6.6	2.4	9.6	113	4	378	530

To keep the oven clean a fixed amount of gas has to be exhausted out of the peak zone. This amount is achieved when 4.2 m³/h of Nitrogen is purged into the slot exhaust.

For the closed loop system the slot exhaust is fixed and not part of the control. The Nitrogen amount in the cooling should be varied to keep the Oxygen level in the peak at a constant value. To see if this is feasible, the next experiment was done with a fixed preheat (6.6 m³/h) and slot exhaust flow (4.2 m³/h).

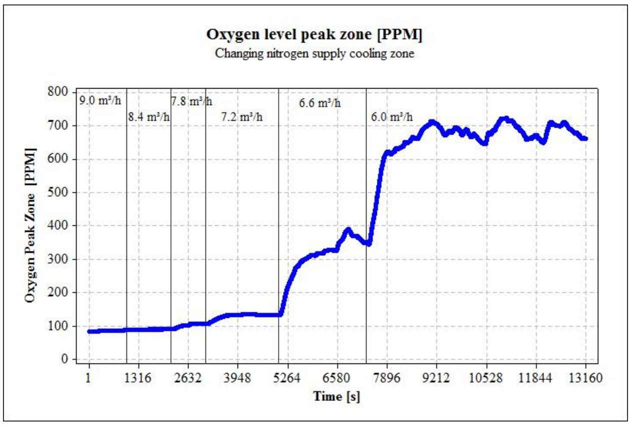

Figure 13. Changing the Nitrogen flow to the cooling is a method to control the PPM level in the peak zone constant.

Table 4. The Oxygen PPM levels in peak, preheat and cooling zone.

Flow [m³/h]			**PPM**		
Preheat	Slot exhaust	Cooling	Peak	Preheat	Cooling
6.6	4.2	9.0	80	100	50
6.6	4.2	8.4	85	100	55
6.6	4.2	7.8	105	110	80
6.6	4.2	7.2	150	124	112
6.6	4.2	6.6	350	267	449
6.6	4.2	6.0	650	500	1055

The PPM level in the peak zone is measured with a lambda sensor, the preheating and cooling with a ZrO_2 analyzer.

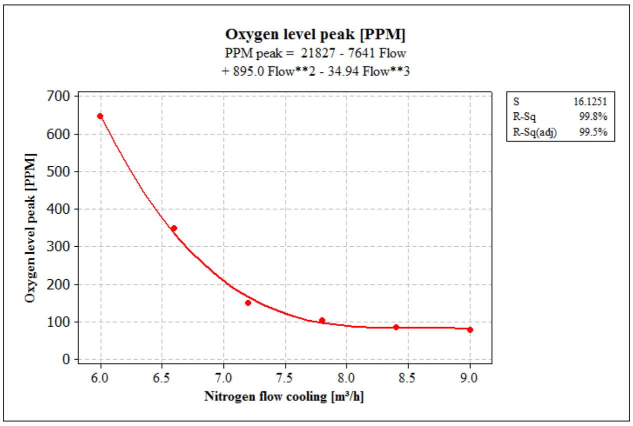

Figure 14. Impact of Nitrogen flow cooling on PPM.

A Box Behnken design of experiment was done to define the next graph showing the Oxygen PPM levels in the peak zone as a function of Nitrogen flow in the preheat and cooling zone.

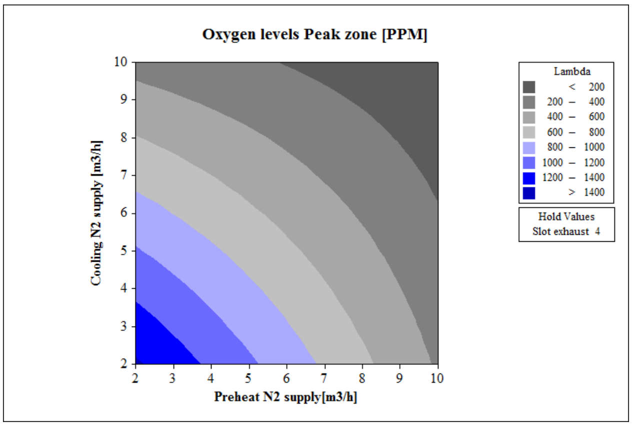

Figure 15. The Oxygen level in peak zone.

The final configuration of the closed loop has a Nitrogen supply in the preheating, a reduced fan speed in first and last zones, and a PID controlled Nitrogen supply in the cooling. The baffles are supplied with Nitrogen out of the oven as described above. The slot exhaust has a fixed Nitrogen flow.

NITROGEN CONSUMPTION AS A FUNCTION OF OXYGEN PPM LEVEL

The closed loop Nitrogen PID will control the Oxygen level in the peak zone by purging more or less Nitrogen in the preheating and cooling zone. The amount of Nitrogen required depends on the status of the oven. It will make a difference if there are many boards in the oven compared to an empty oven. In the next experiments, loading of the oven is included. For this loading condition, aluminum boards (250x250x3 mm) were used and had a spacing of 125 mm in between the boards.

When there are no boards on the production line the oven has the ability to go into an idle mode. In this mode the fan speed of the zones will be reduced by 10 Hz. In case there is a longer production stop, the oven has a sleep mode in which the fans will go into lower mode to save Nitrogen and energy.

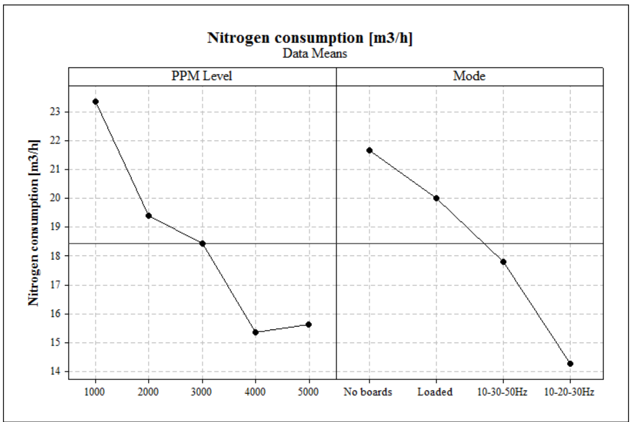

Figure 16. Nitrogen consumption for different oven statuses.

The Nitrogen flow is regulated by the closed loop system. With a mass flowmeter the average amount was measured for 1000, 2000, 3000, 4000 and 5000 PPM Oxygen in the peak zone.

Nitrogen consumption is reduced when the machine goes in idle mode and the power consumption is less.

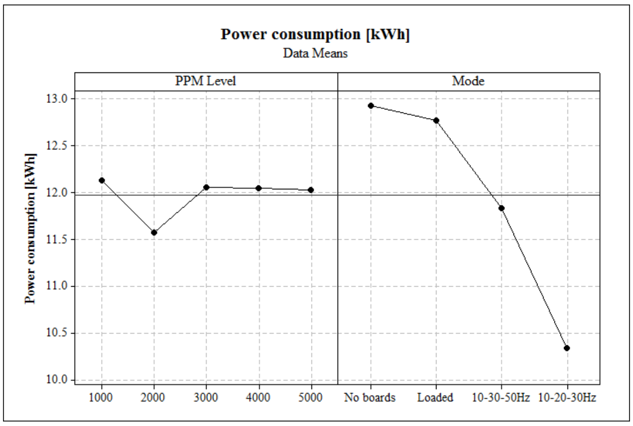

Figure 17. Power consumption for a dual lane 930N oven with catalysts and lead-free profile settings.

As shown in the graph a higher or lower Oxygen level doesn't have an effect on the power consumption.

LOADING TEST DUAL LANE CONVEYOR

The next experiment compares different loading conditions of the oven. The Oxygen level set point for the peak is 1500 PPM. A main board (350x192x1.6 mm) of 250 gram was run through the oven.

The first test showed the results for PPM level and Nitrogen consumption when boards run on the front lane with a spacing of 150 mm. In total 50 boards were reflowed. The profile was a lead free recipe.

Table 5. Reflow set points

	Temperature [°C]		Fan speed
	Top side	Bottom side	[Hz]
Zone 1	100	100	20
Zone 2	130	130	40
Zone 3	150	150	40
Zone 4	150	150	40
Zone 5	160	160	40
Zone 6	170	170	40
Zone 7	200	200	60
Zone 8	240	240	60
Zone 9	240	240	60
Cool 1	90	90	40
Cool 2	60	60	40
Cool 3	40	40	20

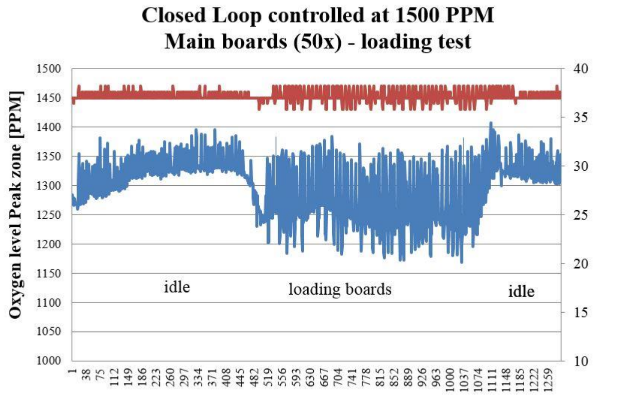

Figure 18. Boards loaded on front rail. Red is PPM level.

In the second part the oven was loaded on both lanes running at the same speed and recipe (boards parallel). A third run was done for both lanes, but with different conveyor speeds. The speed of lane 1 was 160 cm/min and the speed of lane 2 was 80 cm/min. A final run was done on lane 2 only.

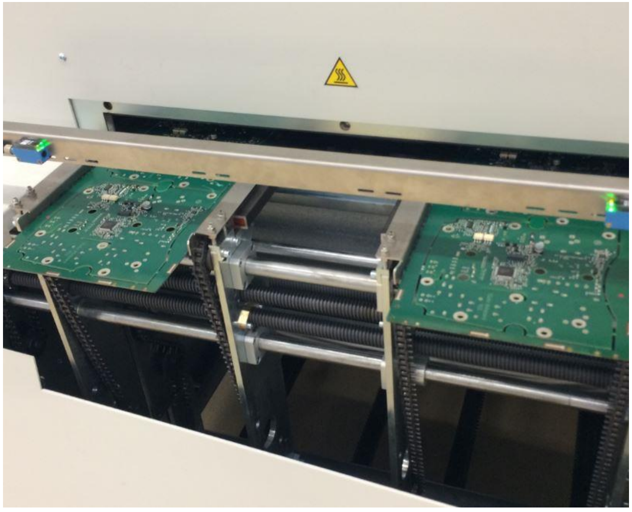

Figure 19. Boards parallel loaded with the same conveyor speed.

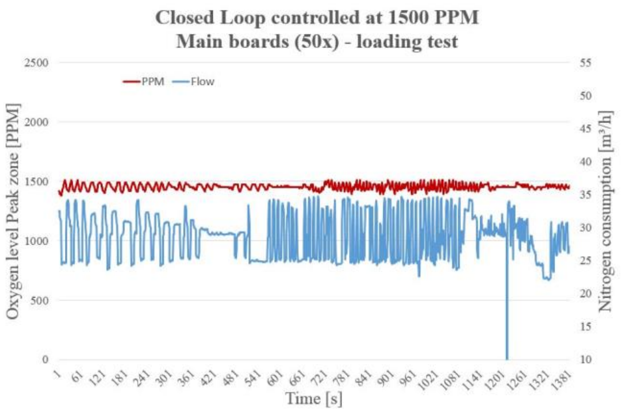

Figure 20. Loaded dual lane both same conveyor speed and boards parallel. Red is PPM level.

The PPM levels and Nitrogen consumption for the different conditions are listed in the next table.

Table 6. Consistency of set point and Nitrogen consumption at 1500 PPM.

		Dual lane		
	Lane 1	Parallel	Dual speed	Lane 2
Set-point [PPM]	Standard deviation [PPM] @ 1500 PPM			
idle	5.4	24.6	17.2	9.7
loading	9.2	27.8	25.5	24.1
idle	9.2	11.0	19.6	12.8
N2 Consumption	Average [m³/h]			
idle	29.54	28.57	26.11	28.72
loading	26.82	28.55	26.34	28.71
idle	27.06	26.22	26.07	29.21

RECIPE CHANGEOVER TIME

The time to change to another PPM level is relatively short. Typical the Oxygen PPM level is part of the recipe and is not changed during production. The only time this value may change is when the line starts to run a different product. Changing to a different recipe in most cases is modifying the temperature settings of the heating zones. This will take a longer time than changing to a different PPM level.
Any changes from PPM levels in the working area of 100 to 2000 PPM will take less than 5 minutes.

SUMMARY

A closed loop system for Nitrogen supply is an option that opens the process window of a reflow process. Since all boards are soldered with identical low Oxygen levels the quality of the reflow products is more consistent and not dependent on loading or other conditions.

The use of a catalyst to clean the gas enables the use of a lambda sensor that responds faster than traditional analyzers.

A closed loop system makes it possible to run the oven in standby or sleep mode when the production is stopped (break, maintenance of machines, lack of components, etc.). The advantage of a standby mode is that the Nitrogen and power consumption will be reduced up to 30%.

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