OX400 Low Concentration Zirconia Oxygen Analyzer

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Targeting the electronics industry, Yokogawa has developed the OX400 zirconia oxygen analyzer capable of measuring a wide range of concentrations from ppm up to 100 vol%O_2. The OX400 has such characteristics as high-speed response and high reliability by using a zirconia narrow tube with proven track record for the sensor. Furthermore, the analyzer was designed so that customers can replace the sensor themselves. To improve maintainability, an aspirator type is provided for gas sampling in addition to a pump type. This report introduces the principle of zirconia oxygen analyzers, and the features, structure, and application examples of the OX400.

INTRODUCTION

In many industries, it is becoming increasingly important every year to measure oxygen concentrations not only to ensure product quality but also to conserve the environment and improve energy efficiency. The required measurement levels of oxygen concentration vary from less than ppm to 100% depending on the application.

Yokogawa has so far provided oxygen analyzers with %-resolution for the process automation (PA) industry, mainly for combustion control. To respond to the increasing demand for measurement of lower concentrations of oxygen in the electronics industry such as manufacturing processes for flat panel displays (FPD) and ceramics capacitors, Yokogawa has developed and released the OX400 low concentration oxygen analyzer, which has the minimum resolution of 0.01 ppm and supports a wider range of measurement from 0-10 ppm to 100% O_2. The OX400 is equipped with a field-proven zirconia sensor and offers excellent reproducibility and high-speed response. In addition, the OX400 analyzer allows users to replace the sensor, making maintenance much simpler.

Figure 1 shows the external view of the OX400 low concentration zirconia oxygen analyzer. This paper introduces the principle of zirconia oxygen analyzers and the features, structure and applications of the OX400.

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Figure 1 External View of the OX400 Low Concentration Zirconia Analyzer

PRINCIPLE OF ZIRCONIA OXYGEN ANALYZERS

At lower temperatures, the crystal structure of zirconia (zirconium dioxide: ZrO_2) usually transforms from cubic to tetragon, and monocline, and the accompanying change in volume during the transition causes the crystal to crack. However, melting bivalent or trivalent metal oxides into ZrO_2 maintains the stable cubic crystal with a fluorite structure, preventing such cracks. Such zirconia containing melted metal oxides is called stabilized zirconia. For example, in zirconia added with 8 mol% yttrium oxide Y_2O_3, oxygen vacancies are created in the crystal and oxygen ions conduct through these holes under high temperature, resulting in high conductivity. It is thus referred to as a solid electrolyte.

Figure 2 shows the measurement principle of the oxygen sensors. The reference gas (ambient air) and sample gas are separated by the stabilized zirconia. Let P_A be the oxygen partial pressure of the reference gas and P_X be that of the sample gas, then an electromotive force E generated between platinum electrodes on both sides of the zirconia is represented by the equation (3). At the electrode on the reference gas side, the reaction expressed by the equation (1) occurs; oxygen molecules receive electrons from the platinum electrode, change to O^{2-} ions, and conduct through the zirconia to the electrode of the opposite side. On the other hand, at the electrode on the sample gas side, the reaction expressed by the equation (2) occurs; O^{2-} ions that have conducted through the zirconia emit electrons and change to O_2 gas, spreading into the sample gas. This conduction stops when the equilibrium between the electromotive force E caused by electron exchanges of oxygen atoms and the concentration difference between P_A and P_X is reached, holding the equation (3) called Nernst's equation. P_X, that is the O_2 concentration of the sample gas, can be obtained by measuring the electromotive force E.

$$O_2 + 4e^- \rightarrow 2O^{2-} \quad \text{........... (1)}$$
$$2O^{2-} \rightarrow O_2 + 4e^- \quad \text{........... (2)}$$

$$E = t_i \bullet \frac{RT}{nF} \bullet \ln\left(\frac{P_A}{P_X}\right) \quad \text{......... (3)}$$

where,

E: Electromotive force
t_i: Ionic transport number (= 1)
R: Gas constant
T: Sensor temperature
n: Valence of electron (= 4)
F: Faraday constant (9.649×10^4 C • mol^{-1})
P_A: O_2 partial pressure (concentration) of the reference gas
P_X: O_2 partial pressure (concentration) of the sample gas

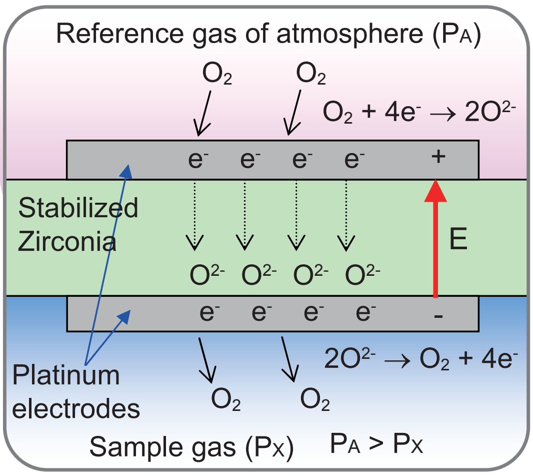


Figure 2 Measurement Principle

FEATURES OF THE OX400

As shown in Figure 1, the OX400 has a simple structure for easy operation: a 4-digit large LED display with good visibility and a large round key at the center of the front surface.

The features of the OX400 low concentration zirconia oxygen analyzer are outlined below.

- High-speed Response
  The zirconia sensor can respond quickly to changes in the oxygen concentration of sample gases because the sample gases flow through the zirconia narrow tube sensor without delay. The sensor achieves a 90% response within 30 seconds.
- Low-concentration Measurement
  The lower the concentration of sample gases is, the more the air inflow from around the sensor affects the measurement. The OX400 can measure low oxygen concentrations precisely by smoothing the zirconia surface and sealing with O-rings.
- Long Life Span
  Because the platinum electrodes formed inside and outside the zirconium narrow tube are long and porous, they hardly deteriorate. The life span of the analyzer is three times longer than that of Yokogawa's existing models.
- Improved Maintainability
  - The sensor can be easily replaced because the inlet and outlet of the sensor are sealed with O-rings and fixed with hand-tightening nuts.
  - The aspirator was adopted in addition to the pump for suction. Since it has no moving parts and so has no limited life components, no maintenance for parts replacement is required.
- Multi-selector Function (optional)
  Contact outputs for a solenoid valve are provided to switch suction points, allowing measurement at up to three points by one oxygen concentration analyzer.

STRUCTURE OF THE OX400

This section describes the structure of the sensor and gas flow pass featuring the OX400.

The structure of the sensor

Figure 3 shows the structure around the sensor. An electric furnace surrounds the center part to keep the sensor at a high temperature (about 700 °C). The zirconium narrow tube is utilized, and platinum thin films are formed inside and outside the tube as electrodes. Sensor signals are drawn from the surfaces of both electrodes. Sample gases flow through the narrow tube, and ambient air serves as the reference gas.

To simplify the replacement of sensors, the inlet and outlet of the sensor are sealed with O-rings and fixed. The sensor can be removed and replaced easily by manually loosening the fixing nuts and pulling the sensor out.

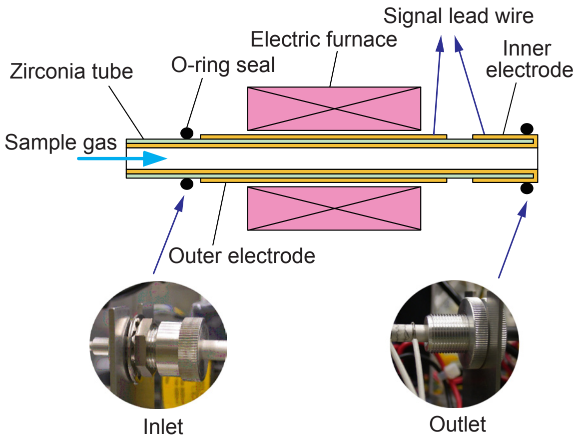
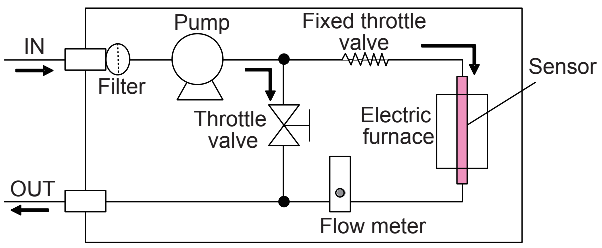


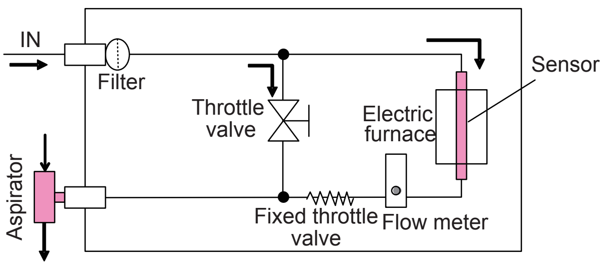
Figure 3 Sensor Structure and O-ring Seal

The Structure of the Gas Flow Pass

Figure 4 shows the structure of the gas flow pass. There are two suction methods: using the pump and aspirator.



(a) Product configuration with the pump suction



(b) Product configuration with the aspirator suction

Figure 4 Configuration of Each Suction Method

In the pump system, the sample gas passes through a dust filter at the inlet and then branches into two streams. One stream passes through a fixed throttle valve, sensor, and flow meter, and then flows out. The other stream passes through the bypass with a throttle valve for flow regulation, and then is discharged. The flow rate of the gas flowing in the sensor is regulated to 200 ml/min by the throttle valve installed in the bypass.

In the aspirator system, the fixed throttle valve is placed next to the flow meter to prevent an excessively negative pressure in the sensor. The aspirator requires air supply from an utility with the pressure of 65 to 100 kPaG. Generally, in the aspirator system, pressurized ambient air (or nitrogen) is added to the gas flow, which increases the flow speed and decreases the pressure surrounding the flow, and the gas is sucked in utilizing the pressure difference between the decreased pressure and that of the atmosphere.

Figure 5 shows the characteristics of gas suction by the aspirator. When the gas constantly flows through the sensor at a rate of 200 ml/min and the air pressure applied to the aspirator is 100 kPaG, the total gas flow rate through the aspirator is about 9 l/min. In this case, the OX400 sucks in the gas at about 1.2 l/min, some of which is conducted to the sensor at 200 ml/min.

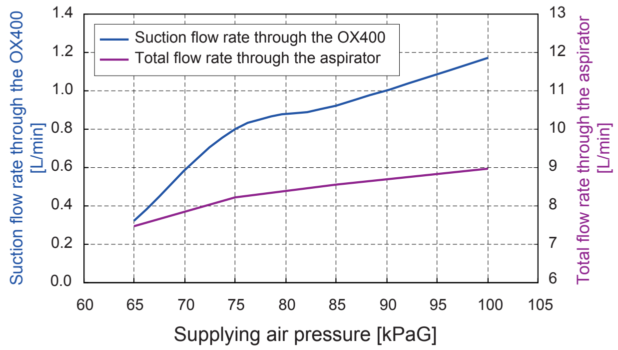


Figure 5 Suction Characteristics of the Aspirator Corresponding to Applied Pressure

APPLICATIONS

Although the OX400 was developed for the electronics industry, it can be applied in a wide range of fields thanks to its ability to measure low-concentration oxygen. The applications include oxygen concentration management in semiconductor diffusion furnaces, semiconductor drying furnaces and liquid crystal display (LED) manufacturing processes; oxygen concentration management in flow solder bathes, reflow furnaces, glove boxes, and gas manufacturing processes; and oxygen concentration measurement to prevent powder explosion during transportation. Some of these applications are described below.

■ FPD Manufacturing Process

Figure 6 shows a schematic diagram of the FPD manufacturing process. This involves a number of processes. The O_2 concentration of the ambient gas is measured and managed in the drying, film forming and annealing processes. In these processes, the O_2 concentration is strictly controlled to be within a range around 10 to 100 ppm to prevent oxidization.

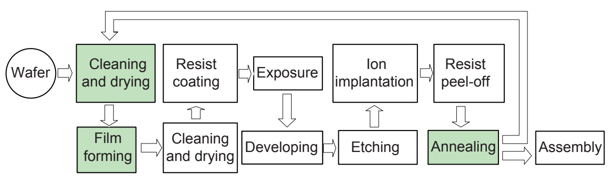


Figure 6 Oxygen Measurement in FPD Manufacturing Process

■ Reflow Furnace in Soldering Equipment

Figure 7 shows the process of the soldering equipment. The ambient concentration O_2 is strictly controlled because oxidized solder immediately causes failures of printed circuit boards and degrade the reliability of printed circuit boards. In particular, the O_2 concentration in the hottest reflow process is strictly controlled to ten to hundreds of ppm.

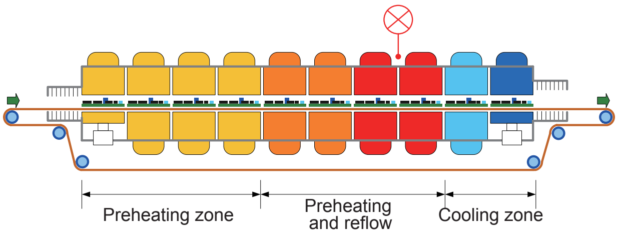


Figure 7 Oxygen Measurement in Reflow Furnace in Soldering Equipment

■ N_2 Generating Equipment Using the PSA Method

Figure 8 shows N_2 generation processes using the pressure swing absorption (PSA) method. When the raw material air is compressed and fed into activated carbon (molecular sieving carbon), O_2 is absorbed in larger quantities and more quickly than N_2 and so N_2-rich air passes through. Then, the remaining air is decompressed, and O_2 is desorbed from activated carbon. By repeating these processes, N_2 can be separated from the air. This method makes it possible to produce N_2 with a purity of 99.998%. The O_2 analyzer is used to control the O_2 concentration of ten to several tens of ppm in order to confirm the purity of the N_2 gas.

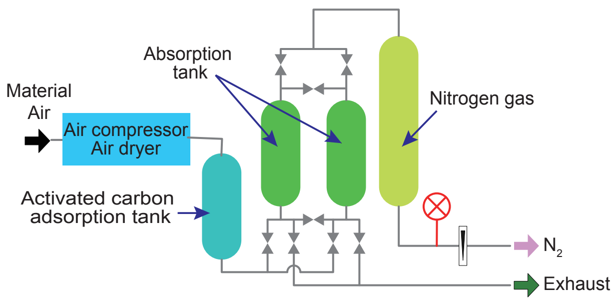


Figure 8 Oxygen Measurement in N_2 Generating Equipment Using the PSA Method

■ Laminated Ceramics Capacitor Manufacturing Process

Figure 9 shows the process for manufacturing laminated ceramic capacitor chips. First, the raw materials are prepared and burnt to form a sheet. Then, the sheet is cut into chips and burnt again, and then external electrodes are formed. In the chip burning and electrode forming processes, the O_2 concentration of 10 to 1,000 ppm in the ambient gas is controlled.

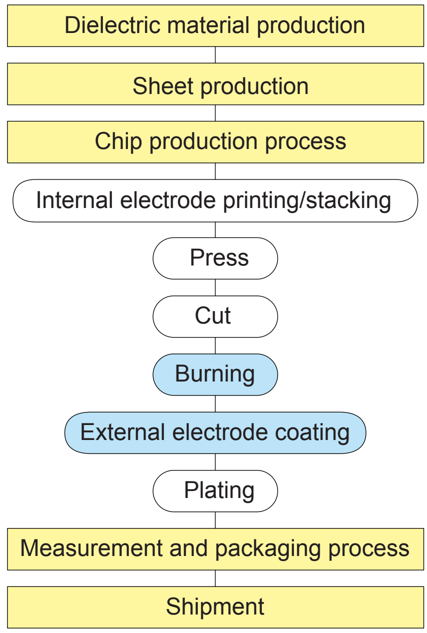


Figure 9 Oxygen Measurement in Laminated Ceramic Capacitor Manufacturing

CONCLUSION

Focusing on the electronics industry, we have developed the OX400 zirconia oxygen analyzer with a wide measurement range from 0-10 ppm to 100 vol%O_2. This analyzer offers not only high-speed response and high reliability but also easier maintenance. Owing to its ability to measure low concentrations, the OX400 can be utilized in many fields other than the electronics industry for improving product quality and safety of operations. We believe our oxygen analyzer will help customers increase production efficiency and safety of their manufacturing operations.