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Product Application Report

Removing Dissolved Oxygen From Ultrapure Water

Membrane Contactor system reduces dissolved oxygen concentration to 10 ppb.

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Key Technologies:

- **Ultrapure water**
- **Oxygen removal**
- **Membrane contactors**

At A Glance:

The ultrapure water (UPW) used to rinse wafers must contain very low levels of dissolved gases such as oxygen, nitrogen and carbon dioxide. The Lucent Technologies fab outside of Madrid, Spain, used a two-phase upgrade project to remove dissolved oxygen from the UPW loop. After introducing a nitrogen sparging system to the loop, a membrane contactor degassing system was installed within the two-pass Reverse Osmosis system. It uses vacuum with a nitrogen sweep gas to reduce the dissolved oxygen level below 10 ppb from a 56 m³/hr UPW loop. It has run continuously, except for scheduled year-end downtime, since May 1995.

The increasing complexity and level of integration in ULSI devices is requiring higher levels of water purity. A typical wafer uses between 20 to 200 separate rinsing steps. Impurities in the rinse water such as particles, high dissolved oxygen content, organic contaminants and gas bubbles can cause defects. Controlling oxide formation on the wafer surface is one of the reasons for reducing residual dissolved oxygen levels. As chip line widths continue to decrease, the quality of the water and the dissolved oxygen content becomes more critical.

At the Lucent Technologies fab near Madrid, Spain, a two-phase plan was enacted to reduce dissolved oxygen levels below 10 ppb. The first step, in 1994, was to install a nitrogen sparger in the filtered water tank. The sparging system provided the first deoxygenation capability at the site and met the goals of the first phase. Using the nitrogen sparger, dissolved oxygen was reduced to 0.6-0.8 ppm.

The second phase of the plan was to lower the dissolved oxygen to <10 ppb. A study was launched to determine the best method to achieve this goal. The options included the following technologies:

- Expanding the existing nitrogen sparger,
- Oxygen scavengers,
- Membrane degassifier,
- Vacuum degassifier.

Expansion of the existing nitrogen sparger was not considered because of its low operating efficiency and nitrogen saturation of the water. The option of oxygen scavengers was eliminated because of the hazards to operating personnel. The remaining alternate processes were membrane degassifier and vacuum tower.

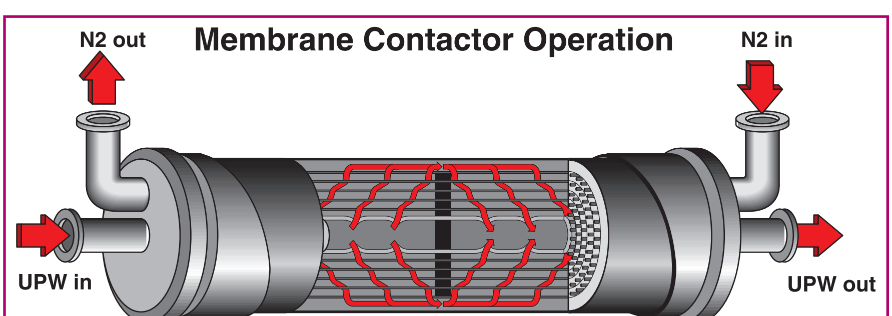
The membrane degassifier and the vacuum tower processes were reviewed using the following criteria:

- System size (footprint) and cost,
- Installation issues,
- Operating characteristics,
- Operational flexibility,
- Expansion capabilities.

System size was critical because of space limitations within the ultrapure water (UPW) plant. The best location for the membrane system was within the existing UPW plant. A ceiling limitation of 3 m and a footprint size of 4 m × 2 m eliminated the use of a tower inside the plant. A tower could be located apart from the UPW plant, but this was strongly opposed because of the long distance from the UPW plant and the expense involved. The membrane system, however, could be built and installed in the designated space at a more reasonable cost.

Installation of a vacuum tower would involve foundation work, on-site fabrication of the tower, high wind stabilizers and long pipe runs. Installation of a membrane unit would consist of moving the skid mounted contactors into position and connecting water, vacuum and electrical lines.

In terms of operating characteristics, the site had no experience with vacuum towers, but it knew of the complexity of the operations



1. The Liqui-Cel® Membrane Contactor uses vacuum and a nitrogen sweep to remove oxygen from UPW.

required. The main issue was maintaining the high vacuum levels required to assure proper oxygen outlet levels. The membrane system appeared to fulfill the requirements of less operator involvement and lower system controls complexity.

The tower design offered little operational flexibility, since vacuum was its only removal mechanism. The membrane system offered three degassing options: nitrogen sweep gas only, vacuum only and a combination of vacuum and nitrogen sweep.

The flow rate and outlet oxygen specification can change depending on the future needs of the facility, and the degassifier must be capable of satisfying those future needs. Since the membrane system is modular in design, it can be expanded by adding more contactors either in series or in parallel. Adding more in series would allow for lower oxygen outlet values, while additions in parallel would allow for any increase in flow rate. A vacuum tower would be restricted in its ability to satisfy such demands.

The membrane degassing system

A 3 × 2 skid of Liqui-Cel® membrane contactors was chosen to bring the dissolved oxygen levels down to the desired levels. Three contactors connected in series are required to bring the dissolved oxygen down to 10 ppb, and two parallel sets of three are required to handle the existing flow rate. Each contactor is 25.4 cm (10in) in diameter and 63.5 cm (28in) in length. With three contactors in series, the oxygen removal in this system is 99.5% at 105 mm Hg. The design and operating conditions are summarized in Table 1.

Table 1. Membrane Contactor System Performance

Water flow rate	56 m^3/hr
Temperature	21°C
Design inlet oxygen concentration	9.5 ppm (actual = 1.32 ppm)
Outlet oxygen concentration	10 ppb maximum (actual = 7.5 ppb)
Vacuum level	50 mm Hg (actual = 105 mm Hg abs.)
Nitrogen consumption	100 standard liters/min (3.85 scfm)
Nitrogen/water flow ratio	1.8 slpm/m^3/hr (0.016 scfm/gpm)
Inlet water pressure	3.0 kg/cm^2 (43 psig)
Outlet water pressure	1.4 kg/cm^2 (20 psig)
System pressure drop	1.6 kg/cm^2 (23 psig)
Inlet TOC level	40-80 ppb
Outlet TOC level	40-80 ppb
Outlet conductivity	<5 μs

Each membrane contactor consists of hydrophobic polypropylene microporous hollow fibers. The fibers have small pores in their walls to allow dissolved gases to pass through. The fiber's hydrophobic characteristics do not allow liquid water to pass through the pores. The membrane is also nonselective, meaning that all free gasses are removed from the water stream. One of the Liqui-Cel membrane contactors is shown in Figure 1.

The contactor design is analogous to a shell and tube heat exchanger. The water stream passes on the outside of the fibers (shellside), while the vacuum and nitrogen sweep gas are on the inside the fiber (lumenside). The difference in the oxygen partial pressure between the shellside and lumenside provides the driving force for gas removal. The nitrogen sweep gas helps to lower the partial pressure of oxygen in the lumenside. That assures the maximum driving force is always maintained and provides a more efficient, economical operation.

Conclusion

The Liqui-Cel membrane contactor degassing system began operation at the Lucent Technologies Madrid fab in 1995 and is meeting the design specification (< 10 ppb Dissolved Oxygen). The benefits of the system are compactness, modularity and ease of operation. The system offers the flexibility necessary to fulfill future expansion demands for capacity and dissolved gas concentration specifications. □

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