

High-purity gases have a key role in LED manufacturing

The availability of a reliable, stable supply of high-purity gases, including ammonia, nitrogen and hydrogen, is a key requirement for a high-volume LED manufacturing facility, according to ANISH TOLIA and IAN TRAVIS.

By Anish Tolia and Ian Travis

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Very large volumes of high-purity gases play a crucial role in the LED chip manufacturing process. The most critical stage in the process – and the cost-intensive one from a materials' point of view – is the growth of the active semiconducting layers by epitaxial deposition. This is accomplished by a process called metal-organic chemical vapor deposition (MOCVD), creating metal-nitride compounds (such as indium gallium nitride, or InGaN) from volatile metal-organic precursors such as trimethyl gallium. The multilayer LED structures are deposited on a wafer, or substrate, which is typically sapphire. Fig. 1a shows the typical cost of wafers, metal-organic precursors and gases in the LED chip-manufacturing process.

A wide range of gases is used in the LED manufacturing process, as shown in Table 1. Ammonia (NH₃) and hydrogen (H₂) represent the largest portions of gas costs (Fig. 1b). These two gases form the majority of the atmosphere in which the crystal layers are grown, and their purity is critical to the performance and quality of the LED device.



FIG. 1.

If moisture (H₂O) or oxygen (O₂) molecules are present in these gases, even in trace concentrations above a few parts per billion (ppb), then oxygen atoms can become incorporated into the crystalline structure of the LED device.

Research has shown that as the moisture concentration in ammonia increases, this leads to a higher concentration of oxygen atoms in the InGaN multi-quantum well (MQW) layers of the LED device. The oxygen contamination has two negative effects. First, the electroluminescence of the device (its light output) is reduced as the moisture content in the ammonia gas is increased (Fig. 2). Second, the current-voltage characteristics of the LED device also deteriorate, with higher leakage current. Both factors reduce the efficiency of the LED as a light source and hence the commercial value of the LED.

High-purity ammonia

Since ammonia is required in high flow-rates during the slow nitride-crystal growth process, even trace quantities of impurities in the gas can lead to a significant number of unwanted atoms being incorporated into the device. To combat this problem, state-of-the-art LEDs are made using ammonia that typically undergoes at least two stages of purification and contains no more than 1 ppb of moisture or oxygen.

Gas	Notes
Ammonia (NH ₃)	Source of nitrogen (N) in nitride-based LED layers
Nitrogen (N ₂)	Carrier gas for nitride crystal growth
Hydrogen	Source of hydrogen for nitride crystal growth
Other trace gases	Source of other elements (Al, Ga, In, Si, Zn, Mg, Cu, Fe, Ni, etc.)
Etching gases	Source of fluorine (F) for etching (CF ₄ , SF ₆ , etc.)
Etching gas	Source of chlorine (Cl) for etching (BCl ₃ , etc.)
Etching gas	Source of boron (B) for etching (B ₂ H ₆ , etc.)

TABLE 1.

Each MOCVD process chamber consumes approximately ten tonnes of ultra-high-purity ammonia gas per year. With the new generation of LED fabrication facilities often incorporating more than 50 MOCVD reactors, cost-effective high-purity supply must be achieved at very high volumes in order to support such large fabs.

There are two challenging properties of ammonia which must be overcome to ensure delivery of consistent high quality at high flow rates: moisture contamination and heat of vaporization.

Ammonia's affinity to absorb moisture (it is hygroscopic) means that purifying the material and then keeping it dry through the filling of containers and delivery to the customer requires very careful design, installation, operation and quality control. The highest semiconductor-industry standards of cleanliness and integrity are applied, including the use of leak-tight valves and high-grade piping systems, to minimize risk of moisture contamination.

Delivery issues

Ammonia is stored and shipped as a liquid, and has a high latent heat of vaporization. A significant amount of energy is required to vaporize the liquid and deliver the flows of 2000 to 3000 liters per minute that modern fabs require.

Compounding this problem is the fact that moisture impurities tend to concentrate in the liquid ammonia. As more liquid is consumed, the high vaporization rates drive higher moisture contamination from the liquid ammonia into the gas supply. To reduce the risk of moisture contamination reaching the MOCVD tool, typically more than 10% of the liquid NH₃ must be left in the container to be returned, re-purified, or disposed of, increasing costs.

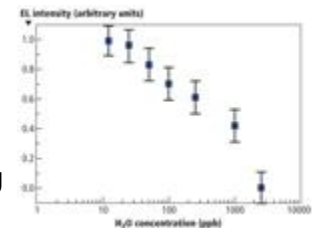


FIG. 2.

The combination of these interacting problems has previously limited the scale of ammonia supply via conventional gas packages such as heated cylinders and drums. As the LED industry grows, and as fab sizes increase, gas suppliers must evolve from being commodity vendors into being expert solution providers for the delivery of such specialist materials.

Linde has developed innovative ammonia supply technology which provides the high flow-rates and stable ultra-high purity required for high-brightness LED manufacturing. The new Spectra Pure high-flow ammonia delivery system features integrated purification, ensuring delivery of the driest possible ammonia at stable flow-rates suitable for a large fab.

High-purity hydrogen

Hydrogen gas purity is also important, for the same reasons as for ammonia. However, purification techniques for H₂ are more mature, and a choice of technologies from catalytic adsorption to palladium membranes can provide 99.999999% purity at the point of use.



FIG. 3.

containers.

Delivering such high-purity hydrogen cost-effectively is impacted by regional variations in supply infrastructure and the supply specifications of raw materials. In Europe, liquid hydrogen is readily available and provides an economic supply mode for many high-flow demands. In China, compressed hydrogen supplied by tube trailers is available, but costs are driven primarily by the distance to the source. For larger LED fabs, an on-site generated supply from a steam methane reformer is often more cost-effective, ensuring a continuous, reliable gas supply without switching

Linde offers a full range of hydrogen supply solutions, from compressed cylinder bundles through to on-site generation systems, which are helping major LED manufacturers to drive down costs while maintaining reliable supply and tight control of material quality.