Bulk Ammonia Supply Solutions for LED Manufacturing

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Abstract

Large volumes of Ammonia (NH_3) gas are needed to provide the nitrogen source for the gallium nitride layers in the manufacture of LEDs. The traditional approach is to use standard 1.5 ft^3 or 47 liter cylinders inside gas cabinets. As NH_3 volumes increased for newer process tools, cylinder supply became impractical. Flow rates were severely limited; the thermodynamic issues causing cooling of the cylinders, requiring their change out at more frequent intervals plus the quality of the ammonia was impacted. Bulk Specialty Gas Supply (BSGS) overcame the many challenges that cylinders in gas cabinets have presented. BSGS uses larger NH_3 source containers fed to a high flow delivery system that is piped to the process tools, analogous to bulk liquid N_2 installations. Many LED manufacturers have embraced the BSGS concept. However, LED manufacturers that have taken an incremental approach may still have issues with flow rates, purity, and reliability. A Total Solutions approach is required that addresses the essentials to ensure reliable NH3 supply. These elements include large-scale NH_3 purification, packaging, and QC, a proven record of integrated bulk delivery system design, with commercial options including on site operations and maintenance. This presentation addresses the basis of bulk high purity NH_3 supply, and dispels some myths, in order to demonstrate the best overall NH_3 Total Solution to LED manufactures.

KEY WORDS Ammonia, Bulk Specialty Gas Supply

INTRODUCTION

Light emitting diodes or LEDs represent the future of lighting. They have the ability to use less energy, run cooler and be more flexible than traditional lighting sources. There is wide acceptance in traffic lighting, automotive lights and architectural applications. LEDs have the potential to revolutionize the world with its cool new technology. Green and shorter wavelength LEDs are manufactured in a process that grows multiple layers of compound semiconductor nitrides, such as Gallium Nitride or Gallium Indium Nitride, into a light generating package. The metal sources are typically organometallic compounds, but ammonia is the Nitrogen source.

Ammonia is a supplied as a liquefied compressed gas that has a vapor pressure of 114 psig at 70° F. NH_3 will readily combine with water to form corrosive ammonium hydroxide (pH of 9.3). Ammonia's flammability range is 16-25% in air. Ammonia has a pungent and irritating odor that is detectable at 5 ppm. Exposure to NH_3 can cause frostbite and chemical burns to the skin, eyes and lungs. Prolonged exposure can cause pulmonary edema, permanent eye damage and death. Handling ammonia safely while maintaining its purity requires experience and a high degree of technical knowledge.

Traditionally ammonia was supplied in 1.5 ft^3 or 47 liter compressed gas cylinders, each containing 50 pounds of NH_3. Each cylinder was in a gas cabinet inside a gas room and piped to each individual LED process tool. As LED Manufacturing evolved, it required larger and purer flow rates of NH_3. Multiple gas cabinets would be piped together to get larger flow rates. To get higher purity, Point of Use (POU) Purifiers were used. In addition, since moisture was the primary contaminant that caused poor LED performance, and it concentrated in the cylinder as it depleted, cylinders were changed out up to half full. Another problem with cylinder supply was that NH_3 has a large heat of vaporization, thus as the NH_3 was withdrawn the cylinder cooled and its pressure dropped accordingly. Large flow rates would cause boiling which would generate aerosols thus mobilizing higher amounts of H2O, further challenging the POU purifiers.

Air Products Solkatronic group improved the traditional supply source with the introduction of their "Blue" grade ammonia at 200 parts per billion (ppB) of H_2O in the early 1990s. However, High Brightness (HB) LEDs require even lower levels of metals and Oxygen bearing impurities. The need for high flow rates and ultra high purity necessitated a completely new approach. Doing things the traditional way was no longer acceptable, nor cost effective. A revolutionary change was required and BSGS was the answer (see Fig 1).

Fig 1 Dual ISO module Tanker Installation Shows Level of Complexity for BSGS

SOLUTION

Bulk Specialty Gas Supply (BSGS) uses large source containers fed to a high flow delivery system that is piped to the process tools, analogous to liquid N_2 bulk installations. Many LED manufacturers have embraced the BSGS concept. However, LED manufacturers that have taken an incremental approach still have problems with flow rates, purity, and the ability to keep costs at a minimum. A Total Solutions approach is needed, and Air Products went to work to address all the requirements. First, a new plant for manufacturing 1 ppB H_2O NH_3 required a clean sheet of paper, but relied on Air Products Air Separation and Specialty Gas purification experience. New analytical methodologies had to be generated and validated to QC this level of purity. Then a new heated ISO module tanker was developed to contain, transport and deliver this higher grade of NH_3 product. That NH_3 ISO gas source had to be meticulously connected to the bulk delivery system (BSGS) which has the necessary redundancy to minimize single points of failures, while ensuring that the NH_3 remains in its gaseous state. Single points of failure were eliminated in the BSGS design using FMEA (Failure Mode and Effects Analysis). Maintaining the ammonia in the gaseous state is a matter of addressing the thermodynamic issues surrounding a gas commonly used in the past for refrigeration. Both the Joule Thompson (JT) cooling that occurs when a gas is reduced in pressure, as well as the energy required for the heat of vaporization to convert the liquefied gas into a vapor, must be accounted for in the design.

This Total Solution is best provided by a specialty gas expert, who has knowledge of the necessary components to ensure reliable NH3 supply:

- large scale purified NH_3 manufacturing, QC and packaging
- a proven track record of bulk delivery system design
- installation, start up and commissioning
- on site operations and maintenance of the delivery system
- various financing options.

The Total Solution requires paying attention to all the details to be successful. Considering ambient conditions versus delivery flow and pressure, site design and safety deliberation to prevent leak impacts, and overall reliability to preclude process gas interruptions

are critical. Clearly, a Total Solutions approach is needed to be successful when dealing with bulk supply of UHP NH_3.

Myths abound on how best to supply large volumes of high purity gaseous NH_3 for HB LEDs at the flow rates and pressures required. Just like the alchemists dream of turning lead into gold, industrial grade NH_3 cannot be reliably transformed on-site by purifiers. Liquid withdrawal, vaporization and subsequent purification via adsorption puts the maximum demand for on-site purifier performance, typically without sufficient proof of quality until it impacts the customer's LED process. On-site analysis for H2O and metals at ppb levels makes both of these approaches more expensive and at greater risk than the tact of preanalyzed source packages. Purity at the Source comes with a contractual guarantee and a Certificate of Analysis to review prior to the supplier's source package acceptance. Vapor phase withdrawal has its advantages. Heating the source package to provide sufficient gaseous NH_3 flow rates with smaller vapor phase H_2O and metals concentrations is the best way. This leaves the heavy liquid phase impurities in the source container instead of sending them to the nitride process. Once the source container is changed, those remaining impurities are disposed of properly at the supplier's plant. Liquid withdrawal with downstream vaporization sends higher concentrations of H_2O and metals to a purifier which risks breakthrough, process contamination and shut down of production lines

CONCLUSIONS

This presentation demonstrates the Total Solutions approach for bulk NH3 supply, and dispels some myths, in order to demonstrate the best overall answer to LED manufacturers. Air Products has the majority market share in supplying Bulk Specialty Gas solutions to the electronics industry because of our extensive experience and proven performance. The most cost effective ammonia solution, which provides the guaranteed highest purity product, is best generated by a large-scale purification plant, and transported to the customer's site while maintaining purity in larger heated ISO containers. However, there is more to it than that. A reliable BSGS is the heart of the delivery solution., Air Products BSGS Design teams have taken advantage of our operating experience in specialty gas plants and in the field to further improve our BSGS offerings. Unique solutions have been developed and patented to enhance our capability to be the best bulk specialty gas supplier in the industry. Couple that with Air Products willingness to own, operate and maintain our BSGS offerings, where others only sell equipment, demonstrate our belief in our Bulk Specialty Gas Supply Solutions. Investing in customer's is a partnership to demonstrate we are working together to ensure their success. The most experienced NH_3 Bulk Supplier in the world has more systems installed than all the competitors combined since the Total Solutions approach has proven successful where others have failed.

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Mr. Ford works in the Electronics Division Specialty Materials group, for the last 10 years managing bulk specialty gas supply (BSGS). In his 20 years with Air Products, Mr. Ford was the Hometown Specialty Gas Plant Laboratory Supervisor, US Specialty Gas QC Manager, Product Manager for Asia Spec. Gas & Equipment, and Electronics Operations Manager - Asia. As an ex-patriot in Taiwan, he worked with Air Products JV partner in developing their specialty gas business. He was responsible for the modernization of the QC laboratory at Hometown, and assisted in the design and start up of three Asian specialty gas facilities - Tsukuba, Japan; Shihwa, Korea; and Chupei, Taiwan. Mr. Ford trained numerous Asian facilities in emergency response techniques. Mr. Ford has seven patents in BSGS along with three publications. He is also the first ever recipient of the Air Products Gas & Equipment Innovation Award in 2003. Prior to joining Air Product, Mr. Ford spent 8 years in environmental consulting laboratories, performing analysis of samples to gauge their ecological impact. His duties included field assignments, underwater assessment, sample collection and evaluations as a scientific diver.

Mr. Conway has worked in the Electronics Davison Process Engineering group since 1995. Prior to this, he was a Startup-Process Systems Engineer for 6 years supporting various projects from Liquid Phase Methanol to UHP Air Separation Plants. As a process engineer in Electronics, he was the lead process engineer on the WF_6, NF_3 West and UHP NH_3 Hometown production plants. His current assignment as NH_3 Technical Program Coordinator is to manage and direct all R&D and Engineering aspects of Air Products Electronics NH_3 business, plus provides technical support to the business area.

ACRONYMS

BSGS: Bulk Specialty Gas Supply
F: Fahrenheit
FMEA: Failure Mode and Effects Analysis
Ft3: cubic feet
HB: High Brightness
H_2O: Water
ISO: International Standards Organization
JT: Joule Thompson
LED: Light Emitting Diodes
NH3: Ammonia
ppB: Parts per Billion
QC: Quality Control
R&D: Research and Development
UHP: Ultra High Purity