CSM

CRYOGENIC

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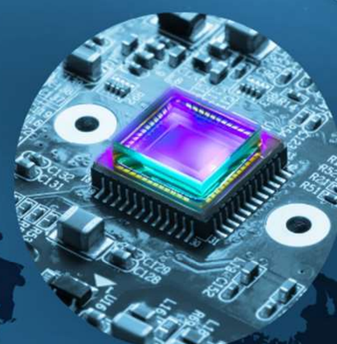

ISO 9001 : 2015

ISO 14001 : 2015 119601/1

Stay cool under pressure

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Molecular Beam Epitaxy (MBE)

What is MBE

Molecular Beam Epitaxy or MBE is an Ultra-High-Vacuum (UHV)-based technique for producing high quality epitaxial structures with monolayer control.

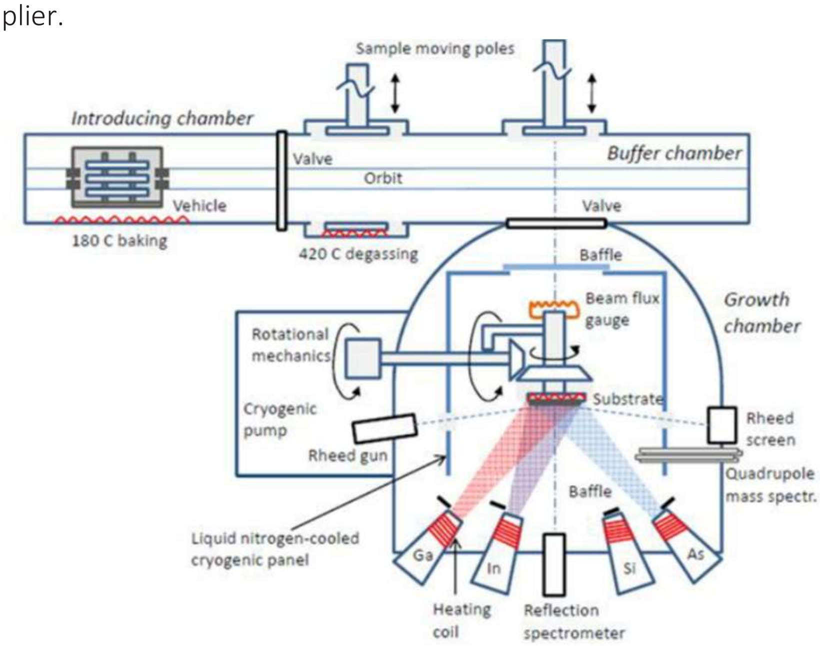
Since its introduction in the 1970s as a tool for growing high-purity semiconductor films, MBE has evolved into one of the most widely used techniques for producing epitaxial layers of metals, insulators, and superconductors as well. Today MBE is an indispensable tool in compound semiconductor industry, both at research and production level.

A typical MBE growth rates for group III-V type semiconductors are of the order of 1 um/hour, obtained for group III partial pressures of ~10e-6 torr. With atomic density in the crystal about 10e22.cm-3, this means that to reduce the impurity concentration below 10e15.cm-3, the impurity partial pressures must be reduced below ~10e-12 Torr.

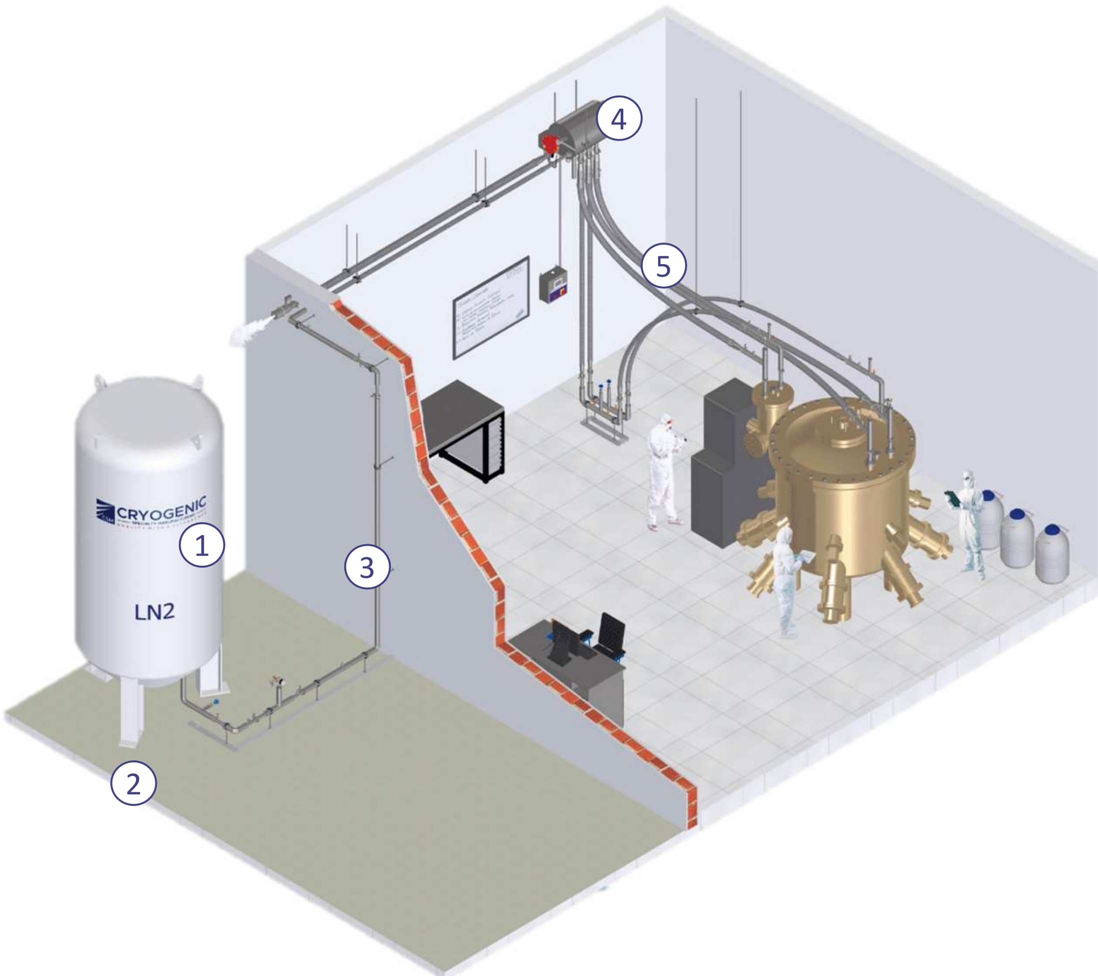
Liquid N2 is very important component for successful operation of MBE. MBE system relies on Liquid N2 cryopanels, built internally around both the main chamber wall and the source flange. Since MBE is a cold wall technique, cryopanels are used to prevent re-evaporation of molecules from parts other than the hot effusion cells.

Besides, they provide thermal isolation among the different cells, as well as additional pumping of the residual gas. Low vapor pressure liquid N2 supply within cryopanels during wafer growing process is paramount to ensure impurities partial pressure are maintain consistently below 10e-12 Torr.

CSM-Cryogenic, has developed a very advanced closed loop liquid nitrogen circulation system for Molecular Beam Epitaxy MBE wafer growing process with benefits not seen with other traditional supplier.



CSM Products for LN2 system in MBE facility



An MBE production requires an LN2 storage tank (1) with a capacity that should be determined based on the LN2 consumption by the MBE system and the LN2 delivery frequency. Typically, a small research MBE system consumes 50-250L/day, while a large MBE system may require 4000L/day. It is highly recommended to utilize a low-pressure (2 to 3 bar) bulk storage tank equipped with a vacuum-insulated withdrawal valve (2). For the pipework (3), it is preferable to use semi-flexible or rigid Vacuum Jacketed Pipe to minimize heat loss of liquid nitrogen, which significantly contributes to the operational cost of MBE production. The piping system can be either static or dynamic vacuum, requiring a small dedicated vacuum pump to maintain vacuum integrity. To ensure the delivery of pure liquid nitrogen to the MBE cryopanel, it is advised to incorporate a phase separator (4) and Triax hoses (5). A well-designed liquid nitrogen system can lead to significant cost savings and achieve a typical return on investment (ROI) of less than one year. For system design and maintenance, it is recommended to consult CSM Cryogenic. Consult CSM Cryogenic for your next LN_2 system design.

Closed Loop LN_2 Circulation System in MBE Process

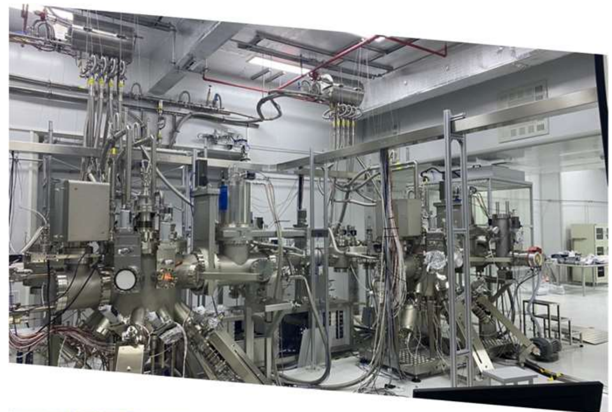
How does Close Loop Liquid Nitrogen System works

CSM design LN2 close loop circulation system for many MBE system in the world, such as Riber, DCA and Veeco. It has been widely use in many customers for compound semiconductor device development such as infra-red, optical communication and many others. Our system is available in static vacuum or dynamic vacuum insulation. A complete close loop system consist of phase separator with multiple outlets, triax hoses, modular fittings and vacuum jacketed valve for liquid nitrogen flow control and isolation.

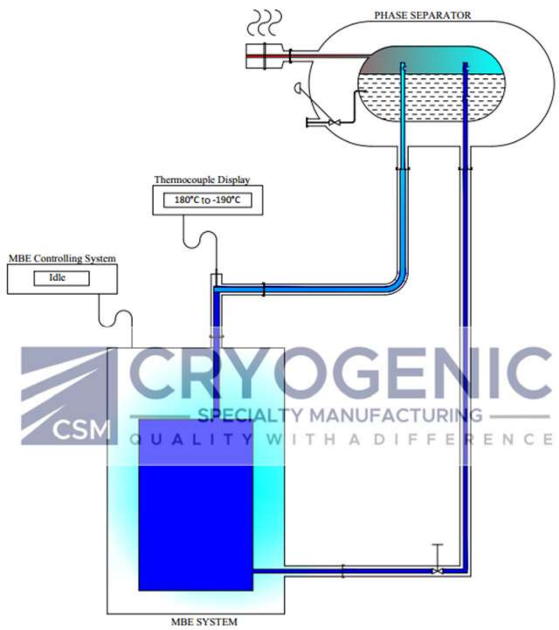
CSM closed loop LN2 circulation system for MBE is maintained at atmospheric pressure at all time. The controller allow the phase separator to self-regulate the LN2 supply flow rate to MBE in a close loop manner, keeping cryopanel filled with liquid nitrogen at different operating conditions.

CSM close loop LN2 system allows the following benefits:

- LN2 saving up to 30%
- Efficient cryopanel cooling to -196 °C
- Ease of LN2 operation - self regulating LN2 flow rate according thermal load at various stages of MBE system operation
- No dewar handling
- Safety



Closed Loop LN_2 Circulation System in MBE Process



MBE at Standby Mode

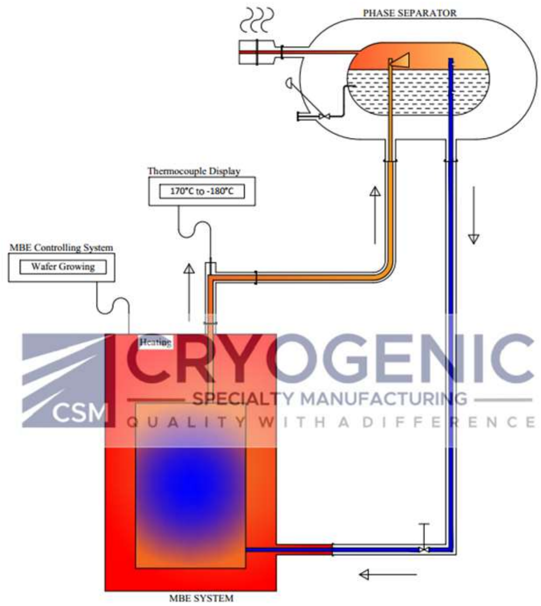
At this stage, the LN2 system is at thermo-equilibrium with cryopanel and process chamber is at room temperature. Cryopanel is insulated under Ultra-High Vacuum environment, and it is at the lowest heat load. The LN2 consume by the cryopanel at this stage is minimum, usually half the amount consumes during the wafer growing stage.

The LN2 flow rate within the close loop system is directly proportioned to heat flux surrounding the cryopanel.

CSM LN2 circulation system will supply single phase “pure liquid” via Triax-Feed line and maintaining cryopanel at temperature -196 °C with density of 0.81, and two phase “warm liquid-vapor mixture” in coax-return line at temperature between -180 °C to -196 °C and density of 0.70 ~ 0.77 depend on the heat load at the cryopanel.

*Note: LN2 flow consumption during standby mode is provided by MBE manufacturer. LN2 system supplier has the responsibility to ensure the phase separator and piping system is engineered and sized to meet MBE flow requirement.

Closed Loop LN_2 Circulation System in MBE Process



MBE at Wafer Growing Mode

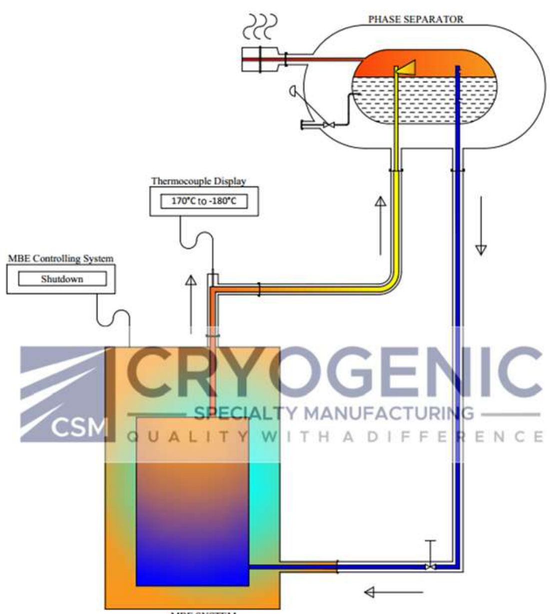
At this stage, thermal load is the highest and so does LN_2 flow rate and consumption. During wafer growing, cryopanel absorbs heat load from the growth chamber. The heat load varies depend on type of wafer growing process.

The pure LN_2 in cryopanel will begins to boil and become two phase liquid-vapor mixture. The warmer liquid begins to rise in the cryopanel and return to phase separator through the coax-return line. The warmer liquid subsequently undergoes phase separation into pure liquid and warm vapor.

The phase separator keep pure liquid nitrogen in reservoir for feeding back into the MBE cryopanel, while warm vapor is vented out to the atmosphere.

During this time, cryopanel is constantly fed with cold LN2 from the phase separator in an increasing amount via the Triax-Feed line until a thermo-equilibrium is achieved. The LN2 feeding flow rate is controlled by the LN2 system in an independent and autonomous fashion in response to MBE heat load variation.

CSM LN_2 circulation system is capable to maintain a constant cold liquid phase in cryopanel at temperature -196 °C, and warmer "liquid-vapor" phase in coax-return line at between -170 °C to -190 °C subject to the heat load at the cryopanel.



MBE at End of Wafer Growing

Once the wafer growing process is interrupted or stopped, the heat load to the cryopanel will gradually reduce. The liquid feed flow rate will reduce in proportion to the heat load.

The fresh LN_2 is fed continuously in reducing fashion into the cryopanel until a thermo-equilibrium is achieved.

The LN_2 circulation system is capable to maintain pure liquid phase in Triax-Feed line at temperature -196 °C, and warmer "liquid-vapor" phase in coax-return line at between -175 to -190 °C depend on heat load at the cryopanel.

LN2 losses in MBE operation

The usage of LN2 represents a significant expense in operating an MBE laboratory. Designing an efficient LN2 cooling system is crucial and involves more than a simple fluid transport setup. Typically, an LN2 cooling system transports LN2 from a dedicated bulk storage tank outside the building to the MBE system's cryopanel. Within the delivery system, LN2 experiences two types of losses: Heat Leak Loss and Flash Loss.

Heat loss occurs due to heat leaks and N2 gas enthalpy within the system. Poorly insulated connections or piping can lead to localized heat leaks, causing LN2 to evaporate into N2 gas. The N2 gas carries heat and permeates the entire pipework system, exacerbating LN2 loss. To minimize this, it is recommended to use vacuum insulated technology throughout the LN2 delivery system, including valves, pipework, and connections. Regular checks and maintenance of vacuum insulation integrity are also crucial.

The second type of loss is flash loss, which occurs when the pressure of LN2 reduces from the bulk tank to the phase separator. This pressure change causes a fraction of the LN2 to flash off as N2 gas. To reduce flash loss, it is advisable to store LN2 in the storage tank under low pressure, sufficient for delivering LN2 to the intended destination at the desired flow rate.

The presence of both GN2 and LN2 in the piping system creates a two-phase flow, which hampers cryopanel cooling due to the presence of N2 gas. To address this, a phase separator is typically employed to separate the gas and liquid phases, ensuring the delivery of high-purity LN2 to the MBE cryopanel.

The phase separator stores LN2 at atmospheric pressure (the most efficient pressure for MBE cooling) and utilizes gravity to drive pure LN2 into the cryopanel via Triax Hose. The fresh LN2 absorbs heat in the cryopanel for cooling purposes. An additional Triax Hose, known as Coax Hose, is designed to efficiently vent the generated N2 gas from the cryopanel without interrupting the entry of pure LN2. This setup allows the cryopanel to achieve optimum efficiency in cooling.

The ideal LN2 cooling system comprises:

- Low-pressure LN2 storage tank with vacuum insulated withdrawal valve
- Vacuum insulated pipework and isolation valve
- Phase separator (operating at atmospheric pressure) with modulating liquid level
- Vacuum insulated Triax hose and Coax hose
- Vacuum insulated N2 vapor venting line from the phase separator to the outdoors.

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Cryogenic Specialty Manufacturing Sdn.,Bhd
No 10, Jalan PPU 3A, 47150 Puchong,
Selangor, Malaysia
Tel : +603-8051 0982
Fax : +603-8051 0913
E-Mail : info@csm-cryogenic.com