

History of Air Separation Unit Technologies Developed by TNSC

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In 1935, we succeeded in domestically producing Japan's first air separation unit. Since then, based on our abundant experience and superior technological capabilities, we have developed various technologies including: process technology to design processes that match customer needs; pre-treatment equipment to remove moisture and carbon dioxide from raw material air; and cryogenic equipment such as rectifying columns, which are the core components of air separation units. Air separation units have a history of more than 100 years, and continue to make progress based on these technological capabilities. This paper introduces the development of air separation units that we have been working on, and describes how we intend to develop these technologies in the future.

1. Introduction

Oxygen is used in a wide range of industrial fields, including steel, chemistry, shipbuilding, nonferrous metals, glass, and medicine. In particular, the increase in demand for oxygen in the steel industry in the 1970s promoted the development of larger air separation units. In the 1970s, the maximum oxygen production per unit was 35000 Nm³/h, but now it is up to 65,000 Nm³/h, and the operability has been greatly improved through automation of the equipment. The specific power consumption has been reduced by about 20% compared to the 1970s.

On the other hand, also nitrogen generation units have been rapidly becoming larger and more power-efficient in line with the development of the electronics industry. The nitrogen production per unit has been up to 25000 Nm³/h, and the specific power consumption has been reduced by about 30% compared to the 1990s¹⁾.

In this way, we have made development to meet customer needs in air separation units. This paper introduces some of the features of the technologies and equipment we have developed so far and describe our future prospect.

In this review, air separation units refer to equipment that mainly produces nitrogen, oxygen, argon, etc. from raw material air using the cryogenic air separation

method, and nitrogen generation units refer to equipment specialized for the production of nitrogen using the same method.

2. Unit type and designation

Air separation units has pre-treatment equipment to remove impurities such as water and carbon dioxide gas from raw material air before introducing the air into the cryogenic air separation section. This equipment was either a Revex or Regenerator system until the 1970s, and has been an MS (molecular sieves) adsorber system since the 1980s. We determine the unit type depending on the outlet pressure of the air compressor and the pre-treatment equipment. When the outlet pressure of the air compressor is low (0.5 MPaG or less) and the pre-treatment equipment is the Revex or Regenerator system, the unit is designated as NR type, which stands for Niederdruck, a German word with the meaning of low pressure, and Revex or Regenerator. This is because the units were initially imported from Germany. When the outlet pressure of the air compressor is medium (0.5 MPaG or higher) and the pre-treatment equipment is the MS adsorber system, the unit is designated as MG type, which stands for Mitteldruck, a German word with the meaning of medium pressure, and Gel (adsorbent).

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However, at present, the pressure classification is not clear, and even units with a low pressure MS adsorber system are also designated as MG type units. In addition, units with a medium pressure liquefaction cycle for the production of liquid products are designated as MNR type or MMG type, the additional M of which stands for middle pressure. Internally compressing units, which compress the product gas using a liquefied gas pump, are designated as MGP type units. For example, an MG type unit with 185,000 Nm³/h of raw material air is called MG185000 type unit. In the case of an NR type unit, the last three digits are omitted and the unit is called NR185 type unit.

In the same way, also nitrogen generation units are designated as MG type nitrogen units, but standard units mainly for Japanese market are designated as JN type nitrogen units. Currently, we are developing GNS type and GND type nitrogen units, which are standard units mainly for the overseas market. These stand for Global Nitrogen Single column and Global Nitrogen Dual column, respectively.

3. Technical cooperation and collaboration

In 1962, we entered into a technical cooperation agreement with Linde, the headquarters of which was in West Germany at that time and is now in the UK, to introduce the world's most advanced technology for air separation units. Based on this agreement, we changed the cryogenic equipment materials of our air separation units from copper and brass to aluminum to reduce costs and improve safety. In addition, by introducing Linde's technology for manufacturing large air separation units, we developed large units and improved our technical capabilities, which greatly contributed to the on-site plant project at that time²⁾.

Since the signing of the technical cooperation agreement, we have been able to obtain the selection, welding, and processing techniques of aluminum materials for the production of equipment and gases, and the basic data and fabrication drawings necessary for the design of major equipment such as rectifying columns. During the 18 years before the contract was terminated, we built 75 Linde type air separation units, including the 35,000 Nm³/h oxygen unit, which was the largest class at that

time. The units built under this contract bear the "Licensed by Linde" nameplate, which is still in place today. Fig. 1 shows the Linde nameplate.



Fig.1 Linde nameplate

(manufactured by former Nippon Sanso Co., Ltd.)

After the technical cooperation agreement with Linde was terminated in 1980, we established and strengthened a development department to improve and enhance our own technology, and the development of air separation units became active. Fig. 2 on the next page shows the history of the development of air separation units^{6, 10, 13, 16, 22, 25-28, 38, 45, 48, 52, 64)}. This was also the beginning of the export of air separation units to overseas markets, which had previously been restricted.

In 2000, we had been able to design and manufacture up to 35,000 Nm³/h oxygen units using our own technology. To further increase the size of the unit, however, there were challenges in manufacturing the rectifying column and MS adsorber. Therefore, in order to meet the subsequent demand for larger units in the domestic market, we started a collaboration with Linde for the fabrication of larger units, which is still ongoing.

As a result of this collaboration, we installed a 50000 Nm³/h oxygen unit at Fukuyama Plant of Kokan Sanso Center (now JFE Sanso Center) in 2002. This unit adopted a DFR type main condenser (Down-Flow Reboiler) for the first time³⁰⁾. In addition, we installed a 65000 Nm³/h oxygen unit at Keihin Plant of JFE Sanso Center in 2005. This unit was the largest class in Japan, and we introduced a krypton/xenon concentration column and a neon concentration column⁴²⁾. Furthermore, we installed a high-purity xenon production unit, the largest

—Taiyo Nippon Sanso (from 2004)

Development theme	1970	1980	1990	2000	2010	2020
Technical cooperation and collaboration with Linde	Technical cooperation (1980 and earlier)		Collaboration (from 2000)		★ 65,000 Nm ³ /h oxygen unit (2005)	
Process development	★ First LNG utilizing unit (1971)				High-pressure LNG utilizing unit	
Rare gas separating process	Kr/Xe separation		Kr/Xe separation		Ne separation Xe purification Rare gas purification	
Demand fluctuation responding process	Demand fluctuation responding		Large demand fluctuation responding			
Nighttime electricity utilizing process	First nighttime electricity utilizing unit (1991) ★					
Low-purity oxygen process	Low-purity oxygen		HIDIC utilizing		Mixing column	
Internally compressing process	First internally compressing unit (2002) ★		Internally compressing liquid co-producing			
High-efficiency process	Standard liquid plant		Low power consumption			
High-purity oxygen co-producing nitrogen production process			First oxygen co-producing unit (2019) ★			
Standard nitrogen generation unit	JN series, GN series		JNT, JNP, JNC, JNM		JNR, JNE, JNF	
Power-saving nitrogen generation unit	First JN unit (1982) ★		Dual column type		GNS, GND series	
Next-generation nitrogen generation unit	Low-pressure reboiler type		Next-generation (packed column)		Ultra-pure oxygen co-producing	
Pre-treatment equipment	MS adsorber		Single-layer MS adsorber First multi-layer MS adsorber (1987) ★ PTSA type		High-performance MS adsorber High-speed flow MS adsorber Radial flow	
MS adsorber simulator	MS adsorber simulator		Improved MS adsorber simulator			
Normal-temperature TSA adsorber	Normal-temperature PTSA		Normal-temperature TSA		Expansion of application scope New MS adsorbent	
Normal-temperature catalyst	H ₂ /CO removing catalyst		New normal-temperature catalyst			
Cryogenic equipment	Rectifying column		★ First packed rectifying column (1996)			
Lower column	Packed rectifying column		Four-path cross-flow lower column			
Packed material	Increased-density packed material		Development of packed material and distributor			
Main condenser	DFR type		Multi-stage type		Cascade type	
Expansion turbine	NET-A type		★ First NET-A unit (1985)		Expansion of lineup	
Gas bearing expansion turbine	NET-M type		Hybrid bearing type, five-axis control type		Expansion of lineup	
Magnetic bearing expansion turbine	NET-C type					
Low-temperature compression-braking expansion turbine						
Operation control	Design simulator		Dynamic simulator		Next-generation dynamic simulator	
Simulator for education	Simulator for education		Simulator for education		New simulator for education	
Automatic control, remote monitoring, remote operation	Automatic start of liquefaction equipment		Expert Fuzzy control		Fieldbus Optimization Introduction of digital technology	
	Full-automatic control		EzMPICS		JN General Monitoring Center	

Fig.2 History of development of air separation units

in Japan ⁵⁶). Through this collaboration with Linde, seven large domestic units have been installed so far ⁶²). Table 1 shows the basic specifications of the 65000 Nm³/h oxygen unit.

Table 1 Basic specifications of the 65000 Nm³/h oxygen unit

Product	Flow rate (Nm ³ /h)	Purity (vol)
Oxygen gas	61400	O ₂ ≥ 99.6 %
Liquefied oxygen	3600	O ₂ ≥ 99.8 %
High-purity nitrogen gas	80000	O ₂ ≤ 0.1 ppm
Crude nitrogen gas	20000	O ₂ ≤ 3 %
Crude argon gas	1580	O ₂ ≤ 1.5 %
Liquefied crude argon	400	O ₂ ≤ 1.5 %
Kr/Xe concentrated liquefied oxygen	80	Low purity
Concentrated Ne gas	8.5	Low purity
Raw material air	320000 Nm ³ /h 456 kPaG	
Specific power consumption *1	0.364kWh/Nm ³	

*1 Specific power consumption = Power consumption / (Oxygen gas + liquefied oxygen) flow rate

4. Development of process

We have developed a variety of processes to meet diversifying customer requirements for air separation units. The following sections describe a part of such processes and equipment we have developed to date.

4.1 LNG cold heat utilizing process

As mentioned in Technical Report No. 1, the introduction of LNG (Liquefied Natural Gas) to Japan began in 1969, when a dedicated LNG tanker from Alaska arrived at the Port of Yokohama ³). Before the introduction of LNG, studies on the use of LNG cold heat had already been conducted and the results of research on LNG utilization technology were published in technical and professional journals. In 1968, we established the "Technical Committee for LNG Cold Heat Utilization" to

study the basic technology, safety, economics, and future prospects, and concluded that the active development of LNG cold heat utilizing unit is promising for air separation units, especially for liquefied oxygen and liquefied nitrogen production facilities. We started designing and manufacturing the world's first LNG cold heat utilizing air separation unit in 1969, and delivered it to Tokyo Liquefied Oxygen Co., Ltd. in 1971. Since then, we have adopted new technologies such as low-temperature compressors and high-pressure heat exchangers, and have completed a unit that reduces the specific power consumption by about half compared to conventional air separation units. We have delivered a total of nine LNG cold heat utilizing air separation units to date, both in Japan and overseas. Fig. 3 on the next page shows a system diagram of an LNG cold heat utilizing air separation unit.

The units we delivered until the 1990s used low-pressure LNG (1 to 3 MPaG). In response to the shift to high-pressure LNG with the introduction of high-efficiency power generation facilities since the 2000s, we have converted existing units to use high-pressure LNG (7 MPaG), and have delivered three units so far. In order to minimize the modification cost, the renewal was made only for the LNG system, and the other components reused existing equipment. In order to maintain the operating conditions of the components using existing equipment, the LNG usage was increased, and it was confirmed that the unit could be operated at the same power consumption and production rate as before as a result ⁵¹).

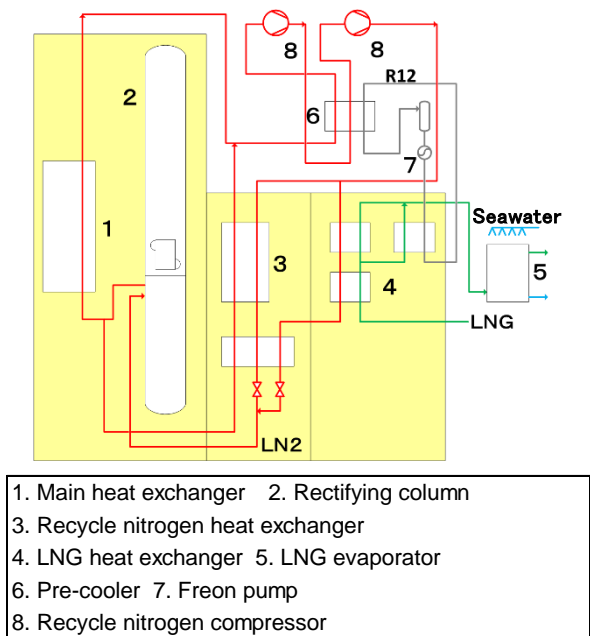


Fig.3 System diagram of LNG cold heat utilizing air separation unit

4.2 Nighttime electricity utilizing process

The liquefaction equipment used to produce liquefied oxygen and liquefied nitrogen consumes large amounts of electricity due to the use of high-pressure, large-capacity compressors. In the 1980s, electricity demand was higher during the daytime and lower at night and on holidays, and electric power companies aimed to equalize electricity demand by reducing the unit price of electricity during nighttime and holidays to less than half that during the daytime. Therefore, by making contracts to use more electricity at night and on holidays, it was possible to use inexpensive electricity.

Thus, we have developed a nighttime electricity utilizing unit in which the liquefaction equipment is operated only during the inexpensive-electricity hours. By operating the air separation equipment continuously during the day and night and the liquefaction equipment only during the night, and by injecting liquefied nitrogen from a storage tank to supply the cold needed during the daytime, the air separation equipment can be kept in constant operation. However, since the liquefaction equipment operates only at night and on holidays, a large liquefaction unit capable of handling a larger flow rate than the average production rate is required. In addition, since the liquefaction equipment is of the DSS (Daily Start and Stop) type, which starts and stops every day, and the compressor, heat exchanger, and other components are subject to a lot of start-up stress, the design and material

selection for withstanding the stress had to be made. The unit is automatically controlled by a DCS (Distributed Control System), which reduces startup and shutdown time and enables startup and shutdown by one-touch operation. Furthermore, this one-touch startup and shutdown operation can be omitted by scheduling it using the calendar in the DCS. In 1991, the first unit of this nighttime electricity utilizing air separation unit was delivered to Toho Acetylene Co., Ltd., and six units have been delivered so far (4). Fig. 4 shows a system diagram of the nighttime electricity utilizing air separation unit.

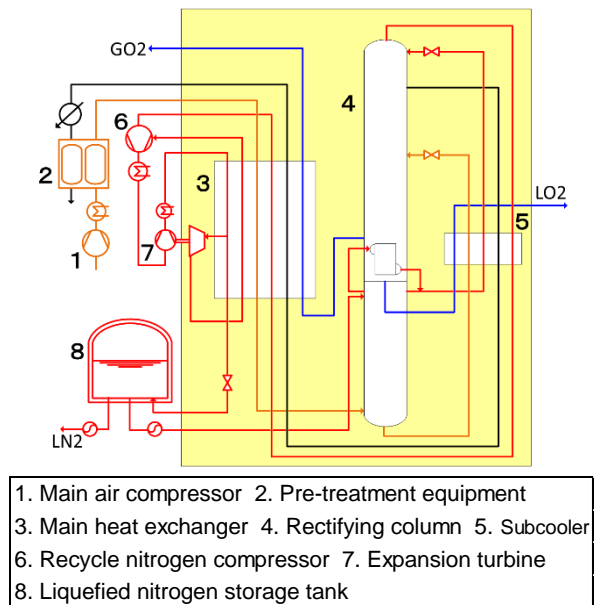


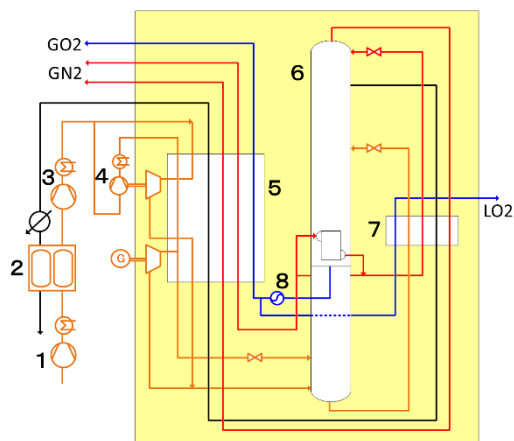
Fig.4 System diagram of nighttime electricity utilizing air separation unit

4.3 Internally compressing process

The MG type unit introduced above employs a total low-pressure process, whereby the product oxygen gas is generated as low-pressure oxygen gas. To raise the pressure of the oxygen gas to the required level, an oxygen compressor is required. However, oxygen compressors are more expensive and have shorter maintenance cycles than air compressors. Therefore, we have developed internally compressing equipment in which liquefied oxygen in the main condenser of the equipment is pressurized to the product pressure by a pump and vaporized and heated by heat exchange with high-pressure air. This system uses a less expensive air booster compressor and liquefied oxygen pump instead of an expensive oxygen compressor, thereby reducing equipment costs compared to conventional equipment.

In addition, since the internally compressing equipment

extracts liquefied oxygen equivalent to the amount of product oxygen from the main condenser, the concentration of hydrocarbons in the liquefied oxygen in the main condenser can be significantly reduced, so that the safety of the equipment is improved. In 2002, we delivered the first internally compressing air separation unit to Shunan Sanso Co., Ltd.³⁴⁾. Furthermore, we have developed and completed an internally compressing air separation unit that can co-produce liquid products without installing a separate liquefaction unit by incorporating a liquefaction process for collecting liquid products into the oxygen pressure boosting section of the unit in order to meet the demand to produce liquid products at the same time as generating high pressure oxygen gas^{47, 49, 58)}. Fig. 5 shows a system diagram of the internally compressing liquid co-production air separation unit.



- 1. Main air compressor
- 2. Pre-treatment equipment
- 3. Air booster compressor
- 4. Expansion turbines
- 5. Main heat exchanger
- 6. Rectifying column
- 7. Subcooler
- 8. Liquefied oxygen pump

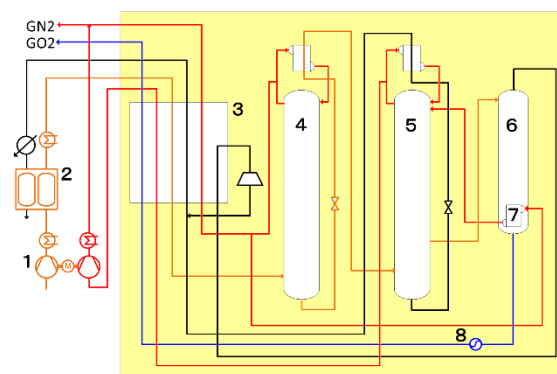
Fig.5 System diagram of internally compressing liquid co-production air separation unit

4.4 Nitrogen and high-purity oxygen co-production process

In 2003, we developed a dual column nitrogen generation unit capable of reducing power consumption by about 30% (specific power consumption 0.195 kWh/Nm³) compared to conventional nitrogen generation units and of efficient reduced volume operation with up to 50% reduced rated product volume^{40, 44, 46, 55, 60)}.

After that, as the demand for nitrogen gas increased due to the development of the semiconductor industry, the demand for the simultaneous use of a small amount of

oxygen gas increased. Therefore, in 2015, we developed a high-purity oxygen co-producing nitrogen generation unit as an improved version of the dual column nitrogen generation unit. The developed unit further improves the nitrogen yield, co-produces a small amount of oxygen gas, and achieves low power consumption and space saving. This unit has a dual column nitrogen generation unit and an additional third rectifying column for oxygen production, and incorporates an internally compressing process. Nitrogen gas is taken out from the high-pressure column as a product. In addition, nitrogen gas taken out from the low-pressure column is boosted by the nitrogen compressor and joins the aforementioned nitrogen to become a product. On the other hand, liquefied oxygen gas is extracted from the bottom of the third rectifying column, pressurized by the liquefied oxygen pump, and vaporized and heated in the main heat exchanger to become a product. In this unit, the air booster compressor generally used in an internally compressing process can be omitted. In addition, the unit is compact due to the adoption of combined equipment of a main air compressor and a nitrogen compressor. Furthermore, the MS adsorber employs normal-temperature adsorbents and hydrogen (H₂) and carbon monoxide (CO) removal catalysts to enable higher purity. In 2019, the first high-purity oxygen co-producing nitrogen generation unit was delivered to the Iwate Kurosawajiri Gas Center⁶¹⁾. Fig. 6 shows a system diagram of the high-purity oxygen co-producing nitrogen generation unit, and Table 2 on the next page shows the basic specifications of the unit.



- 1. Main air compressor/Nitrogen compressor
- 2. Pre-treatment equipment
- 3. Main heat exchanger
- 4. First rectifying column
- 5. Second rectifying column
- 6. Third rectifying column
- 7. Third main condenser
- 8. Liquefied oxygen pump

Fig.6 System diagram of high-purity oxygen co-producing nitrogen generation unit

Table 2 Basic specifications of high-purity oxygen co-producing nitrogen generation unit

Product	Flow rate (Nm ³ /h)	Purity (vol)
Nitrogen gas	20000	O ₂ ≤ 0.1 ppm
		CO ₂ , CO, H ₂ ≤ 0.1 ppm
		H ₂ O ≤ 1 ppm
Oxygen gas	1400	O ₂ ≥ 99.5 %
		CO ₂ ≤ 3 ppm
		CO ≤ 1 ppm
		CH ₄ ≤ 25 ppm
Product pressure	800 kPaG (nitrogen gas, oxygen gas)	
Raw material air	34500 Nm ³ /h	920 kPaG
Specific power consumption*1	0.208 kWh/Nm ³	

*1 Specific power consumption = Power consumption/(Nitrogen gas+Oxygen gas) flow rate

5. Standard nitrogen generation unit

Nitrogen gas is used in a wide range of fields, including the semiconductor industry, petrochemicals, textiles, and glass. Since we manufactured the first nitrogen generation unit that produced nitrogen gas as an independent product, we have responded to domestic and international demand. From that time to the present, we have made improvements and modifications to our units in response to various market demands, and have worked to reduce the costs and improve the specific power consumption. In the 1970s, global competition regarding nitrogen generation units intensified and demand for higher performance and lower cost units increased. In 1982, we completed the first JN type nitrogen generation unit, which achieved an 18% reduction in specific power consumption and a 10% reduction in price compared to conventional units, and made a series of nitrogen generation unit models (named the JN series) available⁴⁾. At that time, nine models ranging from 100 to 1,000 Nm³/h nitrogen gas were available as standard JN type nitrogen generation units in order to meet various demands.

From 1984, a large gas demand was expected for the semiconductor industry, and the TGC (Total Gas Center) project to install nitrogen generation units throughout the country was promoted. The JN series greatly contributed to this TGC project^{8, 11)}.

Furthermore, in 1988, we launched JNS type nitrogen generation unit series for the semiconductor industry, featuring high purity, unitization of components into a container, remote monitoring, and automatic operation.

Then, as an improved version of the JNS type unit, we completed the JNT type nitrogen generation unit, which

employed a normal-temperature PTSA (Pressure and Thermal Swing Adsorption) adsorber in the pre-treatment equipment to eliminate CFC (Chlorofluorocarbon). After that, as a result of further improvements, we developed the JNP type nitrogen generation unit series, which employed a CFC-free normal-temperature TSA (Thermal Swing Adsorption) adsorber for the pre-treatment equipment. In the JNP type unit, high product purity and low noise were achieved and the appearance was improved by eliminating the container used for the JNS type unit and using a low-noise box that accommodated the components.

In 1997, against the background of intensifying competition regarding on-site gasification of liquid supply, we completed the JNC type nitrogen generation unit, a compact on-site unit with a separator and a storage tank on top of the unit for the purpose of smaller volume and space saving. The JNC type nitrogen generation unit does not have an expansion turbine and operates with a liquid injection method in which liquefied nitrogen is injected from the storage tank and an air-cooled cooler is used, eliminating the need for cooling water. As a result of thorough space saving, the footprint of the unit was 12 m²²¹⁾.

In order to meet increasing demand, we developed the JNR150 type nitrogen generation unit (nitrogen gas flow rate 1500 Nm³/h) to replace the JNP100 type nitrogen generation unit (nitrogen gas flow rate 1000 Nm³/h), which was the highest-flow model of the JN series until then, and to further reduce the specific power consumption. The JNR150 was the first unit that employed a waste gas circulation cycle to improve the product yield of nitrogen gas by boosting the pressure of low-temperature waste gas with the expansion turbine braking compressor and introducing it into the rectifying column for redistilling³⁹⁾.

After 2010, we developed the JNF type nitrogen generation unit as a successor high-purity unit to the JNP type unit, which eliminated the box so that the components could be arranged flexibly, thereby reducing the price. In addition, the JNF type unit was the first nitrogen unit to adopt a packed rectifying column to reduce the weight of the cold box⁵⁰⁾.

Currently, we are planning to produce a series of GNS type nitrogen generation unit models and GND type nitrogen generation unit models as standard nitrogen

generation units mainly for overseas markets. Fig. 7 shows a concept image of GNS type nitrogen generation unit.

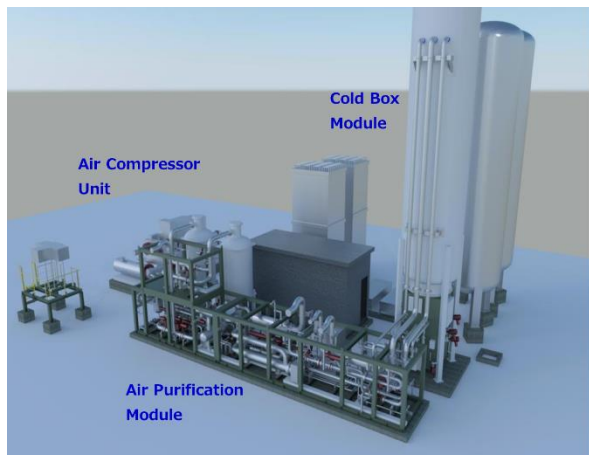


Fig.7 Concept image of GNS type nitrogen generation unit

6. Pre-treatment equipment

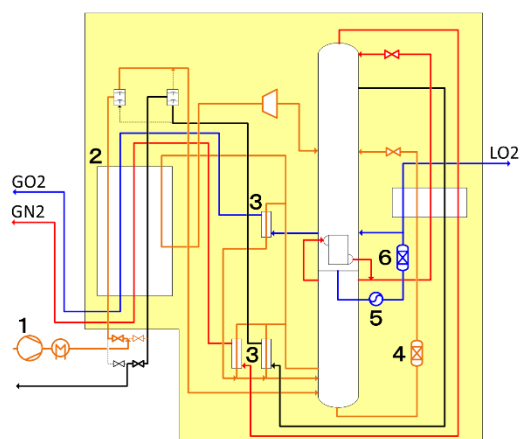
As mentioned above, in order to operate an air separation unit safely, it is necessary to remove impurities such as moisture and carbon dioxide contained in the raw material air by pre-treatment equipment. We have adopted a thermal regeneration type TSA (Thermal Swing Adsorption) adsorber for this pre-treatment equipment.

6.1 Reversing heat exchanger

Before the TSA adsorber, pre-treatment equipment using a reversing heat exchanger was used. Fig. 8 shows a system diagram of an NR type air separation unit using a reversing heat exchanger.

With this method, air is cooled as it passes through the heat exchanger, and the moisture and carbon dioxide in the air precipitate on the fin surfaces in the low-temperature section inside the heat exchanger. As a result, the low-temperature air at the exit of the heat exchanger contains almost no moisture and carbon dioxide. The precipitates on the fin surface are purged by the returning waste nitrogen gas and removed by sublimation and evaporation, but the purging requires a quantity of waste nitrogen gas equivalent to about 50% of the introduced air volume. Therefore, the amount of gas that can be taken out as product nitrogen is limited. In addition, since hydrocarbons contained in the raw material air are not removed, an acetylene adsorber and a circulating

adsorber must be installed inside the separator.



1. Main air compressor
2. Reversing heat exchanger (Revex)
3. Liquefier 4. Acetylene adsorber
5. Liquefied oxygen pump 6. Circulating adsorber

Fig.8 System diagram of NR type air separation unit

6.2 MS adsorber

To overcome these shortcomings of reversing heat exchangers, we began developing adsorption type pre-treatment equipment. In 1961, we developed a system in which water is removed by alumina gel at normal temperature and carbon dioxide is removed by silica gel at low temperature, and in 1966, we commercialized a single-layer MS adsorber for a nitrogen generation unit. This adsorber removes water and carbon dioxide by adsorption with Na-X zeolite. At the time of development, this adsorber was inferior to the reversing heat exchanger in some respects, such as the high regeneration energy of the adsorbent, and it was a little while before the adsorption type was mainly used for the pre-treatment equipment.

After that, we began to concentrate on the improvement of the adsorption method around 1984. Through the development of high-performance MS adsorbents, we employed a multi-layer MS adsorber in which moisture is removed by the alumina gel layer and carbon dioxide is adsorbed by the zeolite layer. The amount of waste gas required for regeneration with this adsorber was about 20% of the air volume, and the regeneration temperature was 140°C. This significantly increased the amount of product nitrogen gas and reduced the energy required for regeneration. In 1987, we adopted the first multi-layer MS adsorber in the MG25500 type unit for Shinnanyo Sanso. The MS adsorber removes a part of the

hydrocarbons, eliminating the need for an acetylene adsorber and circulating adsorber and making the inside of the separator compact. Fig. 9 shows an example of the system diagram of an MG type air separation unit with MS adsorber.

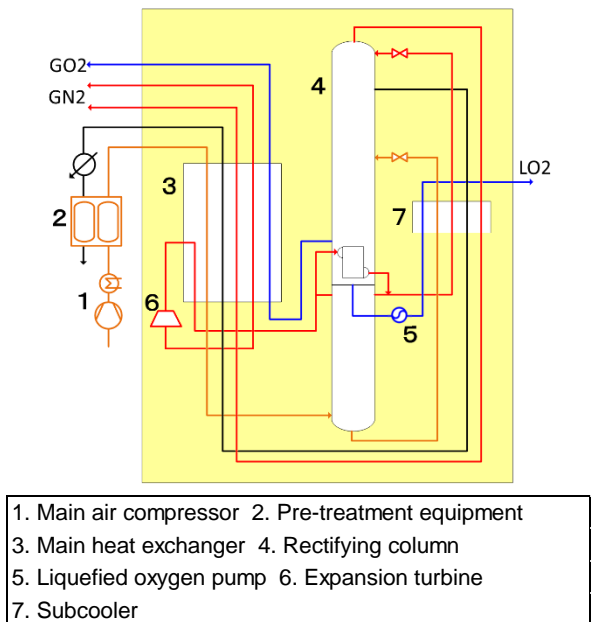


Fig.9 System diagram of MG type air separation unit

6.3 Normal-temperature TSA adsorber

The MS adsorber uses a TSA adsorber. Raw material air is compressed by an air compressor, cooled to about 40°C by a water-cooled cooler, and further cooled to 5°C to 10°C by a CFC cooling system before being introduced into the pre-treatment adsorber. In order to reduce the amount of regenerated gas used to regenerate the pre-treatment adsorber, and to save energy and reduce costs by downsizing the adsorber, it is effective to cool the air entering the adsorber with CFCs. However, Ozone Depleting Substances were abolished at the end of 1995, and it was decided that chlorofluorocarbons substitutes would be phased out starting in 2004. Therefore, we developed a CFC-free, normal-temperature TSA adsorber that eliminated the use of CFC cooler and complied with environmental regulations²⁰⁾.

This adsorber uses a multi-layer MS adsorber packed with activated alumina adsorbent and synthetic zeolite adsorbent. When the raw material air is introduced into the adsorber without being cooled by a CFC cooler, its temperature is about 40°C. In this case, about six times more moisture enters the adsorber than when cooling the raw material air to about 10°C using a CFC cooler, and

the amount of carbon dioxide adsorbed drops to about 1/3 due to the increase in the adsorption temperature. Therefore, to achieve the adsorption and regeneration conditions of the normal-temperature TSA adsorber without increasing equipment costs, it is necessary to develop a high-performance adsorbent, downsize the adsorber, shorten the switching time, and optimize the regeneration gas volume and regeneration temperature. By verifying these optimal operating conditions through experiments, we were able to obtain the data necessary to design a normal-temperature TSA adsorber. Then, as mentioned above, we adopted a CFC-free, normal-temperature TSA adsorber for the first time in a JNP type nitrogen generation unit.

Subsequently, we developed a simulator that enabled optimization studies of the operating conditions of pre-treatment facilities^{17-19, 36, 41)}. Until then it was believed that the components that solidified in the separation section of the air separation unit and caused blockages were water and carbon dioxide. However, it was found that also nitrous oxide (N₂O) may condense and solidify in liquefied oxygen or liquefied air and cause a blockage of heat exchangers and other equipment. Therefore, we used this simulator to predict the performance of adsorbents to develop a high-performance adsorbent that efficiently removes moisture, carbon dioxide, and N₂O⁵⁴⁾. It is expected that the use of this simulator will allow us to develop new adsorbents to further reduce costs and save energy.

6.4 Normal-temperature catalyst

As the density of devices in semiconductor manufacturing processes increases, there is a growing demand for higher purity product gases. In particular, nitrogen gas is used in large quantities, and it is very important to produce high-purity nitrogen. Therefore, we have developed a catalyst that can efficiently remove H₂ and CO, which are trace impurities contained in nitrogen. Trace amounts of H₂ and CO in air remains contained in product nitrogen gas if not specifically treated. These impurities are not considered a problem for general industrial gases, but their amount must be reduced to ppb levels for gases used in the semiconductor industry. Since H₂ and CO contained in air are easily converted to water and carbon dioxide by passing through a precious metal catalyst layer at about 190°C, and the generated water and

carbon dioxide are removed by adsorption in a subsequent MS adsorber, high-temperature catalysts for H₂ and CO removal were used in the past. However, catalysts that function at high temperatures require heating equipment to preheat the gas and catalyst columns packed with catalysts, and in addition, energy for high-temperature heating is necessary.

Therefore, we developed a normal-temperature catalyst with catalytic activity that allows the oxidation reaction to proceed at about 40°C, at which air is introduced into a TSA adsorber, and with the ability to adsorb by the catalyst itself the produced water and carbon dioxide³³⁾.

The developed normal-temperature catalyst is based on a carbon dioxide adsorbent (zeolite layer), coated with an alumina gel layer of about 100 μm on its surface, and carried with precious metals. Water and carbon dioxide produced in the oxidation reaction are adsorbed and removed by the catalyst's own alumina gel layer and zeolite layer.

The heating equipment and catalyst column can be eliminated by loading this normal-temperature catalyst together with adsorbent in the pre-treatment equipment. In addition, by loading the normal-temperature catalyst at the top of the multi-layer MS adsorber (at the rear of the zeolite layer), the effect of carbon dioxide on the oxidation reaction can be suppressed and the catalyst can be regenerated using adsorbent regeneration gas, which reduces the energy required for catalyst heating. Furthermore, catalyst poison contained in the air is removed by the alumina gel and zeolite layers, which extends the catalyst life.

The normal-temperature catalyst was mainly installed in nitrogen generation units that need to produce ultra-pure nitrogen gas. In addition, to produce ultra-pure nitrogen gas with an existing nitrogen generation unit, significant equipment modification was necessary in the case of using high-temperature catalysts, whereas only the addition of a catalyst to the adsorption column was required in the case of using normal-temperature catalysts.

However, since the manufacturing cost of a normal-temperature catalyst varies with the market price of the precious metal carried, it is necessary to continue the development of catalysts to maintain their high performance and keep their low price.

6.5 High-speed flow process

As air separation units became larger, there arose a demand for downsizing of the MS adsorber, which accounted for about 10% of the total installation area of the unit. We responded to this demand by increasing the flow speed of raw material air. By shortening the switching interval of the adsorption column (adsorption/regeneration cycle) and developing a large particle size MS adsorbent that prevents fluidization of the adsorbent, we were able to downsize the installation area and thereby reduce construction costs⁵³⁾.

7. Expansion turbine

Air separation units require an expansion turbine that generates cold to maintain the temperature of low-temperature parts and to compensate for heat ingress and the amount of cold in liquid products. In 1981, we started development of a blower breaking type hydrostatic gas bearing expansion turbine for nitrogen generation units based on our own technology with the aim of establishing of designing and manufacturing technology.⁵⁾

The development steps included the design of hydrostatic gas bearings and establishment of its manufacturing method, the design of high-efficiency turbine rotor blades and its manufacturing method, establishment of a performance test method, and the implementation of performance and reliability tests using actual equipment. As a result, we confirmed the performance and reliability through performance tests on a prototype, and adopted the first expansion turbine for a JN type nitrogen generation unit in 1985. As a result of trial operation of this unit, the expansion turbine efficiency of as high as more than 85% was obtained, compared to the planned efficiency of 78%. This expansion turbine features hydrostatic gas bearings, compact size with high-speed rotation, and a structure that is easy to disassemble and reassemble⁷⁾.

The expansion turbine was subsequently adopted in nitrogen generation units installed in TGCs around the country, contributing greatly to the widening of the TGC project, and is still used in nitrogen generation units. The type of expansion turbine is the NET-A when the process fluid is nitrogen gas or waste nitrogen gas and the expansion turbine is a hydrostatic gas bearing expansion

turbine. The NET-A2 and NET-A3 types are used in ordinary nitrogen generation units, depending on the process flow rate.

Hydrostatic gas bearing expansion turbines use process gas as the bearing gas, so there is no risk of contaminating the process even if bearing gas leaks into the process side, and they are excellent for high-speed rotation and low-temperature environments. However, their disadvantage is that the amount of nitrogen gas that can be delivered is reduced because about 30 Nm³/h of product nitrogen gas is used as the bearing gas.

So, we have developed a magnetic bearing expansion turbine that uses magnetic bearings to eliminate the use of bearing gas. This expansion turbine uses magnetic bearings instead of gas bearings, eliminating the need for bearing gas. The magnetic force generated by the electric current supplied to the magnetic bearings levitates the bearings, and the levitation position is maintained constant by controlling the electric current. In addition, this expansion turbine has excellent stability at high rotation speeds, and the operating condition of the expansion turbine can be observed by monitoring the current in the magnetic bearings. The type of magnetic bearing expansion turbine is NET-M³¹⁾.

As mentioned above, JNR type nitrogen generation units achieve low specific power consumption by employing a waste gas circulation cycle in which waste gas is circulated using the expansion turbine braking compressor. To realize this process, a turbine-driven low-temperature compressor, which is an improvement of the NET-A gas bearing expansion turbine, was developed³⁷⁾.

This process using the low-temperature compressor utilizes the expansion turbine as the compressor drive source, rather than simply as a cold source. This was made possible by taking advantage of the NET-A turbine's characteristics: it uses gas bearings, which have low bearing losses, and is suitable for low-temperature compressors because the bearing section is supported by a gas film on the rotating shaft, even at low temperatures.

By adopting this low-temperature compression-braking expansion turbine in JNR type nitrogen generation units, we achieved about 25% reduction in specific power consumption compared to a conventional system. The type of this low-temperature compression-braking expansion turbine is NET-C. The expansion turbines developed so far were small expansion turbines for

nitrogen generation units. However, we intend to expand our lineup of expansion turbines by developing a medium-sized expansion turbine that can also be applied to air separation units.

8. Operation control, remote monitoring, and remote operation

The operation control of nitrogen generation units is constructed as a total system including startup, shutdown, steady-state operation, and a fail-safe function in case of abnormality. The unit operation is full-automatically controlled. The unit is started by a startup command in both cases of startup from normal temperature and startup from a low-temperature state after a pause, and all processes are automatically operated, including starting and boosting of the air compressor, cooling and purity control of the unit, measurement of product purity, and transition to steady-state operation. The steady-state operation is automatically controlled to obtain high efficiency by constantly making corrections against disturbance factors such as fluctuations in product gas usage and changes in atmospheric conditions⁹⁾.

In 2001, we developed EzMPICS, an operation control system that uses a general-purpose PC and is equipped with a power-saving and low-cost controller, and adopted it for a nitrogen generation unit. The standard system configuration of EzMPICS consists of a control controller, a PC for building control programs, and a PC for operation, each of which is connected via Ethernet. The PC for operation is used to display data in the control controller and to operate the system, and can be monitored and operated remotely using digital lines³²⁾.

In 2002, we established JN General Monitoring Center on the premises of the Chiba Sanso Center to centrally manage JN type nitrogen generation units equipped with EzMPICS, which had been individually operated and managed in various locations throughout Japan⁴³⁾. Furthermore, we installed a web server to enable monitoring of JN units on our intranet, which allows for display alarms and trend data analysis.

On the other hand, in addition to automatic operation control such as automatic startup and shutdown, air separation units must be able to follow load changes over a wide operating range and at high speed while

maintaining high yields. To study such operation control methods, dynamic simulation technology is indispensable. We developed a dynamic simulation system for air separation units, compared the simulation results with plant data to confirm the reliability of the simulator, and used it to study the operability and controllability in planning a unit. The rectifying column using structured packing materials in place of the conventional sieve tray rectifying plates has a faster response speed and a larger response range than the sieve tray rectifying column, so the control method employed in the sieve tray rectifying column cannot be applied as is. In addition, the conventional air separation unit had attained a 100-70% turn-down operation with a changing rate of 3%/min, but a further higher changing rate could not be realized. We therefore used dynamic simulation to investigate the dynamic characteristics of units employing structured packing column, fixed the control model and disturbance model, designed a pre-compensator, and applied it to the actual plant, resulting in that a changing rate of 5%/min, the world's fastest level, was achieved. In this way, we have utilized dynamic simulation developed in-house to meet customer requirements and contribute not only to improvement of operational performance but also to shortening of commissioning periods by adjusting the control parameters in advance ^{12, 15, 24, 29, 35}).

To operate air separation units safely and efficiently, a lot of knowledge and experience is required. We developed an ASP (Air Separation Plant) simulator for training purposes and are utilizing it. This ASP simulator was developed as a derivative technology of the design process simulator used in our plant manufacturing department in the 1990s. This simulator has functions that can be used for basic training for beginner operators and abnormality responding training for intermediate and advanced operators. The simulator is also used by operators at about 30 plants nationwide through the adoption of a remote control system. The simulator has dynamic simulation models that reproduce our three standard ASP models, enabling training tailored to the scale and equipment of each production plant. Each model consists of a process section and a control loop section with the same calculation accuracy as the models used for process design, and is useful for understanding the distillation process by displaying the composition distribution in the rectifying column, as well as being

useful for operations such as turn-down operation of product and adjusting the purity ^{23,57}).

We are also working to improve operations using machine learning (ML) and the Internet of Things (IoT), which have shown remarkable development in recent years. In plants that operate multiple air separation units, it is desirable to combine their gas generation amounts to minimize power consumption according to the customer's usage. However, since the characteristics of power consumption differ among the units, optimization based solely on operator experience has limitations. Therefore, some production plants utilize a method of minimizing the power consumption of multiple air separation units by using a power estimation model constructed based on the operational data to optimize the combination of their product gas volumes ⁵⁹).

The liquefaction equipment at production plants uses a liquefaction cycle that compresses a large amount of nitrogen gas using Recycle Nitrogen Compressor, which consumes an extremely large amount of electricity, and the power consumption is closely related to the adjustment of the two turbines, making it very difficult to balance the two turbines. To address this issue, we have developed a technology to optimize the turbine adjustment by applying ML to the turbine adjustment, and have achieved success at eight production plants in Japan.

9. Future prospect

As mentioned above, we have developed technologies to meet various market needs for air separation units. In our history of the development of air separation units, which we have looked back on in this way, we have continued to develop processes for increasing the size and efficiency of equipment, develop pre-treatment equipment in response to environmental problems and safety measures, develop nitrogen generation units for larger nitrogen equipment and higher purity, and automate and labor-save to improve operability, and have completed and realized various new equipment and technologies.

In recent years, energy saving of equipment in air separation units that consumes a large amount of energy has become an important issue from the viewpoint of carbon neutrality.

The specific power consumption of air separation units

was 0.45 kWh/Nm³ in the 1970s, and improved by about 20% to 0.35 kWh/Nm³ in the 2000s with improved oxygen product yields due to more accurate distillation calculations, lowered pressure losses due to the use of packed columns, reduced lower column pressure due to lowered temperature differences in the main condenser, and improved performance of the main air compressor. However, there has been no improvement in specific power consumption in the most recent 20 years.

Similarly, the specific power consumption of nitrogen generation units was 0.30 kWh/Nm³ in the 1990s, and improved by about 30% to 0.195 kWh/Nm³ in the 2000s with the aforementioned improvements in air separation units as well as adoption of dual column nitrogen generation units through the development of a high-efficiency process. However, there has been no improvement after that and has reached a plateau.

Against this background, for air separation units, as described in Technical Report No. 39, we will confirm the effectiveness of a unit that can further improve the specific power consumption by 10% through development of more efficient processes by improving and enhancing the basic processes, and aim to expand their scope of application to realize air separation units that employ these new processes⁶³.

For nitrogen generation units, we aim to develop and commercialize ultra-pure oxygen co-production nitrogen generation units that allow for further increase in size and higher purification in order to meet the diversifying demands of the semiconductor market.

For large air separation units, larger sizes are expected in overseas markets, but further size increases are not expected in the domestic market, considering the consequent increase in back-up facilities. However, since the number of cases in which rare gas purification equipment is added to large air separation units is expected to increase in the future, we would like to further improve the efficiency of rare gas purification equipment and lower its cost.

Regarding operation control equipment, unmanned operation of units by remote monitoring and remote operation has been already in place in Europe and United States. In Japan, unmanned operation has not yet been realized due to legal restrictions; however, in preparation for future deregulation, we would like to develop equipment and technology that enable safe remote

monitoring and operation, using digital technology for realizing unmanned operation.

In addition, in order to suppress the increase in equipment costs and construction costs due to the recent rise in material prices, we aim to reduce the overall cost of a unit and shorten the overall delivery time to completion by further developing standard equipment and unitizing and modularizing components.

In the future, it will be necessary to maintain and expand these technological developments to achieve higher performance, lower power consumption, and lower price by further improving the efficiency of air separation units, and to develop competitive equipment in the market that matches customer requirements.

10. Conclusion

This paper presented our history of the development of air separation units. For the recent carbon neutral and SDGs initiatives, improving the efficiency of air separation units, which consume a large amount of electricity, plays an important role in contributing to energy conservation. We believe that the development of highly efficient industrial gas production processes, remote monitoring using digital technology, efficiency improvement through remote operation, and optimization of operations through the introduction of advanced control technology will contribute to carbon neutrality and productivity improvement. In the future, by utilizing our sustainable technology, development capabilities, and experience, we intend to contribute to the development of all industries and solutions to social issues by developing air separation units that meet diversifying market needs.

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