Technology

Nitrogen Generation Process for Co-production of Small Amounts of Ultra High Purity Oxygen and Argon

ARAKAWA Moemi* TACHIBANA Hiroshi*

1. Introduction

1.1 Background

In the semiconductor manufacturing process, a large amount of nitrogen is consumed for various applications such as carrier gas and security purposes. In addition, the most advanced processes require ultra-high purity oxygen with impurities kept to a strict level of less than 10 to 100 ppb and argon. Table 1 shows examples of recent product specifications for semiconductor factories. In the past, gas supply for semiconductor factories was a combination of production of nitrogen gas at on-site nitrogen generation units (NGUs) and supply of liquefied oxygen and argon by lorry from their production plants. In recent years, however, as semiconductor factories have become larger, the supply volume of various gases has increased, and on-site production of oxygen and argon is now called for as well. In response to these requirements, we proposed a combination of existing processes, but because of its complicated configuration and high power consumption, the development of equipment suitable for recent product specifications was called for. In addition, due to the increase in the size of equipment, it was necessary to take a different approach to backups in the event of a shutdown.

Against this background, we have developed an NGU capable of efficiently and stably co-producing ultra-high purity oxygen and argon. This report gives an overview of the developed process and introduces the backup technique required for larger NGUs in the event of a shutdown.

Table 1 Examples of recent product specifications for semiconductor factories

Product	Flow rate [Nm^3/h]	Purity
Nitrogen	25000	$O_2 \leq$ 10 to 1000 ppb
Oxygen	1000	$O_2 \geq$ 99.6% to 99.8%
Ultra-high purity oxygen	200	N_2, Ar $\leq$ 10 ppb
Argon	100	N_2, $O_2 \leq$ 1000 ppb

Note: In addition to the above, the purity of each product includes hydrocarbons (THC) ≤ 10 to 1000 ppb.

* Development Section, Engineering Department, Project Solution Management Division, Plant Engineering Center

2. Overview of the developed NGU process

2.1 NGU that co-produces ultra-high purity oxygen

The purity of oxygen produced in a typical air separation unit (ASU) is 99.6% to 99.8%, and argon accounts for most of the remaining 0.2% to 0.4%, and impurities such as hydrocarbons (THC) of several tens to several hundreds of parts per million are also contained. On the other hand, for ultra-high purity oxygen, removing each impurity up to 10 to 100 ppb is required. Argon and nitrogen in oxygen cannot be removed by a purifier, so they must be removed by distillation purification. THC can be removed by a purifier, but needs to be reduced to several tens of parts per million at the inlet of the purifier.

Figure 1 compares the process flow of (a) the conventional process and (b) the new process. In the case of producing ultra-high purity oxygen in addition to nitrogen gas, conventional technology co-produces ultra-high purity oxygen by combining an oxygen column, a high-purity oxygen column, various heat exchangers, and rotating equipment such as a recycle nitrogen compressor (RNC) in the downstream of a two-column NGU [1].

The removal of THC from the produced oxygen is performed in the high-pressure section of the high-purity oxygen column, and the removal of argon is performed in the oxygen column and the low-pressure section of the high-purity oxygen column, and these cause the complexity of the equipment configuration and the increase in power consumption of the conventional process.

The new process, as in the conventional process, is based on a two-column NGU with an oxygen column in the downstream, but has a simplified configuration by eliminating the high-purity oxygen column. The removal of

THC is performed by increasing the number of stages in the lower part of the 2nd distillation column. The liquefied air from which THC has been removed is withdrawn from the middle of the 2nd distillation column, and argon and nitrogen is removed in the oxygen column. By integrating the impurity removal functions in the lower part of the 2nd distillation column and the oxygen column, ultra-high purity oxygen can be obtained with a simpler process and equipment configuration than in the past. It should be noted that the nitrogen yield and power consumption are almost the same as those of a two-column NGU that produces only nitrogen, and the reduction in nitrogen yield and the additional power required due to oxygen production are only a few percent.

2.2 NGU that co-produces ultra-high purity oxygen and argon

Figure 2 compares the process flow of (a) the conventional process and (b) the new process. In co-producing ultra-high purity oxygen and argon in addition to nitrogen gas, the conventional technology combines a conventional ASU, which is a base component consisting of a low-pressure column, a high-pressure column, a crude argon column, and an argon purification unit, with a high-purity oxygen column, various heat exchangers, and RNC in the downstream to enable the co-production of ultra-high purity oxygen and argon. However, since this process is based on the conventional ASU, a large amount of excess oxygen and argon produced as by-products of nitrogen must be partially wasted, which is inefficient,

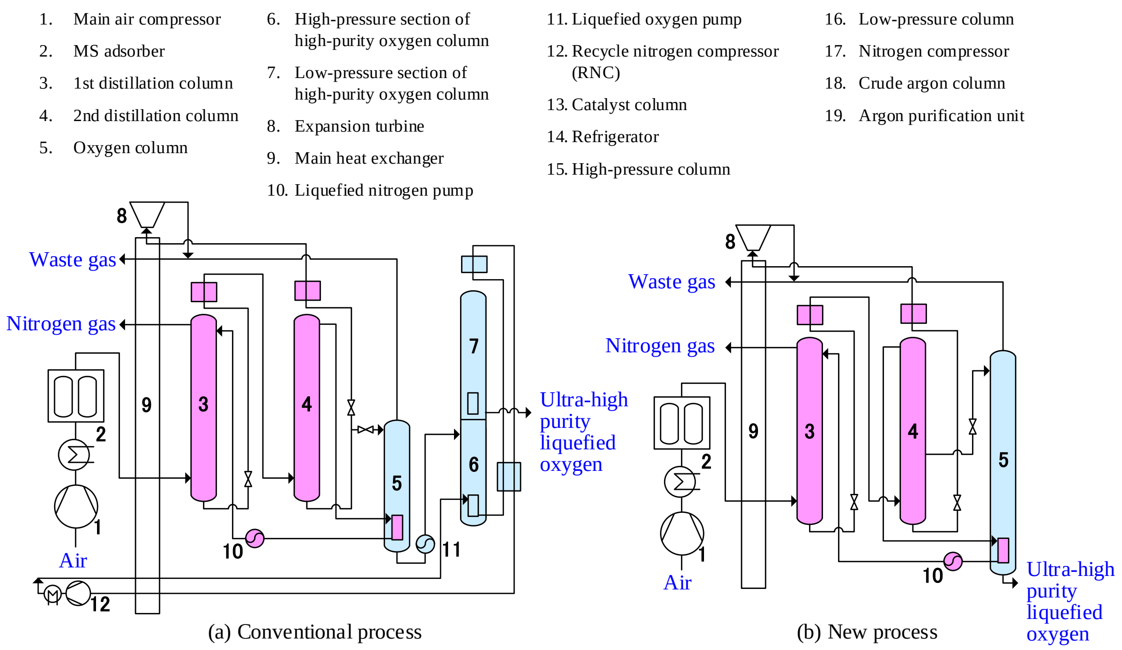

Figure 1 Process flow of NGU that co-produces ultra-high purity oxygen

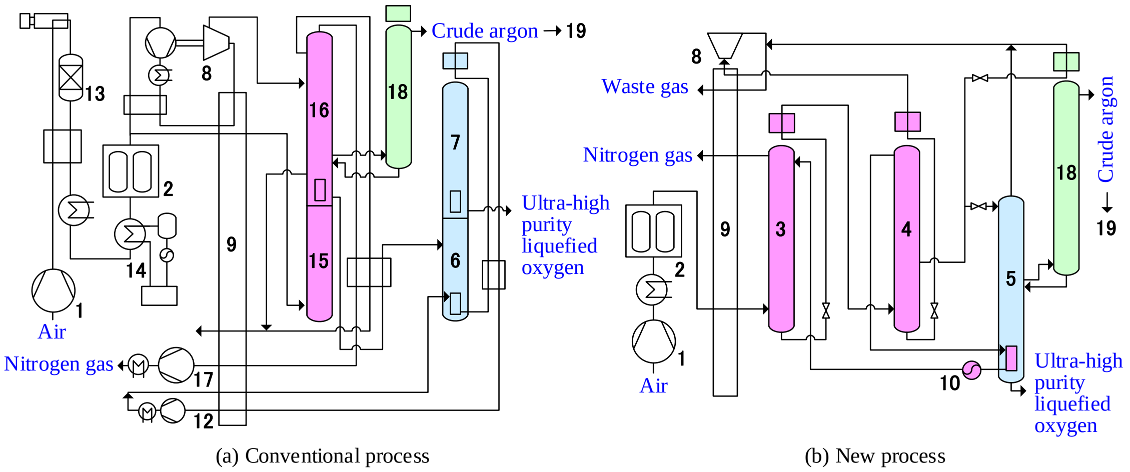

Figure 2 Process flow of NGU that co-produces ultra-high purity oxygen and argon

consumes a lot of power, and requires a complicated equipment configuration. In addition, the discharge pressure of the main air compressor is lower than that of typical NGUs in the conventional process, requiring larger and more complex pretreatment equipment such as a catalyst column and MS adsorbers.

The new process is based on the above-mentioned process of NGU, which co-produces ultra-high purity oxygen. By adding a crude argon column and an argon purification unit in the downstream, the process also co-produces argon in addition to ultra-high purity oxygen. Thus, the new process has a simplified equipment configuration and achieves a power reduction by more than 20% through efficient product production. Additionally, since the discharge pressure of the main air compressor in the new process is equivalent to that of typical NGUs, the pretreatment equipment does not require a catalyst column and refrigerator, making it simpler.

3. Backup during the shutdown of NGUs

3.1 Conventional backup system

Figure 3 shows a comparison of the plant configuration of conventional and the recent large-scale equipment. The conventional configuration shown in Figure 3(a) is a simple setup consisting of one train of NGU and large liquid storage tanks. During normal operation, the NGU is operated at 100% capacity to supply the whole amount of gas demand. During maintenance or emergency shutdowns when the NGU is not operated, the supply is backed up by vaporizing liquefied gas from the liquid storage tanks. While small NGUs have no problem with this configuration, large NGUs producing tens of thousands of Nm^3/h of nitrogen require larger storage tank facilities. In addition, if the backup is required over a long period of time, it may be difficult to supply continuously even with larger storage tanks.

3.2 Recent backup system for large NGUs

Figure 3(b) shows a recent configuration designed to provide a stable gas supply even in the case of large NGUs, consisting of a 2-train NGU and small liquid storage tanks. During normal operation, the product is supplied at 50% turndown operation from each NGU, and in the event that one unit stops, the other unit is increased to 100% to supply the product. In this case, the gas supply from the storage tanks is required only for a few tens of minutes during the load-up operation, thus minimizing the backup liquid capacity.

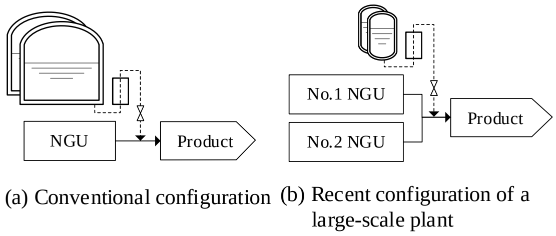

Figure 3 Comparison of plant configurations between conventional and recent large-scale NGUs

3.3 Verification of dynamic behavior at the start of backup operation using dynamic simulation

In order to provide a stable gas supply using a backup system for recent large-scale NGUs, a rapid and stable load-up operation is required in the event of an emergency shutdown. Therefore, a simulation was conducted using a dynamic model in the ultra-high purity oxygen co-production process to increase the amount of the nitrogen product from 50% to 100%, and the dynamic behavior during that time was verified. As a result, although there were temporary fluctuations in product purity as shown in Figure 4, it was confirmed that a stable increase in nitrogen product from 50% to 100% at a increasing rate of 5%/min is possible.

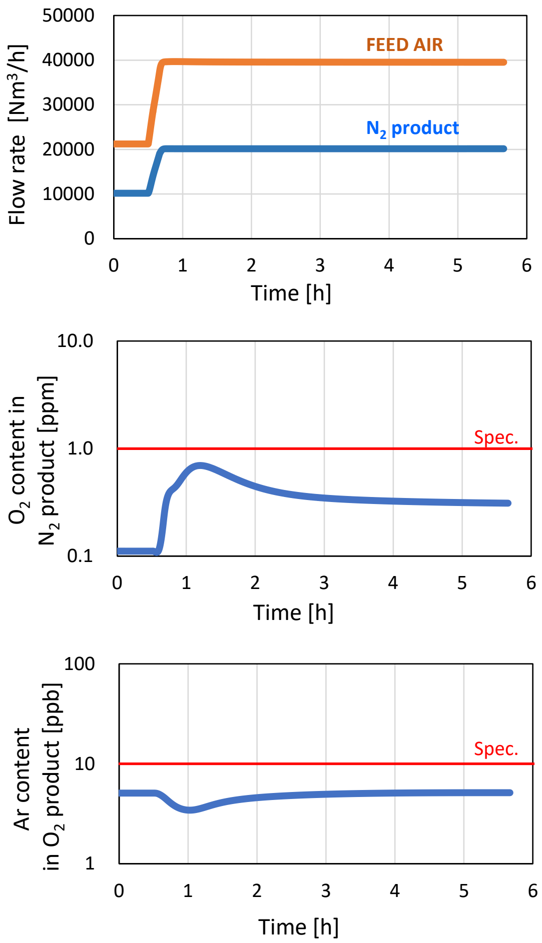

Figure 4 Results of dynamic simulation of load-up operation (from 50% to 100%, increasing rate of 5%/min) for NGU that co-produces ultra-high purity oxygen

4. Conclusion

By modifying the process of a two-column NGU, we have developed an NGU capable of co-producing ultra-high purity oxygen, as well as an NGU capable of co-producing both ultra-high purity oxygen and argon while maintaining the nitrogen yield, with the same power consumption as -the conventional two-column NGU that produces only nitrogen. Both new developed NGUs reduce the number of components compared to conventional systems, resulting in lower initial costs, maintenance costs, and power consumption. In addition, dynamic simulations have confirmed that a load-up operation from 50% to 100% at a rate of 5%/min can be stably performed.

Currently, we have begun applying this process to NGUs that co-produce ultra-high purity oxygen. We will continue to promote the use of this process to contribute to lower unit gas prices, stable supply, and reduction in environmental impact.

Reference

1) Makoto Irisawa, Nitrogen Generation Unit MG10600, Taiyo Nippon Sanso Technical Report, No. 22, 2003