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Economical Clean Dry Air System for Closed Manufacturing System

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Abstract—We demonstrated that the organic carbon contaminants influenced the J–E characteristics of a gate oxide. Molecular-contamination-free clean dry air (CDA) is a choice for the atmosphere control in a closed manufacturing system. The adsorption and desorption characteristics, which are important items for the design and operation of CDA production plants, were confirmed with both a dynamic simulation and a pilot column. Based on the results, we built a CDA plant with the capacity of 4000 m³/h, and confirmed that each contamination concentration was less than 10 ppb. We also proposed the economical CDA system using a power recycle device which has been newly developed. Using this CDA system, it is possible to reduce the power consumption in the CDA plant. Also, it is possible to realize the circulation of the used CDA without not only generating more contamination but also power consumption.

I. INTRODUCTION

AS MANY as 10–100 million active devices are to be integrated on a single chip of a deep submicron design rule ultra large scale integration (ULSI). A huge number of transistors on a wafer must behave exactly as they are designed, and such a tight control during manufacturing must be preserved from wafer to wafer and lot to lot during a long period of production. Perfect uniformity and perfect reproducibility must be guaranteed for the deep submicron wafer manufacturing lines. Low cost production of ULSI is also required for the present manufacturing lines.

The manufacturing costs of ULSI can be suppressed by reducing the running costs of the clean room. One of the measures being taken to reduce running cost of clean room is to minimize intake volume of fresh air. As a result, a large amount of molecular impurities generated in the clean room remains in the clean room. If a wafer is exposed to clean room air just for a moment, a large amount of molecular contaminants, such as moisture, organic carbon, and siloxane adhere to the wafer's surface because the clean room is nothing more than dustless air and is full of oxygen (O₂) and moisture (H₂O), and also other gaseous impurities [1], [2]. Fig. 1 shows the time dependence of organic carbon contamination on a Si surface from clean room air. The initial wafers were obtained by the Dynamic-Spin-Cleaning method with the ozonized ultrapure water[3], and were set under 1 m from fun-filter-unit (FFU)

in order to remove the influence of particle adhesion. The organic carbon concentration in cleanroom was about 50 ng/m³ in terms of n-hexadecane concentration. The organic carbon concentration on wafer was measured by FT-IR-ATR. The vertical axis in Fig. 1 shows a calibrated concentration in terms of CH₂ with the calibration method using the Langmuir–Blodgett-monolayer [2]. A large amount of molecular contamination adheres to the wafer surface when a wafer is only exposed to clean room air in 1 h. The amount of molecular contamination increases with increasing exposure time. In this case, cleaning processes will be required before critical processes. As a result, the number of process steps will increase, and it will lead to an unacceptable increase in production costs. Moreover, the native oxide, which is a very difficult contaminant to remove, grows on the wafer surface within a few hours of exposure [4]. This is because of the coexistence of O₂ and a few percent H₂O. Therefore, the closed manufacturing system [5] consisting of wafer transportation and wafer storage in a molecular-contamination-free ambient atmosphere, such as clean dry air (CDA) seems to be useful in removing unknown external factors and realize perfect process parameter control. For control of the molecular-contamination-free ambient air in the closed manufacturing system, CDA, which consists of 79% nitrogen (N₂) and 21% O₂ with H₂O and organic carbon concentrations of less than 1 ppb and 10 ppb [6], respectively, is necessary for very large volume capacity production lines. The initial cost of cleanroom is estimated to reduce about 300 000 yen/m² by introducing the closed manufacturing system. This is because the cleanliness of cleanroom can be down to class 1000 from, for example, class 1. The running cost of cleanroom is also estimated to reduce about 60 000 yen/year · m² for low differential pressure operations because the chemical filter is unnecessary for removing the gaseous impurities. Although the production costs of CDA are relatively expensive, it is possible to keep the CDA cost to less than 1 yen/m³ or 1 cent/m³ for cost effective manufacturing [7].

The goal of this work is to realize the economical CDA system, which can produce CDA with H₂O and organic carbon concentrations of less than 1 ppb and 10 ppb and with the cost of less than 1 yen/m³ or 1 cent/m³ for a closed manufacturing system. For this purpose, we investigated the basic modules of the CDA production plant, such as the catalyzer and adsorber characteristics with dynamic simulation, and we have developed a CDA production plant with the capacity of 4000 m³/h as the pilot plant using the results of dynamic simulation. We have also studied the CDA production method using a power reuse device. Moreover, we have studied the circulation of the used CDA by the power recycle system for not only realizing

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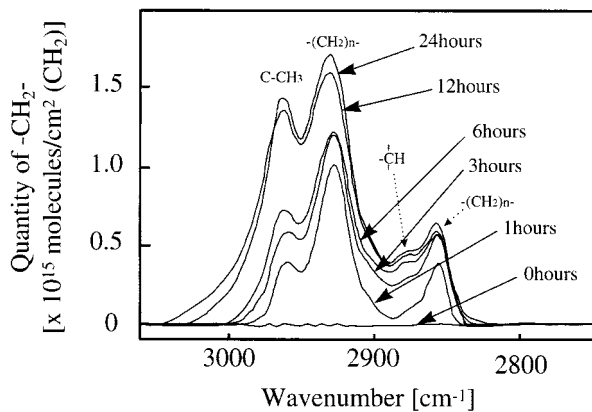


Fig. 1. Organic carbon contamination on Si surface from clean room air.

a contamination-free condition but also for reducing the circulation costs. In this paper, we have demonstrated the influence of airborne contaminants after the gate oxidation processes and have introduced the characteristics of the basic modules and the performance of the pilot plant. We have also proposed the economical CDA system using a power recycle device.

II. INFLUENCE OF AIRBORNE ORGANIC CONTAMINANTS ON GATE OXIDATION PROCESS

MOS capacitors were fabricated with phosphorous-doped polysilicon gates and gate oxides on p-type silicon substrates for investigating the influence of the airborne organic contaminants. A field oxide of 600 nm thick was formed by steam oxidation at 1000 °C. The active area was patterned by wet chemical etching with a surfactant-added BHF (HF-NH₄F) solution. The wafers were then cleaned by the modified RCA cleaning method [8]. After the chemical oxide formed during RCA cleaning was removed by diluted HF (0.5%), the passivated chemical oxides, which were effective for improving gate oxide integrity and reliability, were immediately formed by immersing in 2 ppm ozone (O₃)/H₂O at room temperature for 20 min [9]. The gate oxidation was carried out in the ultraclean oxidation furnace maintained 900 °C using an IR lamp [10], [11]. The oxide was formed in 2% H₂O/argon (Ar) ambient with a thickness of 5 nm. After gate oxidation, wafers are transported after exposed to cleanroom air with organic carbon concentration of about 50 ng/m³ in terms of n-hexadecane or transported without exposure to the cleanroom air. Then the gate electrode is deposited.

Fig. 2 shows the J-E characteristics of the gate oxide with or without exposure to clean room air after the gate oxidation. The measured gate area of MOS capacitor was 4×10^{-2} cm², and the results of typical ten pieces were demonstrated in Fig. 2. As shown in Fig. 2(a), the dielectric breakdown voltage of the gate oxide without exposure to clean room air is 15 MV/cm at a thickness of 5 nm with highly reliability. Since no oxidation agent is used in the gate electrode deposition process, the carbon contaminant remains on the gate electrode/oxide interface. Carbon contamination on the electrode/oxide interface degrades the electrical characteristics of the gate oxide as shown in Fig. 2(c) and (d). Similar results were already reported [12]. Therefore, it is necessary not to expose the wafer to clean

room air between the gate oxidation process and gate electrode process. To meet this requirement, a closed manufacturing system including the closed transportation system must be introduced.

III. CDA MANUFACTURING SYSTEM

A. Basic Modules of Clean Dry Air System

The method using a catalyst made of a noble metal can oxidize organic carbon and hydrogen (H₂) with O₂ in air, while H₂O and carbon dioxide (CO₂) can be mainly removed by the adsorption method using activated alumina and zeolite which needs a regeneration process. The impurities, which adsorbed onto the adsorber surface, must be desorbed from the surface during the regeneration process until the impurities break through from other column in order to continuously obtain CDA. A schematic of the thermal swing adsorption system with two columns is shown in Fig. 3. The adsorption column with a diameter of 1200 mm is filled with activated alumina in the lower one and zeolite in the upper one. The thickness of activated alumina and zeolite were 400 and 1100 mm, respectively. When the adsorption process in column A starts, the regeneration process in column B starts in this thermal swing adsorption. In the regeneration process, at first, the adsorbed impurities with 0.5 MPa air are exhausted till the pressure of 0.1 MPa in depressurization step. The heated CDA at the temperature of 423 K is introduced from top of the column B for reducing a period in the heating step. After cooling, the column B is prepared for next adsorption process during fulfilling with CDA. The period of pressurization step needs about 20 min, because CDA flow rate is small for prevention of CDA pressure variance. As mentioned earlier, because four steps continuously occur in the regeneration process, the step influences the next step. Therefore, the simulation, which can postulate the state in the column in advance, is needed for optimization of the dimensions and operating conditions. Especially, in order to realize a cost-minimum operation, it is important to reduce the quantity of discharge heat with the regeneration gas throughout the system. A dynamic simulation technique is needed for this purpose.

B. Dynamic Simulation

Previous to the dynamic simulation, we produced a two-layer type adsorption column with two gas sampling points and five temperature measuring points, as shown in Fig. 3. Two gas sampling probe tubes were set at the top of activated alumina layer, and at the point of 250 mm from under end of zeolite layer, respectively. The thermocouples in activated alumina layer were set at the points of 20 mm from both of end. The thermocouples in zeolite layer were set at the points of 20, 500, and 750 mm from the top end, respectively. The behavior of the CO₂ concentration and temperature in the adsorption column was studied by measurements and dynamic simulation. We considered the heat of adsorption of adsorbent in the column, assuming that the temperature and concentration profiles for the vertical direction of air flow can be negligibly small. We also used an ideal adsorbed solution (IAS) [13], [14] theory for calculation of the relation during equilibrium adsorption. Fig. 4 shows the breakthrough

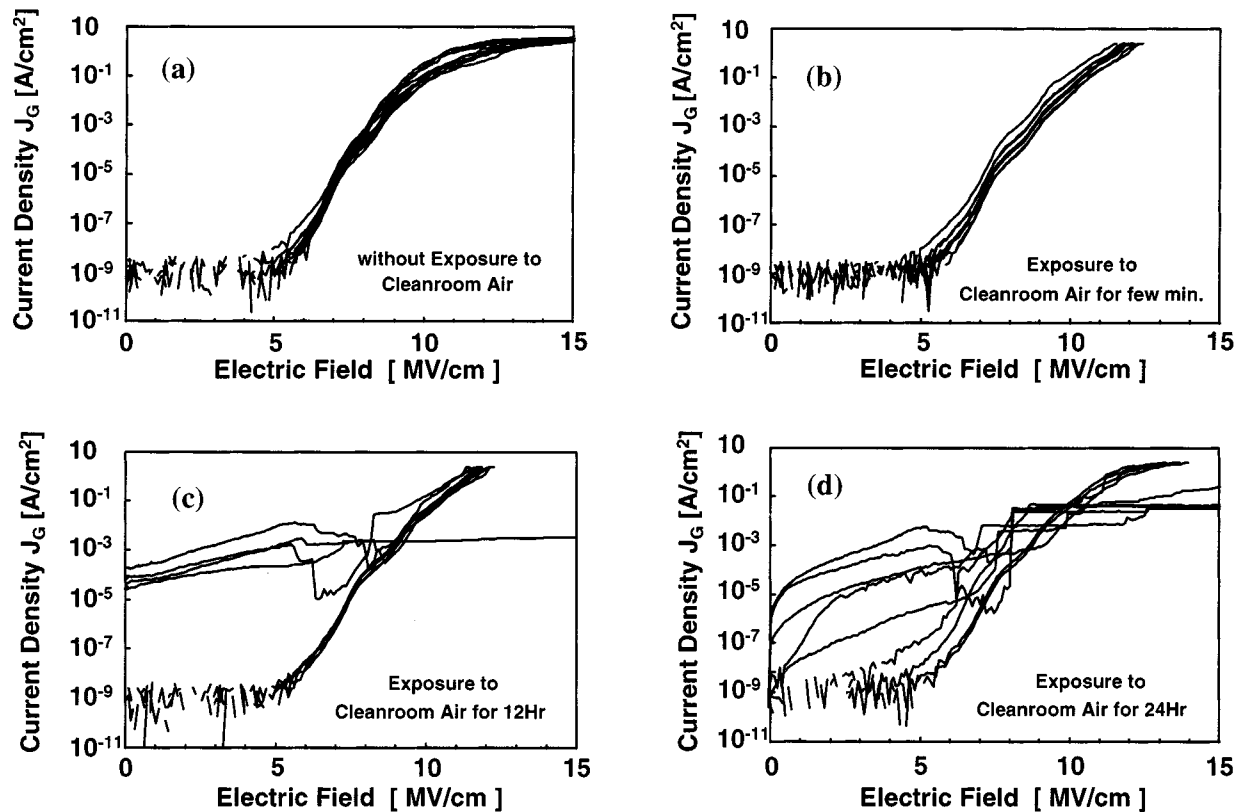


Fig. 2. J-E curves of gate oxides with or without carbon contamination at gate-electrode/oxide surface. (a) Without exposure to cleanroom air. (b) Exposed to cleanroom air for few minute. (c) Exposed to cleanroom air for 12 h. (d) Exposed to cleanroom air for 24 h.

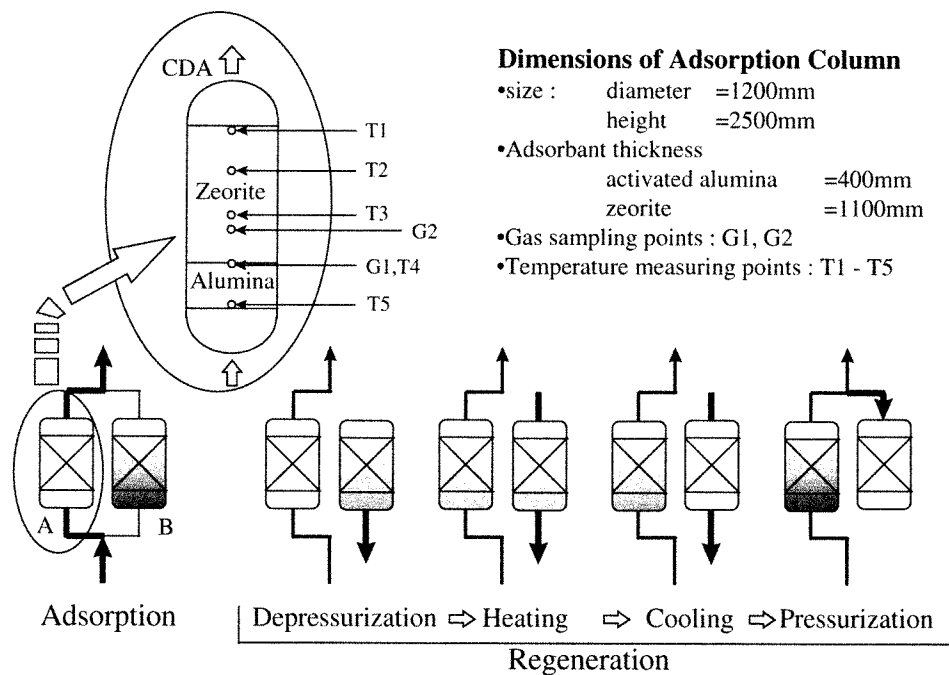


Fig. 3. Schematic of the thermal swing adsorption system with two columns.

curves of CO_2 in the adsorption column. The broken lines are obtained by dynamic simulation, and the solid lines are CO_2 concentrations measured using an IR absorption spectrometer. CO_2 adsorbed in zeolite layer, did not almost remove in acti-

ated alumina layer. The profiles of breakthrough by dynamic simulation agreed with measured breakthrough profiles.

Temperature profiles of the adsorption column during the regeneration process are shown in Fig. 5. The temperature in the

adsorption column varied complicatedly with the position in the column and with the steps such as depressurization and heating. The dynamic simulation used IAS theory that could express the complicated temperature profiles with good agreement. The complicated temperature behavior in the adsorption column can be described as follows: the depressurization step progresses in a few minutes from the start of the regeneration process. Therefore, the temperature of the zeolite layer abruptly drops due to the heat of desorption of the adsorbed N₂. After the depressurization step, the temperature of the zeolite layer is kept constant at 267 K by the heat of desorption of the adsorbed CO₂ in the heating step. However, the temperature of the zeolite layer abruptly rises with continuous heating by high temperature CDA. On the other hand, the temperature of the activated alumina layer slowly dropped due to the heat transfer from the zeolite layer. The plateaus are observed after raising the temperature. It is seemed that the appearance of plateau A is mainly originated the heat of desorption of CO₂ by using dynamic simulation. Also, it seems that the plateaus at B and C are mainly originated the influence both of the heat transfer and the heat of desorption of CO₂, and originated the heat of desorption of H₂O.

As already described, the temperature profiles indicate a complicated behavior with time for the regeneration steps. In order to realize both an easy scale up and optimization of operation conditions in the thermal swing adsorption system, it is necessary to understand the thermal behavior in the adsorption column during regeneration process by dynamic simulation as exactly as possible.

C. Clean Dry Air Plant with Capacity of 4000 m³/h

Fig. 6 shows a schematic of the CDA production plant with the designed capacity of 4000 m³/h based on dynamic simulation. The footprint of this plant is about 160 m². CDA and ultraclean N₂ gases were coproduced by this plant. Air was introduced to the air compressor through an air filter, and compressed at a fixed pressure. H₂ and carbon monoxide (CO) in the air were oxidized with O₂, and H₂O and CO₂ were generated in the catalyzer column. H₂O and CO₂ including generated H₂O and CO₂ were removed in the thermal swing adsorption system that consisted of a two-layer type adsorption column. The pre-cooling unit set between the catalyzer column and the thermal swing adsorption system was used for the load reduction in the thermal swing adsorption system by removing a few percent of H₂O in the air. Two methods exist for obtaining the regeneration gas. One is reusing part of the generated CDA, and the other is using the exhaust gas from the ultraclean N₂ production plant. Regeneration gas could be easily obtained according to the ratio of needs for CDA and N₂.

The contaminants removal effect of the catalyzer in the CDA system is shown in Fig.7. The contaminants were continuously measured at the outlet of the CDA plant using a trace reducing gas analyzer with specific detection of the reduced mercury vapor combined with gas chromatograph with flame ionization detector (GC-FID) [15]. The contaminants for removal are CO and H₂, which usually exist at a ppm level in air. CO and H₂ could be removed below 10 ppb by passing them through the catalyzer column.

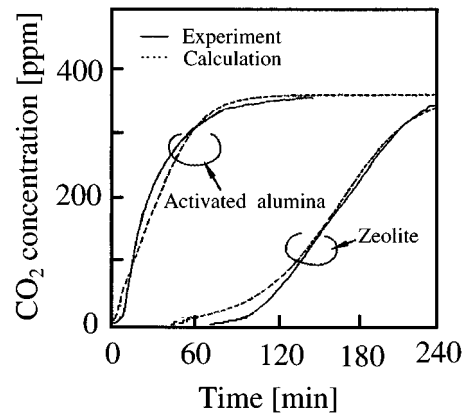


Fig. 4. Breakthrough curves of CO₂ in the adsorption column.

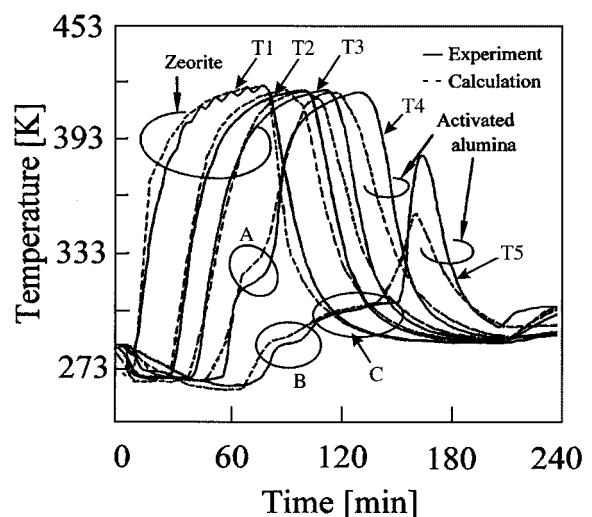


Fig. 5. Temperature profiles of the adsorption column in regeneration process.

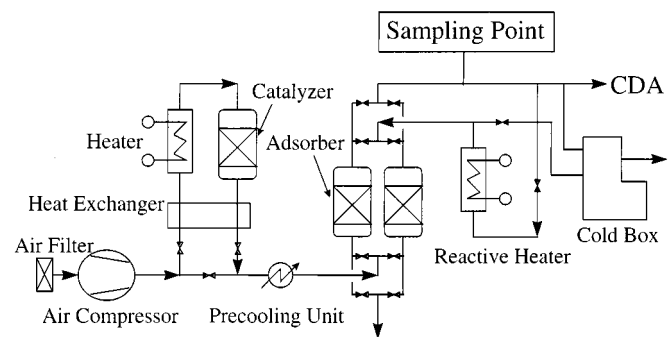


Fig. 6. Schematic of CDA production plant with the capacity of 4000 m³/h.

Fig. 8 displays the H₂O concentration at the outlet of the CDA plant measured using a trace moisture analyzer based on chilled-mirror which is detectable to 0.5 ppb. The average H₂O concentration at the outlet of the CDA plant was about 2.6 ppb with small variation. The H₂O concentration slowly decreased according to elapsed measured time. The reason for this is speculated to be the adsorbed H₂O desorbed from the inner surface of the stainless steel sampling tube having an inner diameter of 4.35 mm and length of 70 m.

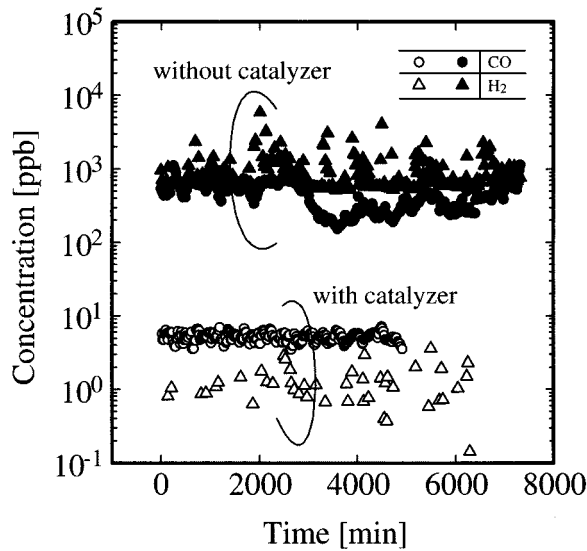


Fig. 7. Contaminants removal effect of catalyzer in the 4000 m³/h CDA system.

The average concentration and the standard deviation of the contaminants are shown in Table I. The average and standard deviation values were calculated from each contaminant concentration which was measured every 15 min for 7000 min. As mentioned earlier, the H₂O concentration does not satisfy the target value of below 1 ppb yet. The impurities concentration except H₂O were below 10 ppb which is the target value.

IV. ECONOMICAL CDA SYSTEM FOR CLOSED MANUFACTURING SYSTEM PROPOSAL

A. CDA Plant

The CDA is recommendable to use at very low pressure, such as 100 mmAq. at the point of use (POU). The CDA pressure at the POU is still low enough, even adding the pressure loss in the CDA supply system, as compared with the pressure of 0.5 MPa, which is usually the operation pressure in the CDA production plant. The difference in pressures is consumed as electric power because the cost of compression is almost proportional to the electric power. In order to reduce the electric power consumption, it is necessary to reduce the pressure for pressurizing raw air. Fig. 9 shows the concept of the power recycle system in CDA plant. When the raw air is pressurized to 0.5 MPa, excluding the pressure loss of α in the conventional plant with a capacity of 4000 m³/h, 84% of the total running cost is consumed as the electric power for pressurizing the raw air. We developed a compressor combined turbine with a shaft and shaft bearing using a gas film as the power recycle system and used it for the CDA plant. The raw air pressure at the outlet of compressor was reduced to 0.3 MPa using the power recycle unit when the inlet pressure of the purification process unit such as the catalyzer is 0.5+ α MPa and the outlet of the power recycle unit is 0.1 MPa. As a result, we have confirmed that the total running cost of the CDA manufacturing can be reduced by about 23%.

The relation of the H₂O enter rate and the H₂O concentration for each purge CDA volume is shown in Fig. 10. The horizontal axis shows the H₂O enter rate, which was calculated by

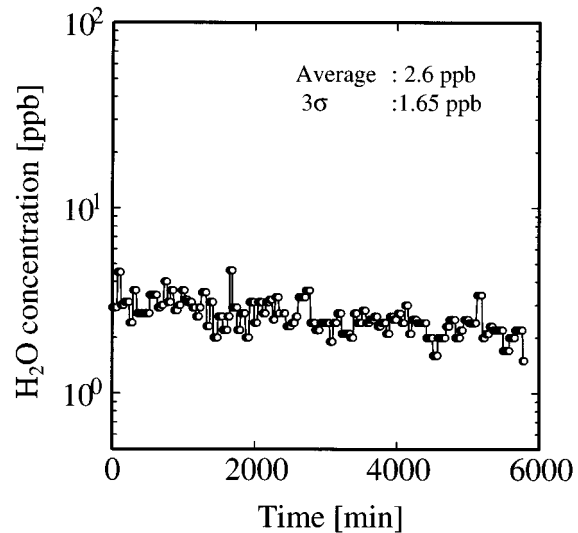


Fig. 8. H₂O concentration at the outlet of CDA plant measured by trace moisture analyzer.

TABLE I
AVERAGE CONCENTRATION AND THE
STANDARD DEVIATION OF CONTAMINANTS

Contaminant	[ppb]			
	H ₂ O	H ₂	CO	CO ₂
Average	2.6	1.3	5.1	2.9
3 σ	1.65	2.13	3.48	5.43

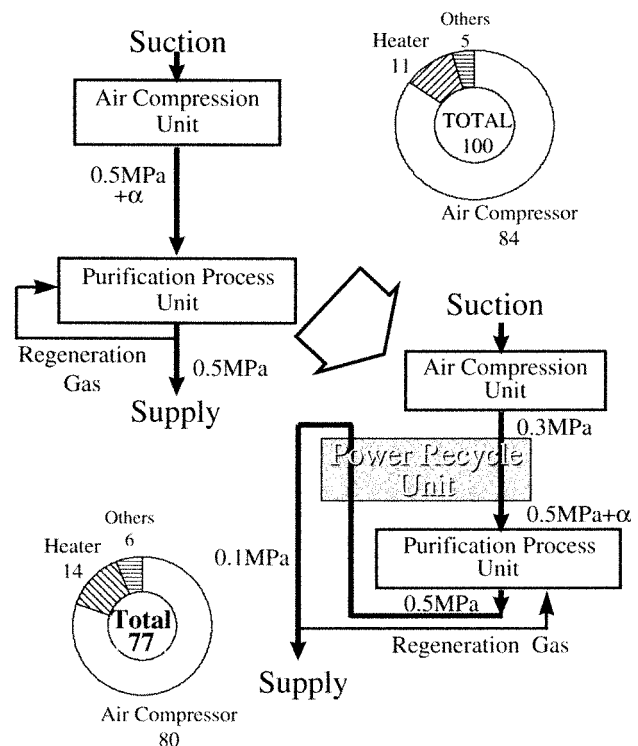


Fig. 9. Concept of power recycle system in CDA plant and its effect.

the throughput of wafers and the equilibrium H₂O concentration on the wafer in the clean room air. Each solid line indicates

the H₂O concentration in the CDA for each purge volume. Here, the material balance of H₂O in a load lock room is expressed by

$$V \cdot \frac{dC}{dt} = Q_1 \cdot C_0 - Q_1 \cdot C_3 + C_1 \cdot S - C_4 \cdot S \quad (1)$$

where

V, Q_1, C_0, C_3 : the volume of load lock room, the flow rate of CDA, the initial concentration of H₂O and the H₂O concentration in outlet of load lock room, respectively;

C_1 : the equilibrium adsorbed H₂O molecules on the wafer surface, which is reported to be about 10¹⁷ molecules/cm² with exposure to cleanroom air [1];

S : the surface area of wafers for unit time.

For example, supposing that the wafers of 10% for number of total process steps of 350 are stored in the wafer storage for 200 mm-5000 wafers/month production line, the enter rate of H₂O ($C_1 \cdot S$) is estimated by

$$\begin{aligned} C_1 \cdot S &= 10^{17} \times 0.1 \times 350 [\text{step}] \times \frac{5000 [\text{W/M}]}{30 [\text{D}] \times 24 [\text{h}]} \\ &\quad \times 628 [\text{cm}^2] \\ &= 1.5 \times 10^{22} [\text{molecules/h}]. \end{aligned} \quad (2)$$

In stationary state, $dC/dt = 0$, and as C_2 is negligible small as compared with C_1 , the relation of the H₂O concentration in outlet ($Q_1 \cdot C_3$) and the H₂O enter rate ($C_1 \cdot S$) is expressed by

$$C_3 = C_0 + \frac{C_1 \cdot S}{Q_1}. \quad (3)$$

In other experiment, we have also confirmed that the equilibrium H₂O concentration on the surface is less than 3×10^{14} molecules/cm² for the H₂O concentration of 100 ppb in the gas phase at room temperature. Supposing that the remained H₂O concentration on the wafer surface is less than 10¹⁵ molecules/cm², the required volumes of purge CDA were estimated as 5000 m³/h in the production line with a capacity of 200 mm-5000 wafers/month, as shown in Fig. 10. The CDA used for purge is exhausted to the clean room, because the H₂O concentration increases due to desorbed H₂O molecules from the wafer's surface. Therefore, it is considered that the minimum CDA volume produced in the CDA plant equals the exhausted CDA.

B. Economical CDA System Including Circulation System of Used CDA

Recycling of the used CDA is indispensable in order to obtain an inexpensive and reliable CDA. It is essentially important to prevent contamination entering into the POU such as the wafer strage, transportation system, etc., including the return duct and circulation device of CDA. However, it is difficult to prevent generation of contamination and particles from the conventional blower and/or fan because the conventional blower and/or fan, which uses an electric motor, has a shaft for transferring the power from the motor. We have designed the system consisting of a wafer supply and store unit (WSSU) and recycle device which can efficiently supply the used CDA for

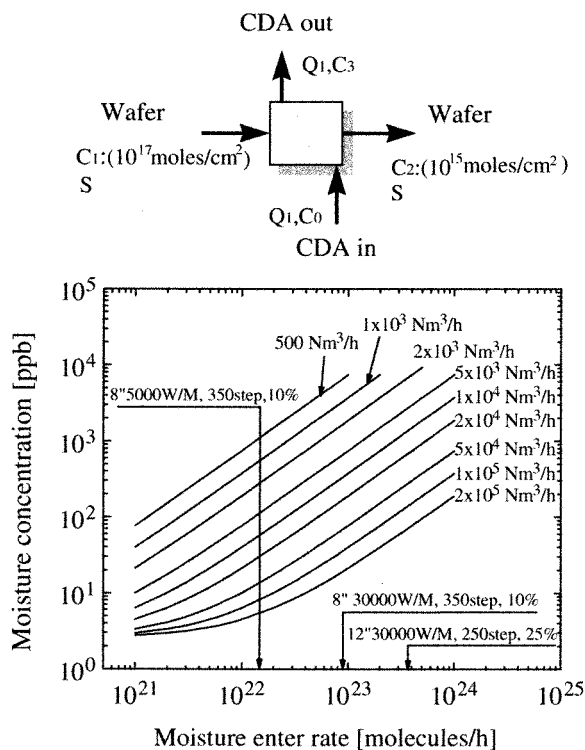


Fig. 10. Relation of H₂O enter rate and H₂O concentration at each purge CDA volume.

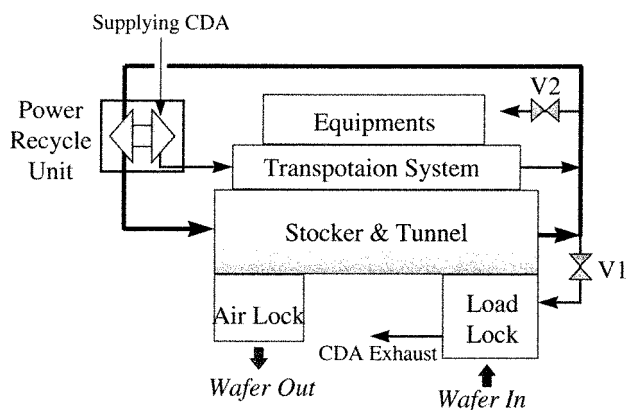


Fig. 11. Concept of wafer supply and store unit (WSSU) and recycle device.

the WSSU. Fig. 11 shows the concept of the WSSU and the recycle device. Because the recycle device is operated using pressure from the replenished CDA of the CDA plant and circulated used CDA, it is possible to reduce the consumption of electric power which is needed for the conventional motor system. Also, as the shaft is included in the outer cover and a distance between it and the shaft bearing is maintained by a gas film, it is possible to realize a leak-free and particle-free structure in this recycle device. The WSSU consists of a load lock room, strage and tunnel, transportation system, and equipment, and each unit is divided by gate valves. First, the wafers are introduced into the load lock room and are exposed to the CDA in order to remove any contamination on the wafer's surface. Since the H₂O levels is very low, the wafers is easy to charge up in the CDA ambient. In order to solve the problem and prevent the adhesion of particles, the soft X-ray irradiation, which is capable of ionizing

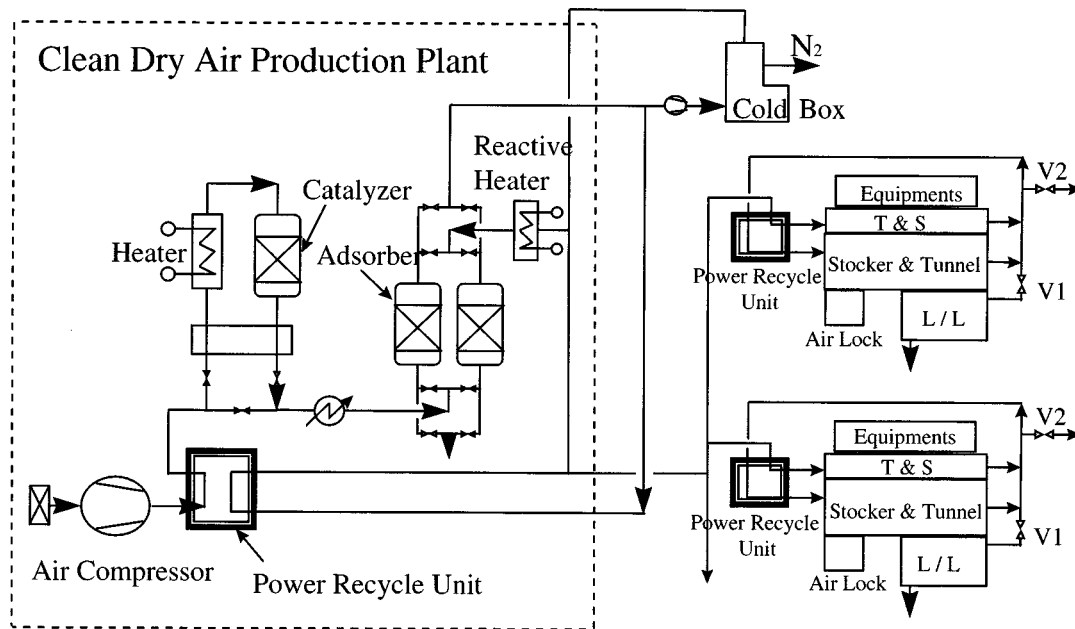


Fig. 12. Schematic of cost effective CDA total system.

air efficiently and removing static electricity without generating any contamination, is effective. Unfortunately, when particles adhered on the wafer, it is desired to be cleaning with advanced spin cleaner [16], which is prepared as equipment in the closed manufacturing system. The contamination on the wafer should be removed until a surface cleanliness is attained, which never influences the succeeding processes, and the wafer, while maintaining such a condition, should be transported to other equipment through the wafer stage and tunnel and the transportation system. Using this system, the exhausted CDA is only used for purging of the load lock room. So, it is possible to reuse a huge volume of CDA that is used in other units of the WSSU. In addition, it is possible to minimize the contamination into the process chamber of equipment during maintenance using V2 in Fig. 11 and to reduce the time of equipment start up.

Fig. 12 is a schematic of the total cost effective CDA system. The multiple WSSU and recycle device should be prepared for one CDA plant and basically set in each bay. It can achieve a simple design and a reliable supply of CDA for a ULSI manufacturing line by construction of a common piping line for replenishing the CDA from the CDA plant. The weighted average cost of the CDA can be estimated at less than 1 yen/Nm³ or 1 cent/Nm³ with a recycle ratio of over 90%.

V. CONCLUSION

We demonstrated that the organic carbon contaminants influenced the J-E characteristics of the gate oxide. In order to implement a high yield and a high efficiency fabrication for ULSI manufacturing lines, it is necessary to realize the construction of a closed manufacturing system, which transfers the wafers between each process without exposing them to the clean room air. For this purpose, we investigated the adsorbing characteristics, which is most important for obtaining CDA both with dynamic simulation and measurement, and confirmed the good agreement between the simulation results and actual measurement.

Based on these results, we built a CDA plant with a 4000 Nm³/h capacity and clarified that the contaminant concentration in the CDA was less than 10 ppb. For economical CDA production and CDA circulation, we have developed a compressor combined turbine with the shaft and shaft bearing separated by a gas film for the CDA plant, and a fan combined turbine with the shaft and shaft bearing separated by a gas film for the circulation device of the used CDA. The economical CDA system, which is applied to these power recycle systems, has been proposed. Using this CDA system, the weighted average cost of the CDA was estimated to be less than 1 yen/Nm³ or 1 cent/Nm³ for the recycle ratio of over 90%. In order to realize a more effective CDA system, it is necessary to establish the technology including the design and construction of piping lines, monitoring technology and other items for the semiconductor manufacturing in the next generation.

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and high-field transport in semiconductors, such as unified theory of space-charge dynamics in negative differential mobility materials, Bloch-oscillation-induced negative mobility and Bloch oscillators, and dynamics in injection layers. In 1972, he moved to Tohoku University, and he is presently a Professor at the New Industry Creation Hatchery Center (NICHe), Tohoku University, Sendai, Japan. He is currently engaged in research on high-performance ULSI, such as ultrahigh-speed ULSI: current overshoot transistor LSI, HBT-LSI, and SOI on metal substrate, base store image sensor (BASIS) and high-speed flat-panel display, and advanced semiconductor process technologies, i.e., ultraclean technologies, such as high-quality oxidation, high-quality metallization due to low-kinetic energy particle bombardment, very low-temperature Si epitaxy particle bombardment, crystallinity control film growth technologies from single crystal, gain size controlled polysilicon and amorphous due to low-kinetic energy particle bombardment, highly selective CVD, highly selective RIE, high quality ion implantation with low temperature annealing capability, etc., based on the new concept supported by newly developed ultraclean gas supply system, ultrahigh-vacuum compatible reaction chambers with self-cleaning function, ultraclean wafer surfaces cleaning technology, etc. His research activities are as follows: 700 original papers and 600 patent applications.

Dr. Ohmi received the Ichimura Award in 1979, the Teshima Award in 1987, the Inoue Harushige Award in 1989, the Ichimura Prizes in Industry-Meritorious Achievement Prize in 1990, the Okouchi Memorial Technology Prize in 1991, the Minister of State for Science and Technology Award for the promotion of invention 1993, the Invention Prize and Fourth International Conference on Soft Computing (IIZKA'96) Best Paper Award in 1996, and the IEICE Achievement Award in 1997. He serves as the President of the Institute of Basic Semiconductor Technology-Development (Ultra Clean Society). He is a member of the Institute of Electronics, Information and Communication Engineers of Japan, the Institute of Electronics of Japan, the Japan Society of Applied Physics, and the ECS.