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Environmental Performance Characterization of Atomic Layer Deposition

Chris Y. Yuan David Dornfeld

Abstract—**Atomic Layer Deposition (ALD) is emerging as a promising nanotechnology for manufacturing dielectrics and insulators on microelectronics devices. Its environmental performance has to be characterized at this early development stage to achieve sustainable manufacturing in the future. In this paper, we report our environmental performance characterization studies on ALD technology through material flow analysis and energy flow analysis. The assessed ALD process is for deposition of Al_2O_3 high-k dielectric films on a 4 inch silicon wafer. The results show that only 50.4% of input Trimethyl Aluminum (TMA) material is turned into Al_2O_3 film, while the other half is transformed into toxic emissions into the environment. Material usage efficiency of water is only 2.03% in current ALD processes. ALD is also featured with intensive energy consumption. For the studied ALD process, a total of 4.09 MJ energy is consumed for deposition of a 30 nm Al_2O_3 film, with averaged energy consumption at 13.6 KJ per cycle.**

Keywords—**Atomic Layer Deposition, Environmental Performance, Energy Flow Analysis, Material Flow Analysis.**

I. INTRODUCTION

Atomic Layer Deposition is a process for depositing highly uniform and conformal thin films by alternating exposures of a surface to vapors of two or more chemical reactants [1]. As a self-limiting and cyclic process, ALD has such superior features as accurate film thickness control, excellent film thickness uniformity, extensive material adaptability, etc. Though still in lab scale study, ALD is emerging as a promising nanotechnology for manufacturing dielectrics and insulators on microelectronics devices. ALD is expected to find its applications on a wide variety of electronic products including CMOS chips, flat panel display, optical filters, etc. in the near future.

While ALD process has to be operated at high temperatures to overcome the high energetic threshold of the chemical reaction, it also requires large amount of precursor material input to maintain sufficient chemical reactions on the deposition surface. As a result, the material consumption and energy use of ALD processes are very intensive, especially when hundreds of cyclic operations have to be performed for a single film manufacturing. The environmental performance of ALD process needs to be scientifically characterized during this early development stage to improve its manufacturability and sustainability for future industrial scale applications.

A wide variety of solid materials can be deposited through ALD process, based on the needs of specific applications [2]. Deposition of Al_2O_3 as high-*k* dielectric films for microelectronic devices is being widely studied as a typical application of ALD technology [3]. The high-*k* dielectric Al_2O_3 film is a promising candidate to replace conventional SiO_2 gate insulators on semiconductor devices. By using ALD technology, the accuracy of Al_2O_3 film thickness can be successfully controlled at 0.1 ± 0.01 nm scale [4], while the roughness of the growing surface can be managed less than 0.3 nm [5].

In this paper, we report our environmental performance characterization studies on the atomic layer deposition of Al_2O_3 thin film on a 4 inch silicon wafer which uses Trimethyl Aluminum as the metal source and water as the oxidant.

$$2Al(CH_3)_3 + 3H_2O = Al_2O_3 + 6CH_4$$

The precursor material TMA, CAS number 75-24-1, is extremely flammable and highly toxic. Exposure to TMA can cause severe skin burns and permanent eye damage. Adopting a Threshold Limit Value at 2 mg/m^3, TMA is regarded five times more toxic than carbon monoxide.

The principal output product of the ALD process is methane which is a major greenhouse gas and can contribute to the global warming significantly when released into the atmosphere. According to IPCC, methane has a global warming potential 25 times higher than carbon dioxide, as computed in a 100 year time horizon [6].

With such toxic materials involved and greenhouse gas generated, wide applications of atomic layer deposition in Al_2O_3 film manufacturing will pose severe risk of damage to both public health and the environment. This project is undertaken for assessing environmental performance of ALD technology and providing decision support for its scale-up in the future. In this study, material flow analysis and energy flow analysis are employed as analysis tools for characterizing the environmental performance of ALD technology.

II. PROCESS CHARACTERIZATION

The ALD system studied is a Cambridge NanoTech Savannah 200 system. The reactor is customized for 4 inch standard wafers, with 150 mm inner diameter and 5mm inner depth. During the experiment, the ALD reactor is heated to 200 °C and maintained constant by a 400 watt McMaster compact disc heater. The inlet pipelines are heated to 70 °C while the outlet pipelines are heated to 160 °C to facilitate the pulsing and pumping of precursor materials.

In the experiment, nitrogen is used as the carrier gas at 10 sccm flow rate to transport precursor vapors to the silicon

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surface and also purge reaction gases from the reactor during each reaction half-cycle. TMA and H_2O are alternatively pulsed into the reactor for 1 second per pulse each, and the purge time for nitrogen is set at 5 seconds in between. An Edwards RV5 Rotary Vane pump is used to vacuum the reactor at beginning and pump out unreacted materials out of the reactor during experiment. In this research, a total of 300 cycles is studied for the ALD process, at an average layer growth rate of 0.1 nm per cycle. The ALD process flow diagram is shown in figure 1 below.

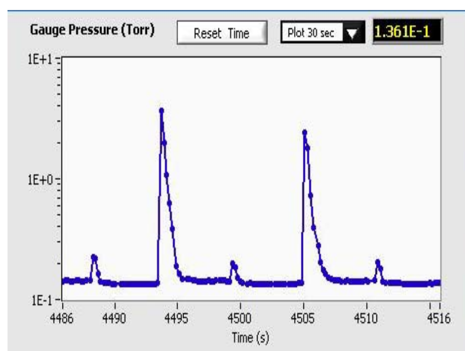


Fig.1 ALD process flow diagram

After the deposition, the Al_2O_3 films are annealed at atmosphere pressure to reduce residual stress and enhance film strength. A McMaster ring heater is used to heat the Al_2O_3 films to 200 °C at a rate of 2 °C per second and held at 200 °C for 2 min. Then the heating is resumed at the same rate and the temperature is increased to 600 °C where the samples are held for 30 seconds. The films are subsequently cooled down to room temperature at a rate of 8 °C per second.

In atomic layer deposition, the film thickness increment and surface roughness profiles depend on the chemical reaction process on the silicon surface, which in turn is decided by the concentration of the reactants and the reaction temperature. ALD is a self-limiting chemical process so Al_2O_3 film deposition rate per cycle is initially proportional to the exposure of precursor materials but saturate to a maximum value after certain reaction sufficiency. Since the unreacted materials are being pumped out of the ALD system, the supplied amount of precursor materials must be carefully decided to ensure a minimum amount of TMA emission.

The ALD deposition of Al_2O_3 films is based on the surface coverage of hydroxyls and the chemical reactions between hydroxyls, TMA and H_2O. The process must be operated at a high temperature to supply sufficient energy to overcome the energetic barrier of surface chemistry, while too high a temperature can cause evaporation of hydroxyls and reduce the number of active sites on the surface which is necessary to form the Al_2O_3 films. It has been experimentally confirmed that the Al_2O_3 film thickness increment per cycle decreases with decreasing temperature at temperatures below 350K and decreases with increasing temperature above 500K [5]. In our experiment, the temperature is set at 200 ^oC to ensure a large Al_2O_3 film thickness increment per cycle.

In the ALD manufacturing of Al_2O_3 dielectric films, the general reaction is split into two half reactions by alternating exposures of the hydroxylated silicon surface to two precursor materials, TMA and H_2O. The formation of Al_2O_3 layer through TMA, OH^- and H_2O interactions is made through a consecutive six-step second-order chemical reaction [5]. Initially, the TMA molecules are physically adsorbed to the hydroxyl surface as the TMA gas molecules approach the solid silicon surface. The physical adsorption mechanism is governed by van der Waals forces between the gas molecules and the silicon bulk surface. Subsequent chemical reactions between OH^-, TMA and H_2O molecules are complicated in which the electronic structures of the gas molecules and the solid surface are rearranged. A general description of the chemisorption process is usually made by the theory of surface states.

The reaction processes are characterized by chemical kinetics and reaction dynamics. According to Arrhenius law, the rate parameter of a chemical reaction is expressed as:

$$k(T) = A \times \exp(-E_a / RT) \quad (1)$$

Where A is the frequency factor; E_a is the activation energy; $R=8.3145\ J\ mol^{-1}\ K^{-1}$; T is absolute temperature.

Activation energy of Al_2O_3 elementary chemical-physical reactions is up to 2.9 eV per molecule, as calculated by using the Transit State Theory [6]. So for Al_2O_3 deposition, the calculated maximum energy demand for activating the chemical reactions is 2.79×10^5 Joules/mole.

The elementary chemical reactions for Al_2O_3 formation are all reversible reactions. In the dynamic reaction process, the equilibrium rate parameter is independent of initial concentrations of reactants and equals to the ratio between the forward rate parameter and the reverse rate parameter. Rate parameters of the Al_2O_3 deposition reactions are shown in table I below.

TABLE I
CHEMICAL REACTION SEQUENCE AND RATE PARAMETERS OF Al_2O_3 DEPOSITION

ALD Process	Chemical Reaction*	Forward Rate Parameter(cm^3/mole/s) *	Reverse Rate Parameter(s^{-1}) *	Equilibrium Rate Parameter (cm^3/mole)
Pulse TMA	$-OH + Al(CH_3)_3 \leftrightarrows -OH\text{-}Al(CH_3)_3$	$0.75\times10^{13}\times\exp(-7000/T)$	0.5×10^{13}	$1.5\times\exp(-7000/T)$
	$-OH\text{-}Al(CH_3)_3 \leftrightarrows -O\text{-}Al(CH_3)_2 + CH_4$	$0.5\times10^{13}\times\exp(-18500/T)$	$0.5\times10^{12}\times\exp(-6000/T)$	$10\times \exp(-12500/T)$
Pulse Water	$-Al(CH_3)_2+H_2O \leftrightarrows -Al(CH_3)_2\text{-}H_2O$	$0.75\times10^{14}\times\exp(-6500/T)$	0.5×10^{14}	$1.5\times\exp(-6500/T)$
	$-Al(CH_3)_2\text{-}H_2O \leftrightarrows -OH\text{-}Al(CH_3) + CH_4$	$10^5\times T^3\times\exp(-18000/T)$	$0.67\times10^{11}\times\exp(-7500/T)$	$1.5\times10^{-6}\times T^3\times \exp(-10500/T)$
	$-Al(CH_3) + H_2O \leftrightarrows -Al(CH_3)\text{-}OH_2$	$0.5\times10^{14}\times\exp(-8500/T)$	0.5×10^{14}	$\exp(-8500/T)$
	$-Al(CH_3)\text{-}OH_2 \leftrightarrows -OH\text{-}Al + CH_4$	$10^5\times T^3\times\exp(-17000/T)$	$0.67\times10^{11}\times\exp(-10500/T)$	$1.5\times10^{-6}\times T^3\times \exp(-6500/T)$

*: adapted from reference [5]

III. MATERIAL FLOW ANALYSIS

In Al_2O_3 deposition processes, input materials are TMA and H_2O, and output materials are Al_2O_3 and CH_4. With the process parameters set for a specific ALD process, the amount of materials supplied, deposited, and emitted can be calculated by applying stoichiometric principles on the ALD chemical reactions. According to mass balance principle, the total amount of materials supplied into the ALD system equals the amount being deposited on the silicon surface and the amount being generated as emissions:

$$\sum M_{input} = \sum M_{deposited} + \sum M_{emission} \quad (2)$$

With an operating temperature set at 200 °C, the TMA and H_2O both are supplied into the ALD reactor in gaseous states. As a result, the amount of input material is calculated through ideal gas law, with process parameters shown in table II.

TABLE II
INPUT PARAMETERS OF PRECUSOR MATERIALS

Process Parameter	$Al(CH_3)_3$	H_2O
Partial pressure (Pa)	12.5	465.8
Process temperature (°C)	200	200
Supplied amount per pulse (moles)	3.1E-07	1.15E-05

In ALD manufacturing, the maximum amount of Al_2O_3 formed on a deposition surface can be decided either theoretically or experimentally. In our research, a lattice model is used for calculating the maximum Al concentration on the 4 inch silicon wafer. In Al_2O_3 molecular structure, the Al-O bond length is 1.8 x10^{-10} m [7]. The Al-O-Al bond angle is 90° in surface distribution [8]. As a result, the Al-Al bond length is 2.545x10^{-10} m. The calculated Al concentration is 25.7 µmole/m^2, which is only 2.6% larger than the maximum limit of Al concentration as experimentally decided on a silicon surface [9].

The 4 inch silicon surface, when fully hydroxylated during the pre-treatment process, can maintain a maximum of 2.08 x10^{-7} moles OH^- on its surface for subsequent chemical reactions. While due to the surface deficiency and incomplete hydroxylation, the surface OH- group concentration is usually 70-80% of the maximum value [10]. In our analysis, we take an average of 75% for the computation.

The Al_2O_3 deposition process involves a series of unstable intermediate materials, at various stages of the reactions. The amounts of materials supplied, formed and emitted from the ALD system for a 300 cycle operation are assessed and the results are shown in figure 2 below.

The material flow analysis results demonstrate that only 50.4% of supplied TMA is turned into Al_2O_3 films through such ALD deposition process. For supplied water, the material usage efficiency is only 2.03%. Methane is formed in three reaction stages during the Al_2O_3 deposition process, with one third formed during TMA pulsing and the other two thirds formed in H_2O pulsing. In ALD manufacturing, pressure and temperature are two process parameters which play important roles in the material flows. In this project, we have assessed the effect of temperature and pressure change on emission generations. On the basis of current operating conditions, we have varied the temperature by 25 °C and the pressure by 20% to study the effect of process parameter change. The amount of TMA and H_2O generated into emissions are analyzed and the results are demonstrated in figure 3 and figure 4.

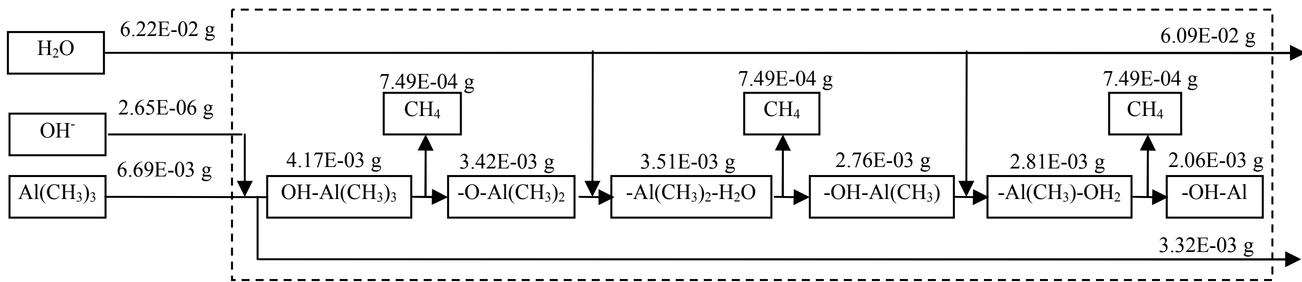
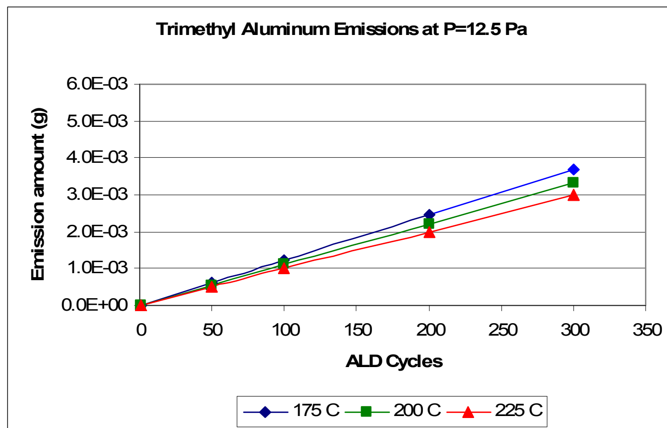
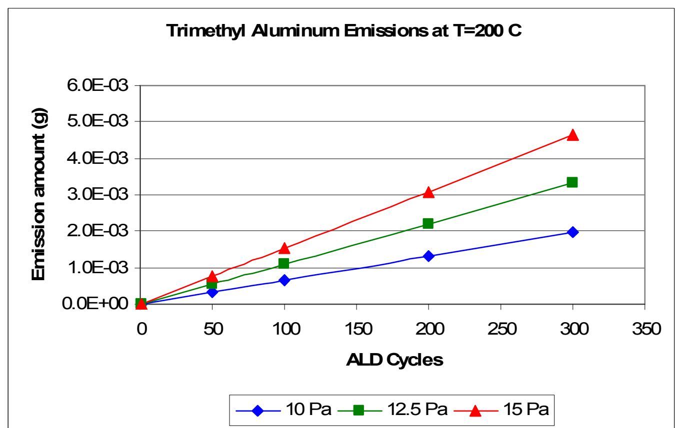


Fig.2 Material flow diagram of 300 cycle ALD process

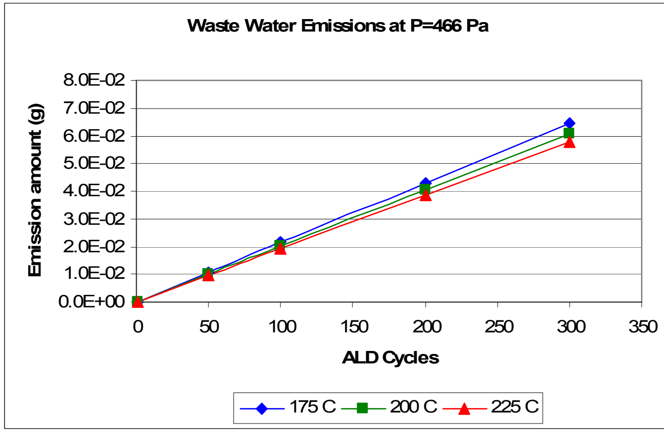


(a)

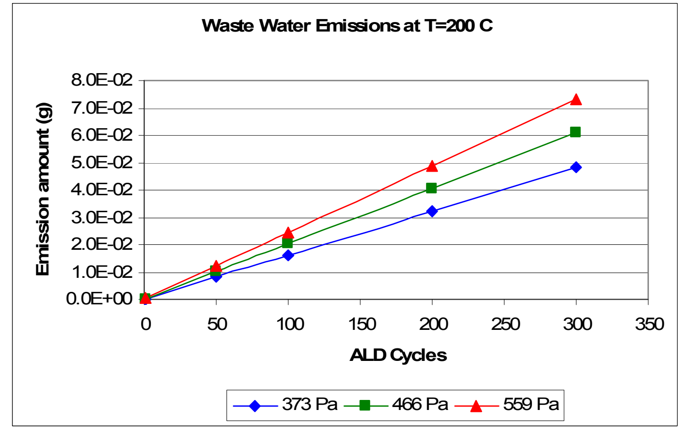


(b)

Fig. 3 TMA emissions at different process parameters



(a)



(b)

Fig. 4 Waste H_2O emissions at different process parameters

It has been found that the amount of emissions can be reduced by increasing process temperature under the same pressure condition. This is attributed to the large amount of energy supplied at a higher temperature which makes the precursor molecules more active in the reaction process and accordingly increases the collision rate of precursor molecules with the silicon surface. However, an increase of the material's partial pressure will supply more materials and consequently generate more emissions from the process.

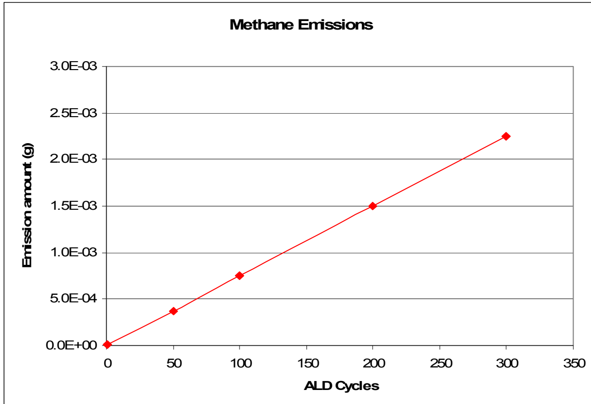


Fig.5 Methane emission in ALD process

In the deposition, methane is generated along with the Al_2O_3 formation on the silicon surface. As a self-limiting process, the amount of methane generated from the ALD process is in direct proportion to the amount of Al_2O_3 formed during the reaction process, but independent of initial concentrations of precursor materials and process parameters. The amount of methane emissions generated in a 300 cycle ALD process is shown in figure 5. Such an emission generation mechanism indicates that any attempt to reduce methane emissions has to be made with a reduction of ALD operating cycles. For a targeted Al_2O_3 layer deposition, the layer thickness is fixed and the amount of methane emission is a fixed value, which can be computed through applying stoichiometric principles on the ALD deposition reactions.

IV. ENERGY FLOW ANALYSIS

During operation, the whole ALD system needs to be heated to maintain the vapor phase of each precursor reactant, and to supply the activation energy, E_a, to the chemical reaction. A typical ALD process, based on the layer thickness requirements, can take a time from 10 min up to 2.5 hours, which consumes a large amount of energy by the ALD system.

Large energy consumption creates both environmental problems and economic barriers for sustainable manufacturing of Al_2O_3 films. In the ALD system, there are a number of processes and components consuming energy through heating, pulsing, pumping, etc. In our analysis, we have categorized the energy consumers into the following five groups and performed detailed analysis on their energy consumptions.

A. System heating

In the ALD operations, the reactor is heated to 200 °C and maintained constant by a 400 watt McMaster compact disc heater, which has a maximum heat output of 400 °C. The inlet pipeline is operated at 70 °C while the outlet pipeline is at 160 °C, each heated by a 144 watt McMaster heat tape which has a maximum heat output of 232 °C. For an accurate assessment of the energy use, we have to decide the actual power usage of heating devices for each specific operating condition.

From the first law of thermodynamics, the energy consumption, Q, of the ALD system can be calculated by:

$$Q = U + W \tag{2}$$

Where U is the internal energy gain of ALD system; W is the dissipated energy by convection and radiation.

The internal energy gain, U, can be calculated by:

$$U = m \times C_g \times \Delta T \tag{3}$$

Where m is mass of the heated component; C_g is specific heat capacity of the material; ΔT is temperature difference.

The dissipated energy, W, can be calculated through:

$$W = q_{convection} \times t + q_{radiation} \times t = h \times A \times \Delta T \times t + \varepsilon \times \sigma \times A \times T^4 \times t \tag{4}$$

Where $q_{convection}$ is the rate of heat dissipation on a surface by convection; $q_{radiation}$ is the rate of heat dissipation on a surface by radiation; t is the heat dissipation time; h is convection coefficient. A is surface area of heat dissipation; ε is emissivity of material; σ is Stefan-Boltzmann constant; T is absolute temperature.

For the ALD system at 200 °C, the heat dissipated by radiation only accounts for 2.8% of total dissipated energy. So we consider it as negligible and only count the heat dissipated

through convection in our analysis. As a result, we get:

$$Q = m\times C_g \times \Delta T + h\times A\times t\times \Delta T = (m\times C_g + h\times A\times t)\Delta T \quad (5)$$

With m, C_g, h, and A all invariable, the total energy consumption, Q, is directly proportional to the temperature interval between the ALD components and ambient air. As calculated, the compact disc heater has a load factor of 0.5 at 200 ^{o}C, and the heat tape has a load factor of 0.24 at 70 ^{o}C and 0.66 at 160 ^{o}C. The ring heater used for annealing of Al_2O_3 films has a load factor of 0.31 at 200 ^{o}C and 0.924 at 600 ^{o}C.

B. Pumping

The vacuum pump used in the ALD system has a power rated at 300 watt. In ALD operations, the pump is always running on a full load between pulsing of precursor materials. As a result, the energy consumption of pumping is the multiplication of the power demand and the operating time.

C. Compressed Air

Compressed air is another energy consumer in ALD system which is used to control the valves for pulsing, purging and pumping. Generation of compressed air requires tremendous energy input [11]. The air compressor used for this study is a Kaeser ASD40S compressor which is rated at 40 HP and 166 feet^3/min flow rate. Take load factor as 1 and motor efficiency as 90%, the energy consumption of compressed air system could be calculated as:

$$Q = \frac{HP\times L\times 746\times 60}{M_E \times 166} = 11983.9 J / ft^3 \quad (6)$$

Where HP is Motor horsepower; L is load factor; M_E is the motor efficiency.

The compressed air is consumed by the diaphragm valves in ALD system which has a 1/8 inch orifice. The energy consumption of compressed air is calculated by assuming a 1% leaking possibility.

D. Computer

The ALD system uses a Thinkpad T61 Notebook computer with Labview program installed for automatic control of the experimental system. Under full load operations, the T61 notebook computer has a power demand between 44.6 and 58.0 watt [12]. Here we take the average of the two values, 51.3 watt, as the normal power demand of the computer.

E. Electronic Control System

A data acquisition and instrument control assembly is used in the ALD system to monitor and control the experimental system, and to collect process data for the Labview program. The total energy consumption of the circuit assembly is the sum of the energy consumed by each component.

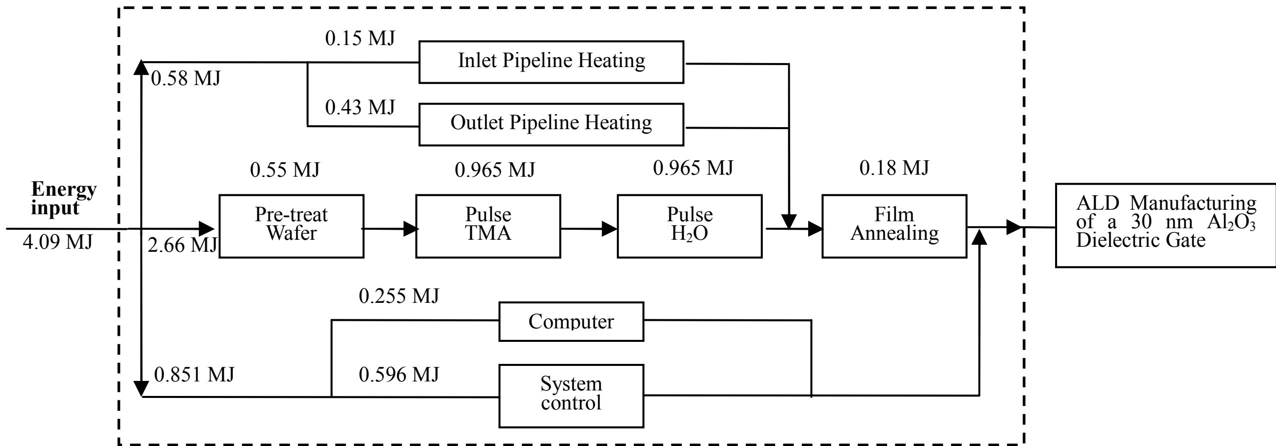


Fig. 6 Energy Flow Diagram of 300 Cycle ALD Processes at 200 ^{o}C

Actual energy use of the ALD system is calculated for each process step and system component. The energy flow of the Al_2O_3 layer deposition process is demonstrated in figure 6. For deposition of a 30 nm Al_2O_3 thin film, the ALD processes consume a total of 4.09 MJ energy, with 14% consumed by pipeline heating, 65% by process operation and 21% by system control and data acquisition. The averaged energy consumption is as high as 13.6 KJ per cycle.

The energy flow analysis demonstrates that the outlet pipeline heating consumes three times as much energy as the inlet pipeline heating, while the energy consumed in pulsing TMA and H_2O are at the same level. The electronics used for system control is the third largest energy consumer in ALD system. A break-down analysis of the energy consumption is shown in figure 7.

As assessed, the energy consumed by system heating accounts for 42.16% of the total energy consumed in ALD manufacturing. Pumping consumes 28.6% of the system energy, while the electronics used for system control and data collection consume 20.79% of total energy.

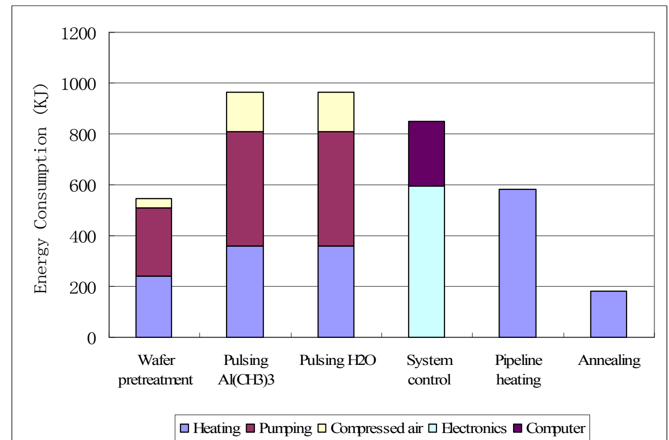


Fig. 7 ALD Process Energy Consumption

The energy consumption of ALD process is mainly decided by the operating temperature of the ALD system. We have performed computational analysis on three different operating temperatures at 175 °C, 200 °C and 225°C, respectively, to assess the effect of temperature change on total energy use. By assuming the operating temperatures of the inlet and outlet pipeline unchanged, the calculated energy consumptions, in the form of KJ per ALD cycle, are shown in table III below. The results demonstrate that at 200 °C temperature level, a reduction of 25 °C in system temperature operation can save only 3~5% of total energy. The saved energy is only from heating, while the energy consumed by compressed air, computer and system control are non-reducible.

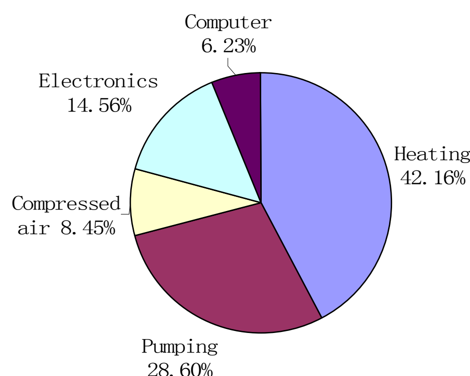


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Fig. 8 ALD Energy Consumption Demography

TABLE III
CYCLIC ENERGY CONSUMPTIONS OF ALD PROCESSES unit: KJ

Temperature	1 cycle	50 cycles	100 cycles	200 cycles	300 cycles
175 °C	708.46	1243.14	1788.74	2879.92	3971.11
200 °C	738.76	1288.14	1848.74	2969.92	4091.11
225 °C	769.06	1333.14	1908.74	3059.92	4211.11

Both the material flow and energy flow analysis results are useful for informing the development of the ALD process for energy efficiency and process optimization. At this early development stage of ALD processes, sustainable manufacturing analysis is especially valuable which can lead to a reduction of the environmental impact and improvement of the productivity of ALD technology in future.

V. CONCLUSIONS

In this paper, we report our environmental performance characterization studies on ALD technology through material flow and energy flow analysis. The modeled process is a 30 nm thick Al_2O_3 film deposition on a 4 inch silicon wafer which uses Trimethyl Aluminum as the metal source and water as the oxidant. The material flow analysis results demonstrate that only 50.4% of input Trimethyl Aluminum material is transformed into Al_2O_3 film with current deposition process, while the material usage efficiency of water in the ALD process is as low as 2.03%. Emission mechanisms of ALD processes are studied at three different temperature and pressure levels to understand the effect of process parameter change. Under the same pressure, ALD process favors a high temperature in the viewpoint of emission generation. In the ALD reaction process, methane is generated along with the Al_2O_3 formation, but any attempt to reduce methane emissions has to be made with a reduction of ALD operating cycles. The energy flow analysis results demonstrate that ALD is featured with intensive energy consumption. For deposition of a 30 nm thick Al_2O_3 film, a total of 4.09 MJ energy is consumed, with 42.16% consumed by system heating, 28.6% by pumping, 20.79% by system control, and 8.45% by compressed air. The average consumed energy is as high as 13.6 KJ per cycle. Although energy consumption of ALD process is governed by the operating temperature, a reduction of 25 °C from current operating temperature can only save 3~5% of total energy consumption.

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