## **Case Study of Micro-Contamination Control**

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## Abstract

Airborne contaminants (gases and particles) pose serious threats to hi-tech industries with the critical dimension of a micro-electronics chip quickly shrinking to nano-scale and the glass substrate of an LCD panel substantially enlarging to 1.9 m by 2.2 m (Generation 7.5). Due to the fast technology evolution, the cleanliness requirement of cleanroom air quality is highly critical. For example, for the 45 nm technology node, ammonia concentration is required to be less than 2.5 part-per-billion (ppb) and concerned particle size is down to 23 nm. To tackle the contamination problem for the rapidly evolving processes, the related information and technique should be shared and learned quickly. In this study, four cases of contamination control are described and they are: (1) corrosive gases (e.g., HBr, HCl, HF, SiF<sub>4</sub>) outgassing from newly dry-etched wafers, (2) gas leaking from an exhaust pipeline in a semiconductor cleanroom, (3) optical lens hazed by fine particles in a thin-film-transistor liquid-crystal-display (TFT-LCD) fab, and (4) re-entrained boron contaminating the glass substrates inside an LCD stocker. By sharing the case scenarios, it is expected that similar micro-contamination problems could be avoided and the product yield be enhanced. Besides, new monitoring and control techniques for micro-contamination might also be developed based on the results found in this study.

*Keywords:* Airborne molecular contamination (AMC); Cleanroom air quality; Micro-contamination.

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## INTRODUCTION

Airborne contaminants render various defects on an electronic device. For example, molecular acid (MA) causes corrosion problem (Higley and Joffe, 1996); molecular base (MB) induces T-topping damage (Ruede *et al.*, 2001); molecular condensable (MC) shifts the deposited film property from Si-N to Si-O (Saga and Hattori, 1997) and hazes the photo lens (Barzaghi *et al.*, 2001); molecular dopant (MD) swaps the p-type and n-type properties (Lebens *et al.*, 1996); ozone (O<sub>3</sub>) lowers the device capacitance (Park *et al.*, 2001). The contaminants in each of the five airborne molecular contamination (AMC) categories are shown in Fig. 1 (Ayre *et al.*, 2005). Besides, a particle larger than 1/3 of the feature size might fail a die (Kitajima and Shiramizu, 1997). To reduce the wafer losses resulting from airborne contamination, the yield enhancement committee of the International Technology Roadmap for Semiconductors (ITRS) annually publishes a guideline to detail the tolerable concentrations for various airborne contaminants in each technology node (ITRS, 2006). For the forthcoming 45 nm process, for instance, ITRS recommends that ammonia concentration be less than 2.5 ppb and critical particle size be 23 nm.



Fig. 1. Schematic of five AMC categories.

In theory, the best strategy to control the contaminants is to locate the emitting origin and to terminate the releasing source. As shown in Fig. 2, in a cleanroom, the contaminants mainly originate from two sources: (a) incoming outdoor air, and (b) internal activities. For the incoming outdoor air, as long as the contaminants are identified, a combination of particle and chemical filter can effectively remove the pollutants at the make-up air unit (MAU) (Kanzawa and Kitano, 1995; Muller, 2004; Yeh *et al.*, 2004).

However, for the contaminants releasing from internal activities, the contaminating chemicals could easily damage the electronic device (e.g., wafer, glass substrate) due to proximity and localized high concentration. Therefore, huge product losses generally result from internally generated contaminants. Based on the literature, the internal contaminants could come from several sources: MA and particles from preventive maintenance (PM) activities (Li *et al.*, 2005), MB from neighboring chemical-mechanical-polishing (CMP) and photo-resist stripper areas (Demandante *et al.*, 2000), MC from plastic utilities (e.g., wafer cassette, gloves) (Saga and Hattori, 1996; Schnabel *et al.*, 2001), MD from glass fiber filter (Stevie *et al.*, 1991) and filter sealant (Lebens *et al.*, 1996), O<sub>3</sub> from a O<sub>3</sub>-water mixing chamber (Li *et al.*, 2003), particles from persons, garments and wipers (Kitajima and Shiramizu, 1997; Bhattacharjee and Paley, 1998; Ensor *et al.*, 2001). For the aforementioned cases, applying appropriate sampling and analytical techniques are crucial to accurately spot and then control the contamination sources. In this study, beside the already referenced internal sources, four additional AMC tracking and controlling cases will be presented.



Fig. 2. Schematic of air flow circulation and OP-FTIRs monitoring lines in a cleanroom.

## MATERIALS AND METHODS

To determine the gas compositions and concentrations outgassing from the newly dry-etched wafers, as shown in Fig. 3, a compressed dry air (CDA) flow of 3 L/min, regulated by a mass flow controller (MFC), was used to continuously purge a batch of wafers (no. = 25). The purging  $N_2$  flow was then directed to the 10 m gas cell (EA-2 L/10 m, Gemini Scientific Instruments,

Calif., U.S.A.) of a movable extractive fourier transform infrared (FTIR) spectrometer (Work IR-104, ABB Bomem, Quebec, Canada). The contaminants in the purging  $N_2$  were instantly identified and quantified by the FTIR because of the fingerprint nature of the IR absorbance spectra for each gas and the advances of computer calculation capability.



Fig. 3. Experimental set-up for wafer outgassing tests.



Fig. 4. Major components of the gas-leaking detection system (GLDS).

For the leaking spot locating, the gas-leaking-detection-system (GLDS) (Li *et al.*, 2007) shown in Fig. 4 was applied to pinpoint the emitting source among thousands of gas pipelines. The GLDS is composed of two open-path Fourier transform infrared (OP-FTIR) spectrometers, a gas composition database and a diagnosis software to help determine the emitting origin. As Fig. 2 shows, one OP-FTIR is installed at the make-up air unit (MAU) to continuously monitor the incoming pollutants; the other OP-FTIR is situated at the recirculation air unit (RAU) to sense airborne contaminants inside the cleanroom. Upon detection of the contaminants at the RAU, as shown in Fig. 4, the diagnosis software will compare the gas compositions measured by two OP-FTIRs to determine if the pollutant originates inside the cleanroom. If yes, the software automatically searches through the database to match the most probable leaking source and then sends a warning signal to the emergency response center (ERC) or to a responsible engineer for a follow-up mitigation action.

To realize the chemical compositions that caused the optical lens hazing, a personal porous-metal denuder (Tsai *et al.*, 2001) with a sampling flow rate of 2 L/min was applied to sample the air inside the optical equipment (193 nm) for 2 hours. After the sampling, the used, chemical-impregnated porous-metal disks were carefully sent back to the laboratory, extracted by distilled, deionized water (DDW) and then analyzed by an ion chromatography (IC) (ICS-90, Dionex, Calif., U.S.A.).

For the boron contamination investigation, the air samples inside and outside three suspected stockers were simultaneously taken by using KOH-impregnated cellulose filters (Fogg *et al.*, 1983) for 48 hours at a sampling flow rate of 10 L/min. The used 47 mm cellulose filters were extracted by a 10 mL DDW solution added with 2 M HCl (~100  $\mu$ L) and analyzed by an inductively coupled plasma/mass spectrometer (ICP/MS) (7500s, Agilent Technologies, U.S.A.). After the air sample analysis, it was found that only in one LCD stocker the inside boron concentration was higher than the outside one. It means that there is a contaminating source inside this stocker. To trace the contamination origin, the stainless-steel frames of this stocker were wiped by the KOH-soaked Teflon filters. The wipe samples were then extracted by DDW-HCl solution and analyzed by an inductively coupled plasma/mass spectrometer (ICP/MS).

### **RESULTS AND DISCUSSIONS**

#### (a) MA outgassing from newly dry-etched wafers

Fig. 5 shows the releasing gas concentration profiles from a batch of newly dry-etched wafers. High concentrations of acid gases (SiF<sub>4</sub>, HBr, HCl, HF) continuously release from wafers for more than 30 minutes. Without appropriate control measures, the MAs would emit into the cleanroom to contaminate near-by wafers and to cause corrosion on equipment surfaces. In four MAs, SiF<sub>4</sub>, a dry-etching product (Si + F  $\rightarrow$  SiF<sub>4</sub>), constitutes the major emissions with a peak concentration of 48 part-per-million (ppm); HBr and HCl are residual process gases and HF is the by-product of SiF<sub>4</sub> hydrolysis.

To prevent the MAs from spreading around the cleanroom, a well-vented storage cabinet shown in Fig. 6 was constructed. The dimensions of the storage cabinet are 1.4 m by 1.5 m by 0.75 m and the internal circulation air flow rate equal to 4200 L/min. Based on the findings of this study, the newly dry-etched wafers were temporarily arranged inside the cabinet for 20-60 minutes to diffuse out the MAs before being sent to the next process equipment. With this arrangement, the effect of throughput losses can be minimized.



**Fig. 5.** Concentration variations of acid gases outgassing from newly dry-etched wafers (no. = 25).



**Fig. 6.** Photo of a wafer storage cabinet for capturing the outgassing acid gases.(Dimensions: 1.4 m by 1.5 m by 0.75 m, circulation air flow rate = 4200 L/min).

By effectively controlling the acid gas emissions, the contaminating particle counts on the wafers were significantly reduced (Li *et al.*, 2003). This is because the MAs react with the MBs (e.g., NH<sub>3</sub>, amines) present in the cleanroom to form fine salt particles (Kanzawa and Kitano, 1995). Due to proximity, the in-situ generated fine particles could easily deposit on the wafer surface and cause device defects. However, when the MAs were drawn away from the wafer surface, the number of chemically-generated fine salt particles would be greatly reduced, which

resulted in the decrease of particle counts on the wafer.

#### (b) Gas leaking from an exhaust pipeline

Although no odor complaint nor gas-sensor alarming was issued, the GLDS shown in Fig. 4 continuously sent a gas leaking warning to the responsible engineer. As Fig. 7 shows, there is an SF<sub>6</sub> emission source inside the fab because the measured SF<sub>6</sub> concentrations at the RAU (inside the fab) are significantly higher than those at the MAU (outside the fab). With the assistance of the computer software and the database, the suspected leaking source was identified as the exhaust pipelines of poly-etchers. By comparing the measured on-off times of SF<sub>6</sub> (by OP-FTIR) with the operation records of poly-etchers and by applying a mobile extractive FTIR along the exhaust pipelines, the leaking spot was then determined to be a cracking duct behind a poly-etcher pump. By applying the GLDS, the gas leaking can be sensed in a very early stage and the losses caused by the gas leakage can also be greatly reduced.



**Fig. 7.** Measured SF6 concentrations at the RAU (diamond symbols) and at the MAU (square symbols) by two OP-FTIRs.

#### (c) Optical lens hazed by fine particles in a TFT-LCD fab

In a TFT-LCD manufacturing plant, the production lines were halted because the optical lens of an exposure equipment were blurred. The air quality inside the exposure tool was then evaluated by using a personal porous-metal denuder. After analyzing the sampled porous-metal disks by an IC, it was found that the hazing chemical was composed of ammonium-nitrate (NH<sub>4</sub>NO<sub>3</sub>). The results were very similar to those found by Johnstone *et al.* (2004) in a semiconductor manufacturing plant. It indicated that the contaminant causing the optical lens hazing might be the fine salt particles generated from the reaction of NH<sub>3</sub> and HNO<sub>3</sub>.

To find the emission source, an extractive FTIR was used to measure the air quality around a

suspected photo-resist stripper during its preventive maintenance (PM) activity. As Fig. 8 shows, high concentration of NH<sub>3</sub> was continuously emitted into the cleanroom during the PM of the photo-resist stripper, which used mono-ethanol-amine (MEA) as its stripping chemical. The three photos shown on the top of Fig. 8 represent NH<sub>3</sub> emissions from used wiping cloth (left one), from the gap around the chamber door rim (middle one) and from the activities of cleaning the chamber inner surfaces (right one). To control the NH<sub>3</sub> emission, air-tight chamber lids were applied around the rims of the process tool and distilled-water flush steps were added to reduce residual chemicals condensed on the internal walls. For the potential HNO<sub>3</sub> (or similar chemical) source, unfortunately, it was not found.



**Fig. 8.** Fugitive NH<sub>3</sub> emissions in the PM activity of an LCD stripper which uses mono-ethanol-amine (MEA) as its stripping chemical.

#### (d) Boron cross-contaminating the glass substrates inside an LCD stocker

Fig. 9 shows the measured boron quantity on each frame surface of an LCD stocker by randomly wiping an area of 0.1 m-by-0.1 m with KOH-impregnated filters. The results clearly demonstrate that the stainless-steel frames, which are used to support the LCD glass substrates in a stocker, are a boron emission source. When the glass substrates were stored inside the stocker to wait for the next process equipment, they were contaminated by the boron emitting from the surfaces of the stainless-steel frames. The boron on the frames could originally come from the fugitive emissions of the glass substrates. With long-time (~1 year) accumulation, the boron quantity on the frames became significant and detrimental although the boron emission from each glass substrate was insignificant. To resolve the boron contamination problem inside the LCD stocker, it was recommended that the stainless-steel frames be cleaned periodically (~once per month) with KOH-DDW solution to avoid significant boron accumulation.



Fig. 9. Accumulated boron quantity on each support frame of an LCD stocker.

## **CONCLUSIONS**

Locating the contamination source and then mitigating the releasing origin proves to be an effective way to tackle the airborne molecular contamination (AMC) problem. In this study, a mobile extractive FTIR showed its capability to successfully identify and quantify the point contaminating sources; the GLDS, composed of two open-path FTIRs, accurately and quickly pinpointed a leaking spot among thousands of gas pipelines. Besides, a personal porous metal denuder demonstrated to be a useful tool to sample trace amount of MAs, MBs and particles; chemical-impregnated filters appeared useful for collecting the surface contaminants. After finding the contamination sources, based on on-site situations, different mitigation measures were then adopted. The results and experiences found in this study would be useful and beneficial for solving various contamination problems, which originates from dynamic hi-tech manufacturing processes and their demanding requirement on cleanroom air quality.

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