Aerosol and Air Quality Research, 12: 104–112, 2012
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ISSN: 1680-8584 print / 2071-1409 online
doi: 10.4209/aaqr.2011.07.0107

Metal Corrosion of Al-Si-Cu Pattern Wafer Due to Chloride Ion Contaminants

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ABSTRACT

Chloride ions (Cl^-) induce metal corrosion of integrated circuits and cause wafer scrap events in the clean room environment. In this study, Al-Si-Cu pattern wafers were designed to monitor critical Cl^- concentration which leads to metal corrosion effects in a simulated airborne molecular contamination (AMC) mini-environment. The simulated Cl^- contamination was generated by HCl permeation tube in a mini-environment; meanwhile, the HCl concentrations in the mini-environment and the real fab were monitored by a real-time Ion Mobility Spectrometry (IMS) instrument. The exposed Al-1%Si-0.5%Cu pattern wafers were analyzed by the surface scanner, the optical microscope (OM), scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) analyzer. The critical HCl concentration where metal corrosion defects could be occurred and found on the wafer surface was around 2.9–4.7 ppbv for simulated mini-environment exposure within 1 hour, and 2.9–4.2 ppbv for real fab ambient air exposure within only 10 minutes. The results indicated that the Al-Si-Cu pattern wafer with TiW barrier layer is more sensitive than the Al-Cu pattern wafer with TiN barrier layer. It also proved that the permeation tube method can be served as a stable Cl^- simulated source, which can control the simulated mini-environment to be within ~4% error in ppbv levels.

Keywords: Airborne molecular contaminants (AMCs); Chloride contamination; Cleanroom micro-contamination; HCl; Pattern wafer; Semiconductor device.

INTRODUCTION

Understanding the effect of airborne molecular contaminants (AMCs) in cleanroom environment is an increasing concern as the feature size continues to shrink in the semiconductor industry. These contaminants can induce a variety of problems which are important in all types of semiconductor process. The air quality of a cleanroom environment must be tightly controlled to avoid yield reduction and performance deterioration of semiconductor devices (Kinkead *et al.*, 1995; Kitajima and Shiramizu, 1997; Yeh *et al.*, 2004; Chien *et al.*, 2007). The monitoring and control of AMCs are major challenges for the present and future production of integrated circuit.

The AMCs cover a wide range of contaminants, including volatile organic compounds, organometallic and inorganic species. The SEMI (Semiconductor Equipment and Materials International) standard classified the AMCs into acids, bases condensables, dopants and non-classified species (SEMI, 1996). The International Technology Roadmap for Semiconductors (ITRS) has made efforts on tightening specification for AMCs. For example, the requirement of Cl^- concentration in the wafer environment should be below 10 pptM in 2007 (SIA, 2006), which corresponded to below 0.242 ppbv of gas phase Cl^- concentration at 22°C.

AMC could be introduced from many sources including materials used in construction, compounds from cleanroom process, and outgassing from the local environment (Li *et al.*, 2007). Large amounts of inorganic acids are used in semiconductor processes which include the cleaning process (H_2SO_4, HCl, HNO_3, HF, etc.), etching process (H_2SO_4, HNO_3, HCl, HF, Cl_2, HBr, F_2, BCl_3, etc.) and chemical vaporization process (Trichlorosilane) (Lin *et al.*, 2010; Soo *et al.*, 2011). Acid gases of HF, HCl, HNO_2, HNO_3 and SO_x are known to create corrosion problems throughout the fab (Juozaitis *et al.*, 2007; Lin *et al.*, 2010). Ionic contamination, especially Cl^- contaminants, in microelectronic circuit can have a detrimental effect on device reliability and yield (Hayasaka *et al.*, 1991; Ishida *et al.*, 1992).

There are three possible sources of external Cl^- contamination in the clean room: (1) metal etching process chamber preventive maintenance (PM), (2) exhaust and pump line leakage, and (3) outdoor air (Ito *et al.*, 2001; Zhu, 2002; Chien *et al.*, 2007; Huang *et al.*, 2009; Shih *et al.*, 2009). In a semiconductor fab, in-situ Cl^- contamination has been well controlled by improving process parameters, but limited researches have been done on Cl^- contamination due to chamber PM or exhaust leakage, where the HCl

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concentration could possibly reach as high as tens of ppmv (Li *et al*., 2005).

It is evident that the Cl^- ionic contaminants deposit on wafers within the device-fabrication region and cause Al-Cu corrosion and metal cross contamination (Wu *et al*., 2010). The Cl^- contaminants could lead to harmful precipitates and result in irreversible damage to process wafers (Hayasaka *et al*., 1991; Ishida *et al*., 1992). One of the significant factors leading to metal corrosion is exposure time after metal film formation (Chapman and Nowak, 1980; Lawrence and McPherson, 1991; Morilla *et al*., 2001). In addition, the mass of metal corrosion defects is positively correlated with the Cl^- exposure concentration (Fan and McPherson, 1988). Higley and Joffe (1996) proposed that when HCl concentration was controlled under 9 ppb, then metal corrosion wouldn't occur. But our prior study (Wu *et al.*, 2010) showed that at Cl^- concentration of much less than 9 ppbv the metal corrosion defect was clearly observed.

In our prior study, the designed pattern wafer is a cost-effective monitoring method for the monitoring of metal corrosion (Wu *et al*., 2010). However the stability of the simulated AMC concentration via HCl wiper was not good enough, which still had about 15% deviation among the sampled values. That is because the design purpose for clean wiper being able to capture and hold contaminants instead of releasing contaminates (Shiue *et al*., 2011). In this study, a permeation tube method is proposed to simulate Cl^- concentrations for future reducing the error range of the simulated Cl^- contamination environment in ppbv levels.

In addition, the pattern wafer design that employs Al-Si-Cu pattern wafer with TiW barrier layer as the witness wafer is proposed to understand its sensitivity to the gaseous HCl exposure. This study might be the first attempt to use pattern wafer as evidence collector in a real fab's exposure tests, as well as announcing the critical HCl concentration that causes the metal corrosion effect on a wafer surface.

EXPERIMENTAL METHODS

Pattern Wafer Design

The effect of in-situ Cl^- residue on the Al-Si-Cu pattern wafers after the metal etching process is studied. Compared with Al-Cu pattern wafer material, the electron migration velocity is better than that of Al-Si-Cu pattern wafer material. Fig. 1 shows the schematic diagram of a 150 mm pattern wafer, it was prepared by the following procedure:

(1) Wafer cleanness by wafer cleaner and surface cleaner.
(2) SiO_2 layer formed by Tetraethyl Orthosilicate based Plasma Enhanced Chemical Vapor Deposition process.
(3) TiW and Al-1%Si-0.5%Cu layer formed by Metal Sputtering process.
(4) Photo resist (PR) formed by PR coater and developer (Recipe: 0.5 μm Logical Metal-1 Lithography).
(5) Al-1%Si-0.5%Cu metal circuit with 0.7 μm width formed by Metal Etching process.
(6) Complete pattern wafer after PR stripping.

Cl^- Exposure in Mini-environment

Mini-environment Experimental Design

A mini-environment was used as the simulated contamination environment and the witness wafer was placed in the mini-environment for exposure experiments (Bai *et al*., 2002; Kang *et al*., 2006; Ku *et al*., 2010). Fig. 2 shows the schematic diagram of the wafer exposure test system. The mini-environment chamber was made of Acrylics (L: 30 cm, W: 30 cm, H: 30 cm). There were three gas streams flowing into the mini-environment chamber: HCl gas source, moisture, and dilution air. The above gas sources use clean and dry air as the carrier gas. The simulated contamination source was induced by a HCl permeation tube, while the humidity control was made by the passing clean and dry air into an impinger containing de-ionized water. All the gas flow rates were controlled by mass flow controllers (Ares, FC-780, USA). The three gas streams were mixed in a gas manifold and then it entered the mini-environment chamber. During the test, a HCl real time monitor (IMS, Ion Mobility Spectrometer, Model 55 series, Molecular Analytics, USA), and a humidity sensor (HM70, Vaisala, USA) were employed. The mini-environment chamber was controlled to have a target HCl concentration of 2, 4, 6, 8 and 10 ppbv and a 45 ± 3% relative humidity at 22 ± 1°C during the 1 hour exposure test.

Exposure Test

Before the exposure test, the pattern wafers were pre-scanned by a surface scanner (2130, KLA-Tencor Corporation, USA) to check if there was any observable corrosion defect. And the surface scanner pre-scan data showed that no corrosion defects were found under normal conditions. The pattern wafers were then placed in the mini-environment chamber shown previously in Fig. 2. After the simulated 1 hour exposure tests under a target HCl concentration of 2, 4, 6,

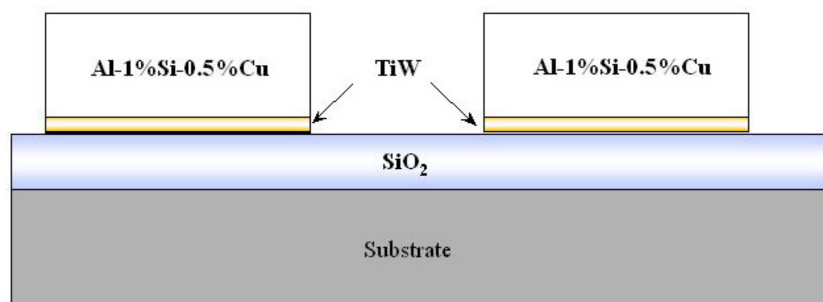


Fig. 1. The process flow of a 150 mm pattern wafer design.

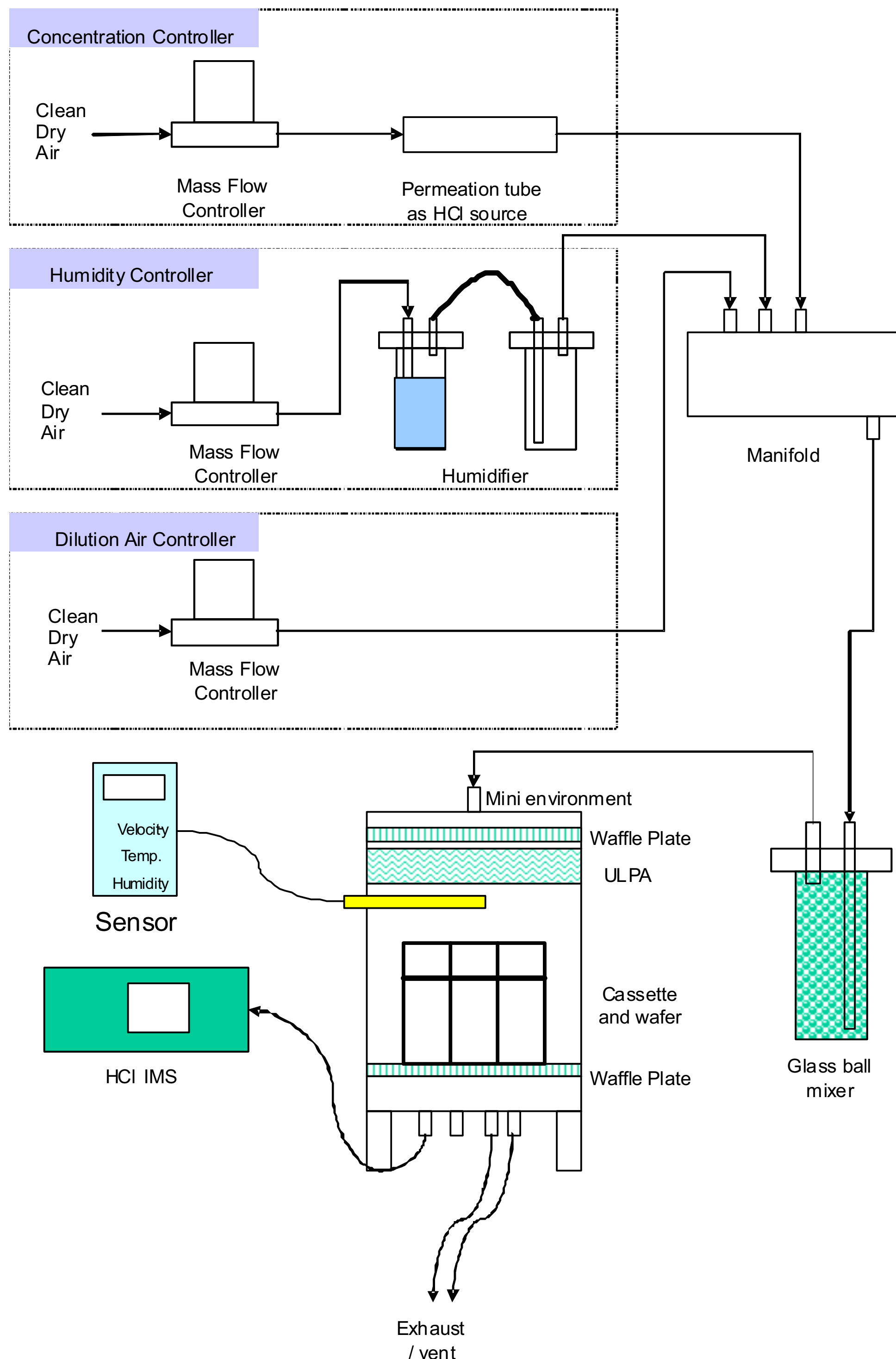


Fig. 2. The schematic diagram of pattern wafer AMC exposure test in a mini-environment Cl^- contamination control system.

8 and 10 ppbv, the exposed pattern wafers were characterized via surface scanner, optical microscope (OM, Optistation, Nikon, Japan) and scanning electron microscope/energy dispersive spectrometer (SEM/EDS, 4700, Hitachi, Japan) analyses to evaluate the metal corrosion defect.

Cl^- Exposure in the Real Fab

Monitoring of HCl Concentration

Real fab tests were performed in an actual clean room environment. The study cleanroom was located in a Taiwan semiconductor 150 mm fab, it was consisted of different process areas including photo areas, diffusion areas, a thin film area, an implant area and etch areas of class 100. A total of 23 sampling points were chosen and the measurement height was 1.5 m for each sampling point. The Cl^- concentration was on-line monitored by the multi-sampling

real-time monitor (IMS, Ion Mobility Spectrometer, 815910-20-100b, Saes, USA) for 6 months. The IMS method has been proven to be a useful technique for determining trace ionic concentrations with detailed description of the sampling and analysis procedure referred to our prior study (Lin *et al*., 2008; Lin *et al*., 2009). The real time monitoring instrument of IMS took one data point for every 5 minutes located in each process area. The STATISTICA software (Version 7, STATSOFT, USA) was used for obtaining contour plots of HCl concentration distribution.

Values of the relative humidity in all the sampling areas were well within 45 ± 3% and temperature were 22 ± 1°C. Hence the humidity effect on the metal corrosion should be the same throughout the sampling areas. The average value of laminar flow velocities in the cleanroom was 0.45 m/s with less than 10% variations.

Pattern Wafer Exposure in the Real Fab

The pattern wafer was placed in the etch process area of a real fab. The sampling frequency was once per week and the sampling time was 12 hours for each test. An optical microscope was used to evaluate the defect. The inspection frequency by the optical microscope was once per hour for the first 2 hours, and then for the remaining 10 hours it was once per 2 hours. If metal corrosion was observed by the optical microscope, it would be further confirmed by the surface scanner and EDS analysis. In the experimental period, the multi-sampling real-time HCl IMS was employed which provided the real-time HCl concentration during the 12 hours exposure.

RESULTS AND DISCUSSION

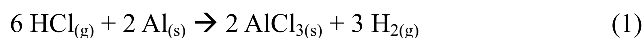
Simulated Tests in the Mini-environment

Monitoring of Cl⁻ Micro-contamination

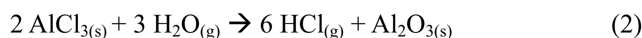
Fig. 3 shows the time variation of the HCl concentrations in the mini-environment and Table 1 lists their average values, standard deviation and error range. One can see that the HCl concentration of level A which used 2.0 ppbv as a target concentration ranged from 1.8 to 2.5 ppbv, with the average and standard deviation of 2.4 ± 0.1 ppbv (error range 4.2% by ignoring the first sampling data). One can observe from Fig. 3 that the first HCl concentration is always smaller than the remaining sampled concentrations of all levels A–B, which it could be took some time to reach equilibrium concentration in the simulated mini-environment. Then one can see that during the one hour sampling time, the permeation tube method provided a fairly good HCl source for AMC study where the HCl concentration could be well controlled to be around ~4% error range.

The Influence of Cl^- Concentration on TiW Pattern Wafer

The metal layer composition of the TiW pattern wafer is Al-1%Si-0.5%Cu. Thus the metal corrosion effect could be caused by HCl induced metal corrosion or the local cell effect induced metal corrosion. The contact of substrate Si is a key factor for the local cell induced metal corrosion instead of the HCl induced metal corrosion. The HCl corrosion can be caused by both metals of Al and Cu. And since Al contributes to 98.5% of the major composition of the pattern wafer, the chemical reaction mechanism of chloride-induced corrosion study is proposed via the reaction of HCl with Al as (Fan and McPherson, 1988; Hayasaka *et al*., 1991; Ishida *et al*., 1992):



$$6\ HCl_{(g)} + 2\ Al_{(s)} \rightarrow 2\ AlCl_{3(s)} + 3\ H_{2(g)} \quad (1)$$



$$2\ AlCl_{3(s)} + 3\ H_2O_{(g)} \rightarrow 6\ HCl_{(g)} + Al_2O_{3(s)} \quad (2)$$

Based on the chemical reactions, Al metal line reacts with HCl and H_2O to produce Al_2O_3 as the reaction product and results in metal corrosion defects.

Fig. 4 shows the surface scanner pictures of defect density on pattern wafers for different levels of Cl^- exposure. No defects were found at the HCl exposure concentration of 1.8–2.5 ppbv (level A). And further analysis shows that

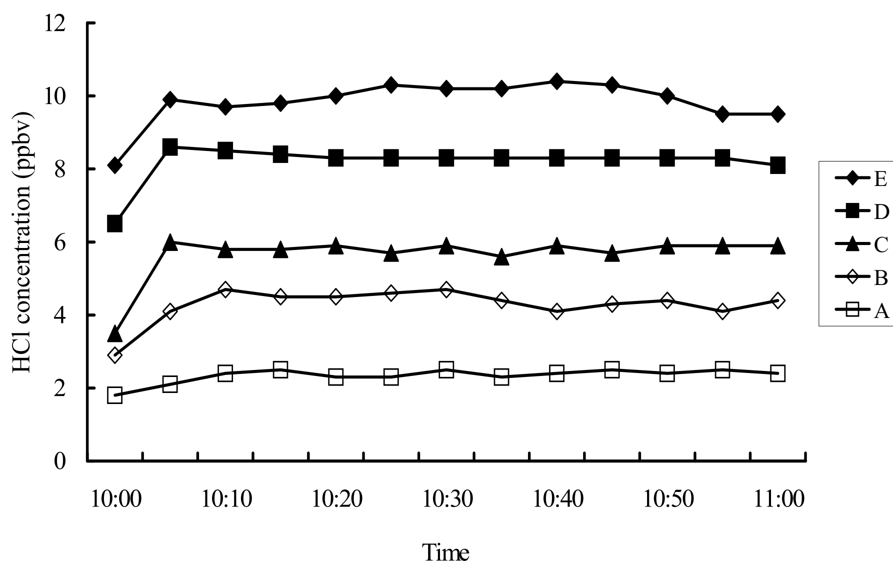


Fig. 3. Measured data during levels A-E of HCl exposure in the mini-environment experiment.

Table 1. HCl concentration in the mini-environment as measured by the IMS (Unit: ppbv).

Level \ Measured Concentration	Ave. ± SD (Error range)[a]	Range
A (target: 2 ppbv)	2.4 ± 0.1 (4.2%)	1.8–2.5
B (target: 4 ppbv)	4.4 ± 0.2 (4.5%)	2.9–4.7
C (target: 6 ppbv)	5.8 ± 0.1 (1.7%)	3.5–6.0
D (target: 8 ppbv)	8.3 ± 0.1 (1.2%)	6.5–8.6
E (target: 10 ppbv)	10.0 ± 0.3 (3.0%)	8.1–10.4

[a] Note: Standard deviation was calculated by ignoring the first sampling data at each level.

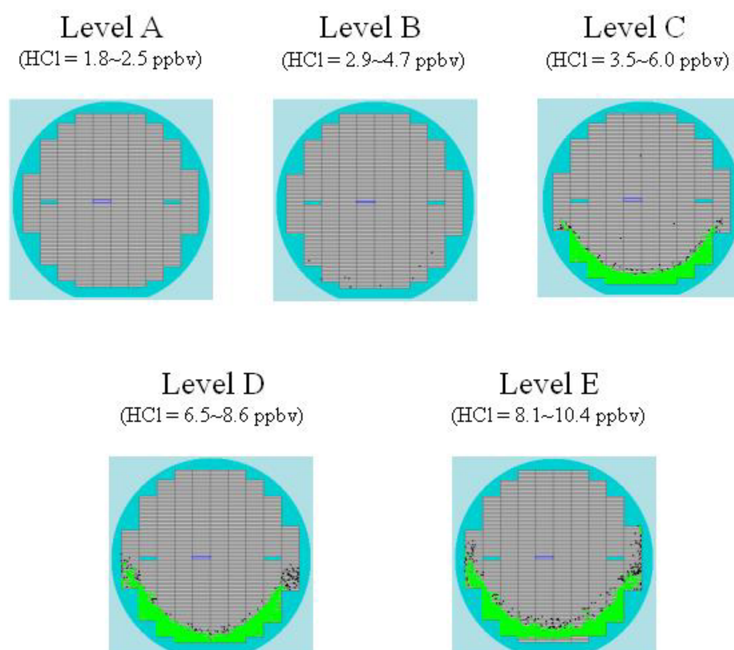


Fig. 4. Surface scanner analysis for the mini-environment exposure experiment.

there was no Cl element found on pattern wafer particles of level A by EDS analysis, and no metal corrosion was observed by the SEM image. Thus, under the HCl exposure concentration around 1.8–2.5 ppbv, the possibility of metal corrosion is very low during one hour exposure time. But when the pattern wafer was exposed to level B–E where higher HCl concentrations > 2.9 ppbv were measured, metal corrosion defects were observed.

More severe metal corrosion defects were found at higher Cl^- concentration exposure and metal corrosion defects were easily observed by an optical microscope (OM) under the exposure condition of level C (3.5–6.0 ppbv Cl^- concentration) or above. The distribution of metal corrosion defects were clearly observed on the edge of the pattern wafers around the flat side. One possible cause was that the wafer edge does not have a good uniformity as compared to the wafer center, and this increased the deposition rate of HCl molecules corrosion capability. The other cause might be due to that the flat side was nearby the cassette opening which also led to higher HCl exposure possibility.

Result on the SEM/EDS analysis of pattern wafer under level B exposure is shown in Fig. 5. One can see that metal corrosion defects were clearly observed by the SEM image and the Cl element was found by the EDS analysis. Similar observations on the metal corrosion defects were observed and Cl element was detected in all pattern wafers exposed at levels C to E. In order to prevent metal corrosion defects, the HCl concentration should be controlled under 2.5 ppbv for Al-1%Si-0.5%Cu pattern wafer with TiW barrier layer.

Real Fab Tests on the Metal Corrosion

Monitoring of Cl^- Micro-contamination in the Cleanroom

The HCl concentration distribution measured by the IMS monitor were obtained and the sampling duration was over 6 months, and the contour plot results from the STATISTICA software are shown in Fig. 6. One can see that measurement data at the two etch areas show higher HCl concentrations than other process areas. The process gases used by metal etchers include Cl_2, HCl and BCl_3. Dry etch process induces the process gases being ionized to Cl^- with residue process gas. The releasing of HCl gas from either the PM of metal etchers or the pump line outgassing will cause the Cl^- contamination in the etch process area of the clean room.

At the etch area, the highest average concentration of HCl during the sampling period was 1.16 ppbv and the lowest average concentration of HCl was 0.65 ppbv. The gaseous concentration of HCl can be diffused from etch

areas to adjoining process areas. The ambient air recirculation is from the etch tools downstream flowing to sub-Fab, it then enters the side-wall return plenum, and goes upstream to the mechanical chamber, and then downstream to adjoining process areas. The other source of HCl was from the chemical usage in the adjoining process areas including HCl chemical. And insufficient exhaust ventilation of wet cleaner in diffusion area could also induce HCl emission. The wet chemical station in diffusion area tended to have higher levels of HCl contaminant which is probably due to the

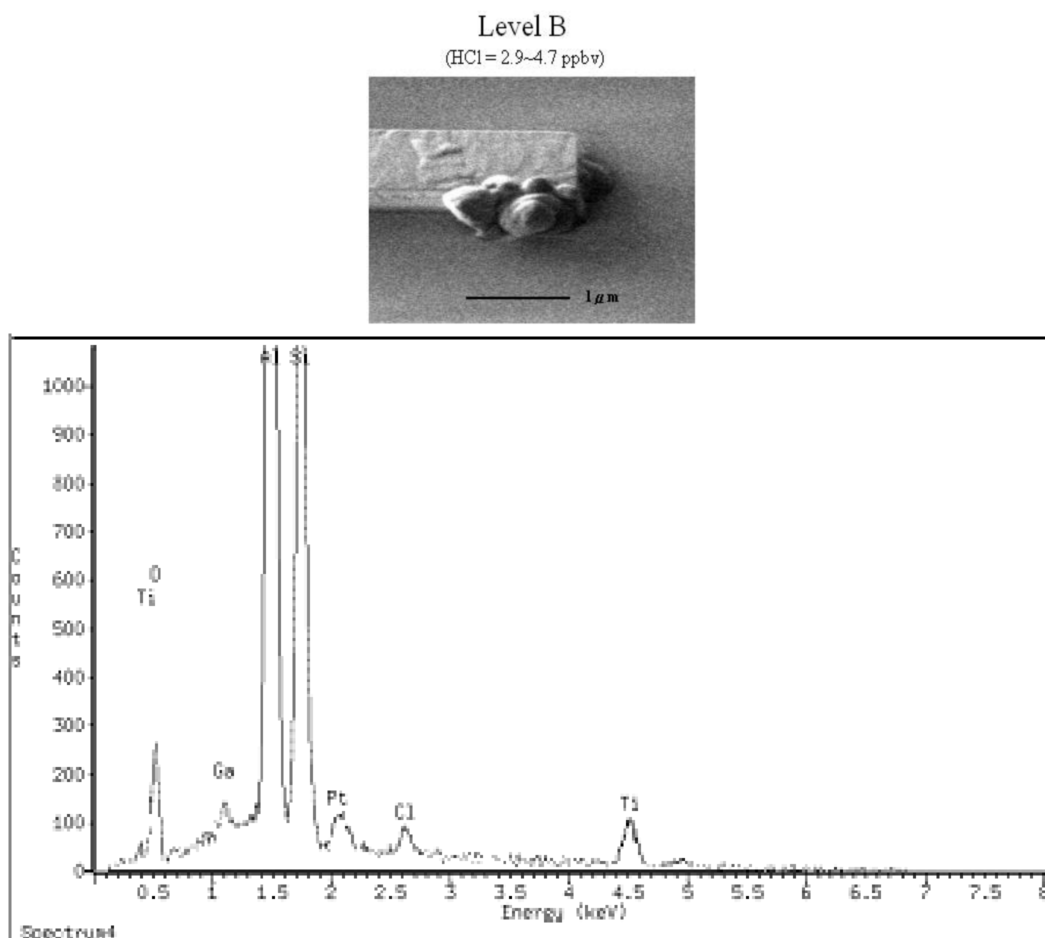


Fig. 5. OM/SEM/EDS analysis of pattern wafer under level B exposure.

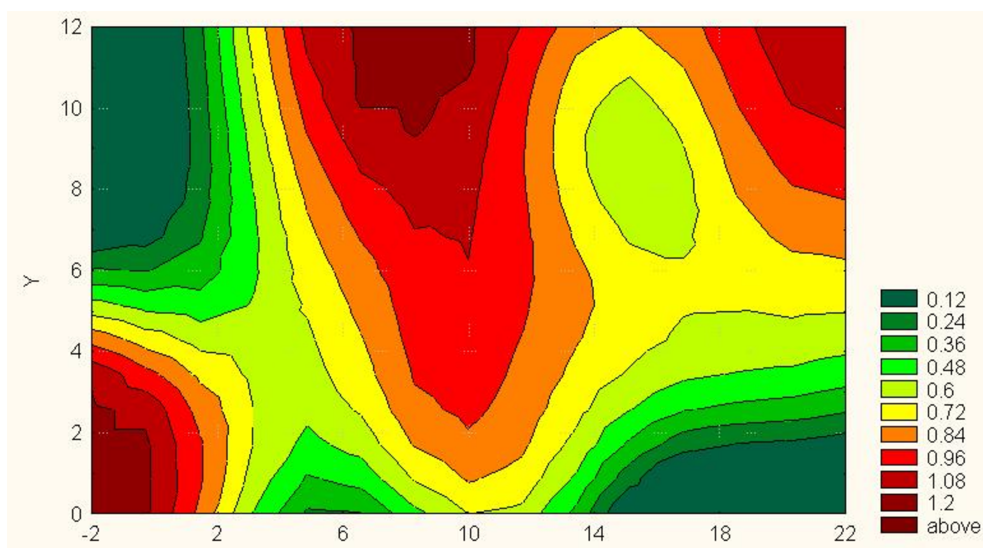


Fig. 6. The contour plot of monthly averaged HCl concentration distribution measured by IMS.

vaporized acids used in wafer cleaning and rinsing processes. The outdoor environment could also be the source of HCl as it had been detected to be the inorganic gas with the highest concentration (Tsai *et al*., 2003).

Pattern Wafers Test in a Real Fab

After knowing that the HCl concentration in the etch area was the highest, pattern wafers were then placed in the etch process area. In the experimental period, the IMS was employed which provided the real-time HCl concentration of 12 hours exposure during each test. Table 2 displays the HCl concentrations of Tests 1–9 measurements by IMS. The highest HCl concentration for Tests 1–8 was 0.7 ppbv, and there was no observation of any metal corrosion defect for Tests 1–8.

For the sampling data of Test 9, metal corrosion defects were observed by optical microscope inspection after the first hour wafer inspection, and they were confirmed by surface scan and EDS analysis. Fig. 7 shows the HCl concentration variation during the 2 hours IMS measurement and the highest concentration was 4.2 ppbv as occurred during the first hour. Fig. 8 shows OM/KLA/EDS analysis of the pattern wafer used for Test 9 after the first hour exposure and the metal corrosion defects were clearly observed by the OM inspection. The EDS result also shows that the Cl element was detected on the pattern wafer deposited particles. Thus, the result shows that the metal corrosion defect was observed although the duration of HCl concentration over 2.9 ppbv was only 10 minutes with the highest concentration of 4.2 ppbv.

Comparison of Al-Cu Pattern Wafer and Al-Si-Cu Pattern Wafer

One of the major differences between 200 mm and 150 mm pattern wafers is the composition of the metal layer. The metal layer used in the 200 mm wafer is Al-Cu film while it is Al-Si-Cu film for the 150 mm wafer. And based on the semiconductor textbook (Wolf and Tauber, 1986), the pattern wafer with Al-Si-Cu metal layer is more sensitive than with Al-Cu layer. In this study, HCl concentrations > 2.9 ppbv were measured, then metal corrosion defects were observed. Compared with the prior study (Wu *et al*., 2010), the critical HCl concentration that results in metal corrosion defects is 4.1 ppbv.

According to this case study in a real fab environment, metal corrosion defects were found with HCl concentration at 2.9–4.2 ppbv during a 10 minutes short exposure test. This result shows that the pattern wafer exposure time can be shortened from 1 hour to 10 minutes for a real 150 mm fab application.

CONCLUSIONS

In this study, a pattern wafer corrosion monitoring method for 150 mm fab was proposed and it has been proved to be very useful for the detection of Cl^- contamination and thus the contamination sources can be traced as early as

Table 2. HCl concentration in cleanroom during the real fab tests as measured by the IMS (Unit: ppbv).

Test	Min.	Max.	Ave. ± SD
1	0.1	0.3	0.2 ± 0.1
2	0.1	0.4	0.3 ± 0.1
3	0.1	0.7	0.4 ± 0.2
4	0.1	0.5	0.4 ± 0.1
5	0.1	0.4	0.3 ± 0.1
6	0.1	0.4	0.3 ± 0.1
7	0.1	0.7	0.4 ± 0.2
8	0.1	0.4	0.3 ± 0.1
9	0.1	4.2	1.2 ± 1.1

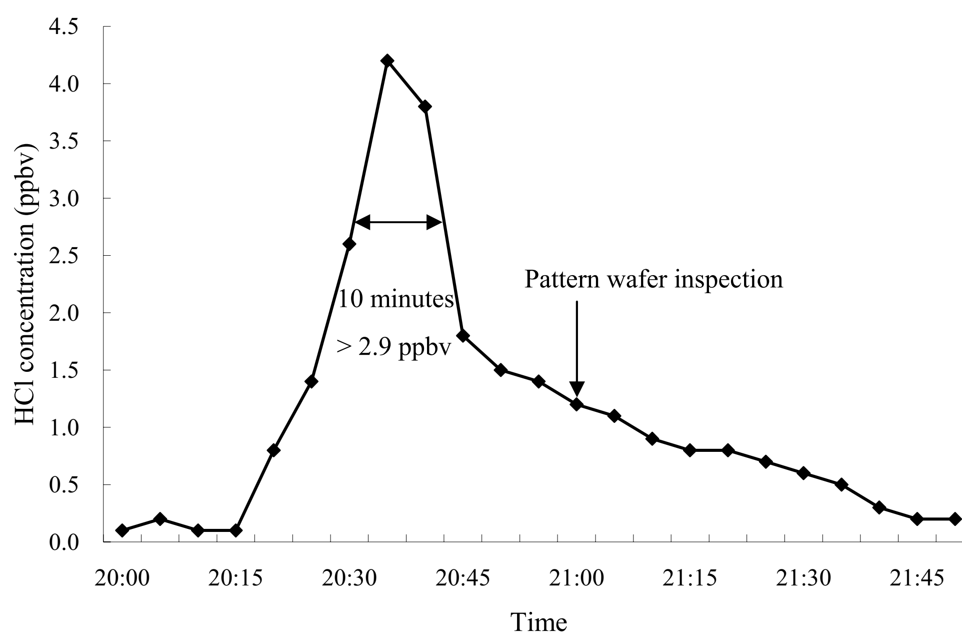


Fig. 7. Two hours measurement data during test 9 of HCl exposure in the real fab test.

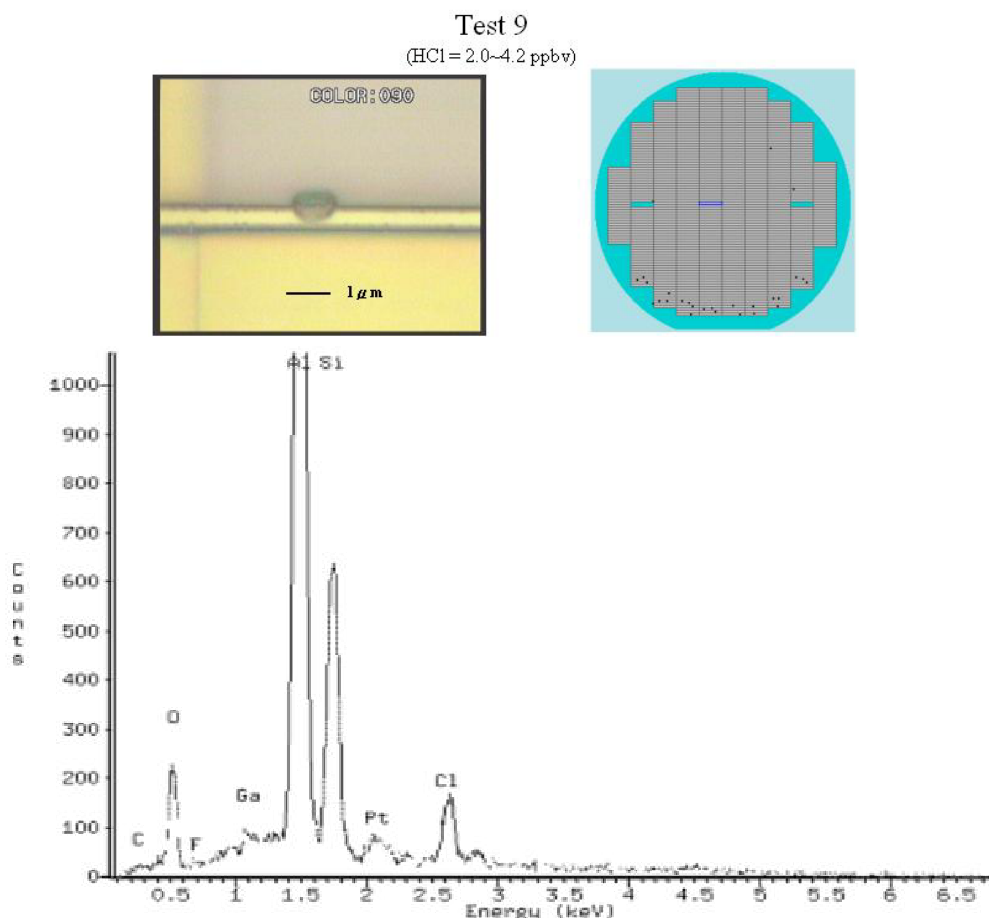


Fig. 8. Optical microscope /Surface scanner/ Energy dispersive spectrometer analysis of pattern wafer under test 9 after the first hour exposure.

possible. The designed pattern wafer in this study can shorten the exposure time from 2 hours to 10 minutes. It is valuable for a real fab application and can be verified in a real fab environment. It has also been proved that permeation tubemethod as a Cl^- source could be used as a simulated contamination source which can control the simulation environment to be in ppbv levels and within ~4% error range. The results of actual clean room sampling by IMS showed that the higher HCl concentration was measured at etch process area as demonstrated by the contour plots of HCl concentration distributions. For a 0.5 µm logical metal layer lithography technology (TiW film as a barrier layer), metal corrosion could occur when placing pattern wafers in an HCl molecular contamination mini-environment of 2.9 to 4.7 ppbv for one hour, and 2.9 to 4.2 ppbv in a real fab exposure for 10 minutes. This means that even in a mature fab for 150 mm wafer production, the HCl real-time monitoring method is quite important, and the pattern wafer method provides a cost-effective solution.

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Received for review, July 25, 2011
Accepted, October 3, 2011