Rapid Monitoring of FOUP Outgassing with ClearFab AMC Solutions

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FOUP Outgassing

Semiconductor manufacturing often involves hundreds of processes. Between manufacturing stages, wafers are transported and stored in specialized plastic enclosures; Front Opening Unified Pods (FOUPs). Wafer defects have been related to increases in the time between processes ("queue times") and the interaction of wafers with compounds that outgas from the surfaces inside the FOUPs [1]. Precise and sensitive monitoring of outgassing compounds guides process adjustments to decrease defects and optimize FOUP cleaning processes. Measurements also inform the development of new FOUPs, using novel polymeric materials, and new surface treatment procedures [2]. This work demonstrates use of a ClearFab AMC Monitor continuously monitoring FOUP outgassing after standard cleaning procedures.

Experimental Procedure

FOUP outgassing (~50 liters) was monitored using a ClearFab AMC Monitor (Figure 1). The monitor directly sampled the air and instantaneously reported concentrations of trace organic and inorganic compounds.

Experiments were conducted by spraying a solution containing hydrochloric acid (HCl), hydrobromic acid (HBr), formic acid (CH_2O_2), acetic acid (CH_3COOH), and nitric acid (HNO_3) into the FOUP and then flushing with nitrogen to sanitize. The equivalent mass deposited from the solution ranged between 0.15 µg to 1 µg. Hydrofluoric acid (HF) was introduced using a permeation tube with an emission rate of 125 ng/min. The internal volume was flushed with a constant flow of N_2 (2 L/min) to ensure the FOUP interior was well mixed and to simulate the cleaning of the FOUP container. This resulted in a FOUP ventilation rate of < 60 minutes.

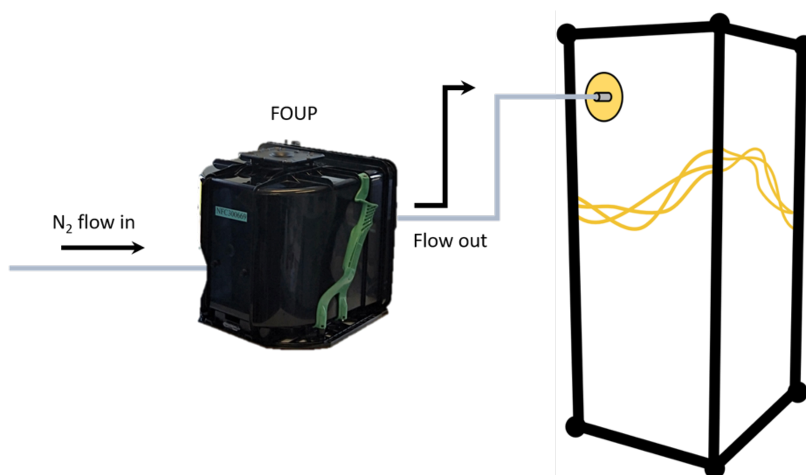

Figure 1. *Schematic diagram of the experimental procedure*

The measurement protocol had three steps: (1) measure the FOUP background for 5 minutes, (2) place the HF permeation tube inside the FOUP for two minutes and immediately inject the acid solution, (3) continuously measure the mixing and subsequent decay of the compounds until concentrations return to background values.

Results

After injecting the acid solution, the mixing took approximately 3-4 minutes (including evaporation of the injected solution) before the flushing initiated a decay of analyte concentrations. Figure 2a shows the decay of nitric acid and the reproducibility of acetic acid decay between repeat experiments (Figure 2b). All compounds showed a double exponential decay, with sticky compounds persisting at trace concentrations (10-100 pptv) even 100 minutes after injection. The double exponential fit (Equation 1) was used to retrieve the compound dependent time constants, which represent the flushing timescales of each compound from the FOUP. τ1 in equation 1 represents the e-folding time for the fast decay (gas volumetric exchange in the FOUP) and the second time constant (τ2) represents the slower outgassing from FOUP surfaces. The latter is significantly longer and depends on the interactions of the acid with the FOUP surfaces. Figure 2a shows an example of the double exponential fit for HNO_3 which has a significant interaction with the walls of the FOUP and persists for much longer than the other acids.

Equation 1 $C(t) = C_1 e^{-t/\tau_1} + C_2 e^{-t/\tau_2} + C_b$

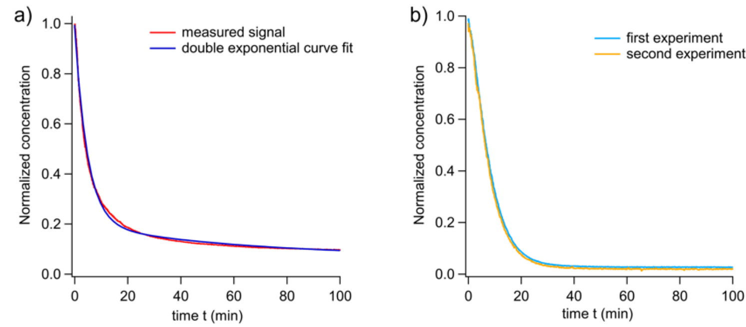

Figure 2. *a) Normalized concentration C(t) of nitric acid (red) and its double exponential fit (blue). b) Normalized concentration of acetic acid after deposition of 1 µg (first experiment) and 0.15 µg (second experiment), showing the reproducibility of the system.*

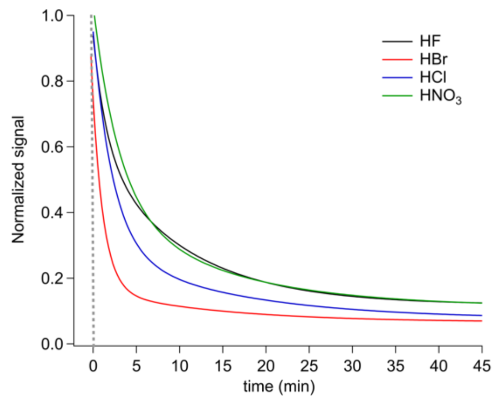

Figure 3. *Exponential decay of different inorganic acids during the first 45 minutes of flushing. The response of the acids to flushing is related to their vapor pressure and surface interactions with the inner surfaces.*

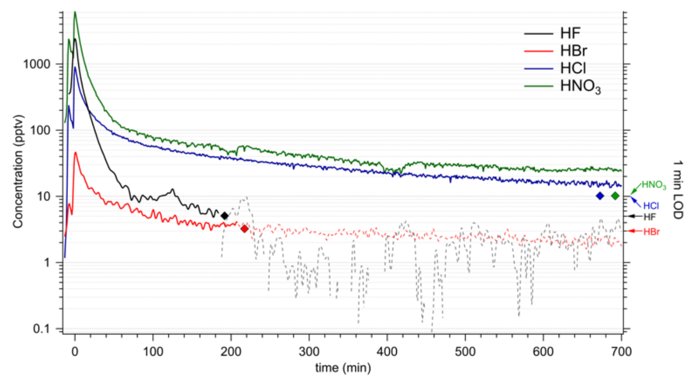

Figure 4. *Concentration decay of common inorganic acids in the environment. The markers show the quantification limit of each compound. Arrows on the right axis show the ClearFab AMC Monitor's LODs after 1-minute. Diamonds show the point in the 11-hour long flushing where the measured signal falls below the monitor's LOD. For HCl and HNO_3, measurable signal persists even after 11-hours.*

Figure 3 shows the response of HF, HBr, HCl and HNO_3 to nitrogen flushing over the first 45 minutes after reaching stable concentrations. Table 1 summarizes the time constants (τi) from the double exponential fits in Figure 3. Most of the acids have similar response in the first few minutes when volumetric flushing dominates, however, as shown in Figure 4, on longer timescales some acids persist at trace concentrations (10-30 pptv) for hours. Formic, acetic, hydrofluoric and hydrobromic acids all reached near background concentrations (90% decrease) in the first 60 minutes, implying no severe attenuation or memory on the inner surfaces (Table 2).

When using a ClearFab AMC Monitor it is easy to estimate the optimal end point of a FOUP cleaning process. The slow decay of nitric and hydrochloric acids suggests that cleaning processes which are not optimized for slow acid outgassing, or that cannot sufficiently detect at low concentrations, could suffer from continued contamination risk. Table 2 summarizes the performance of ClearFab AMC Monitors for the detection of organic and inorganic acids.

Compound	τ_1 (min)	τ_2 (min)
HCl	2.3	15
HF	1.9	12
HBr	1.3	29
HNO_3	4.2	35

***Table 1**. Decay time constants (τ) for each acid shown in Figure 3. The values of τ2 are calculated according to the fit when the concentration starts to stabilize.*

Conclusion

The capabilities of these monitors are well suited for both multi-port facility monitoring or mobile measurements. Outstanding detection limits and simple, autonomous operation presents a paradigm shift in the ability to quantify airborne and surface-bound AMCs at low concentrations as line widths are pushed to smaller dimensions.

Name	Formula	1 s LOD (pptv)	1 min LOD (pptv)	T90 (s)
Hydrochloric acid	HCl	230	10	2.4
Hydrobromic acid	HBr	128	3	1.5
Hydrofluoric acid	HF	24	5	4.0
Nitric acid	HNO_3	41	5	11.1
Formic acid	HCOOH	90	11	1.9
Acetic acid	CH_3COOH	314	40	1.9

Table 2. *Detection limits and response time*

References

1. Jeong et al. Control of Wafer Slot-Dependent Outgassing Defects during Semiconductor Manufacture Processes. 2019 DOI: 10.1109/ASMC.2019.8791794
2. Gonzalez-Aguirre et al. Control of HF Volatile Contamination in FOUP Environment by Advanced Polymers and Clean Gas Purge. 2015 DOI:10.4028/www.scientific.net/SSP.219.247

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