# Defectivity decrease in the photolithography process by AMC level reduction through implementation of novel filtration and monitoring solutions

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## ABSTRACT

A case study of drastic photolithography defectivity reduction on i-line and Deep-UV (DUV) tools is presented. We show how this result is linked with reduction of Airborne Molecular Contamination (AMC) in clean room by combined installation of novel type of filters on tracks and on the recirculation air treatment. The root cause was identified to be the presence of acetic acid in clean room created by a reaction with the filters (mounted on track tools to exclude ammonia contamination of the process) and the photo solvent itself (here mainly 1-methoxy-2-propanol acetate: PGMEA). Crucial for the project success was the use of a real time monitoring tool to detect the sources of Volatile Organic Compounds (VOC). Finally, a model of chemical reaction of satellite defects creation is discussed based on a Time of Flight Static SIMS (TOF SSIMS) analysis together with new AMC specification for acetic acid for the photolithography area.

**Keywords:** Photolithography, defect, acetic acid, filtration, PGMEA, AMC, VOC, monitoring

## 1. INTRODUCTION

It is universally known that particle control is an essential part of the semiconductor fabrication process. Clean room technology, which relies on the use of HEPA and ULPA filtration, has in the past almost exclusively focused on the control of incrementally smaller particles. Nevertheless, as a consequence of device reduction, AMC has also become a key detractor of yield. This is particularly true in photolithography area where a variety of species can impact the process. However, interactions regarded as most relevant and therefore taken care of are:

- Influence of bases, in specific ammonia, to influence Critical Dimension (CD) by T-topping effect to CAR (Chemical Amplified Resists) on an acid amplification basis.
- Refractive organic compounds contaminate optics [1, 2]: Silicon compounds which are photolysed into radical fragments by 193 nm UV light can combine with oxygen to create amorphous silicon dioxide on optical surfaces. This consequently can lead to non reversible lens damage.
- Acids react with ammonia to create a salt deposit on lenses or reticles. Here again, this is especially true for 193nm tools where the reaction is photo-initiated (Figure 1).

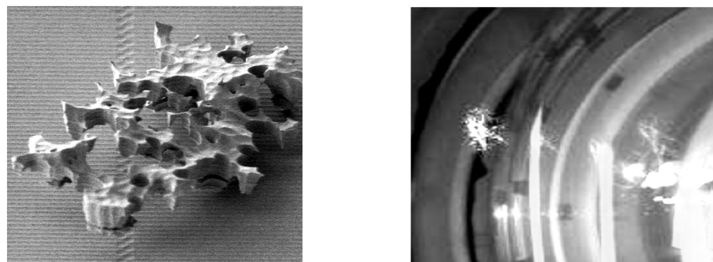

Figure 1. Micro-crystallite salt on a lens of a 193 nm exposure tool (picture provided by STMicroelectronics Crolles).

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In our previous AMC control concept (Figure 2), these interactions have been accounted for by:

- Track mounted AMC filtration targeting bases.
- Scanner mounted AMC filtration targeting acids, bases and refractory compounds.
- Periodic monitoring of the AMC content by bubbling and Tenax® tube sampling and analysis on the basis of a fixed grid measurement approach.

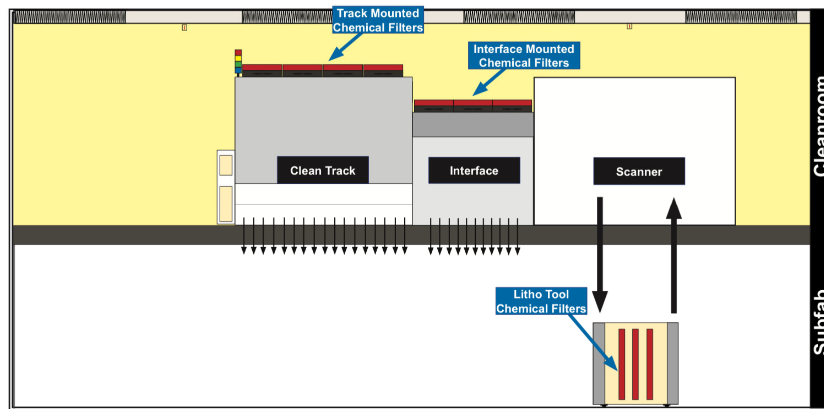


Figure 2. Initial AMC control strategy: lithography tool filters treated VOC, acid and bases. Track and interface mounted filters treated amines/bases only.

All measures and AMC values analyzed have been in-line with the ITRS AMC limits 2007 [3] demands for the Year-of-Production 2008 DRAM ½ pitch 65 nm mode, limits for photolithography (clean room environment). This refers in specific for the limits set for inorganic acids (< 5 $ppb_v$), ammonia (< 50 $ppb_v$) and condensables (< 26 $ppb_v$). Nevertheless we had to face a crisis in the photolithography area with the following effects:

- Lens contamination on 193 nm DUV exposure tools by microcrystal formation.
- Defectivity increase on wafers on i-line and DUV scanner/track lines (black points and satellite defects).

Analysis of AMC content in the clean room air spotted a constant increase of acetic acid for one year. Our thesis was to track it back to a non explained excursion of PGMEA in Q5 which however showed a recovery to lower levels again (Figure 3).

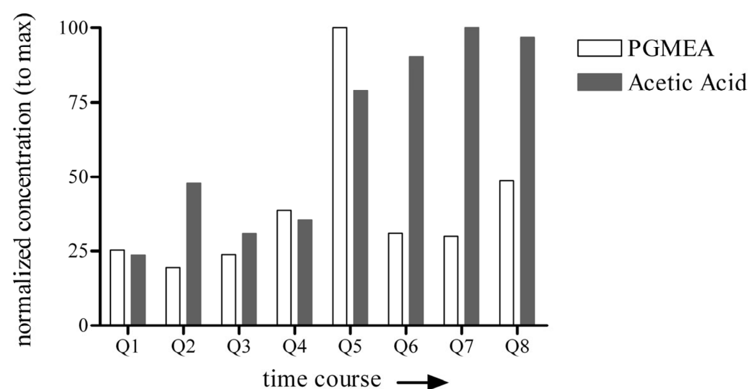


Figure 3. PGMEA & acetic acid trend in the photolithography area (2 years time course).

For acetic acid measurement, a clean room air sampler (sampling pump and bubblers) was used. A blank (another sampling module that is treated in the same manner) accompanies the sample. This ensures that only contamination from the air or gas is quantified (not contamination generated by sampling, shipping, or handling). Acetic acid is then analyzed by Ion

Chromatography (IC) DIONEX DX500 "ultra traces" and the quantification limit is 0.1 $ppb_V$ for the 8 hours sampling time we applied in our study. For PGMEA analysis, special stainless steel sampling tubes containing distinct beds of proprietary adsorbent materials are provided by TERA to trap organic compounds in its gaseous state. This technique allows to pre-concentrate the volatile organic compounds and the condensable at very low concentrations. Gas Chromatography-Mass Spectrometry (GC-MS) is a method that combines the features of gas chromatography and mass spectrometry to identify different substances within a test sample. Applications of GC-MS include the identification of unknown gas constituents. The GC-MS has been widely heralded as the gold standard for substance identification because it can be used to identify an isolated compound. In our case, PGMEA is analyzed by an Automatic Thermal Desorption (ATD) GC-MS PERKIN-ELMER Clarus 500 at the detection limit of 0.01 $ppb_V$ for 4 hours sampling.

For the generation of acetic acid, we suspected a leakage of EC-solvent/PGMEA to the clean room air or the subfab to be the initiator, followed by the adsorption of the compound to base filter media of tool filters and an acid catalyzed hydrolysis reaction (Figure 4). Acid catalysis of this reaction is provided by strong acid impregnated onto standard adsorptive amine filtration media.

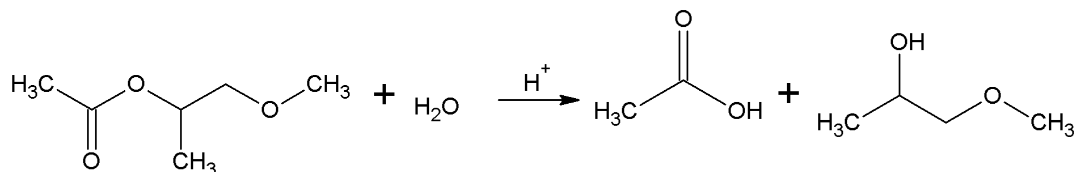


Figure 4. Acetic acid creation by PGMEA hydrolysis.

This hypothesis was checked by impinger measurements at the outlet of the track filters as well as the scanner inlet filter: the results showed acetic acid values 8 to 20 times higher at the outlet compared to the inlet values (Figure 5).

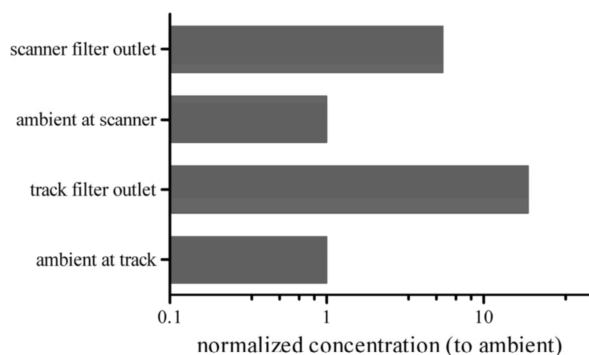


Figure 5. Acetic acid levels on ambient and downstream tool-interface filters (initial status, high defectivity).

Literature says that the origin of "satellite spots" and "black points" on DUV tools (248 nm or 193 nm), represented on Figure 6 by a SEM image, is caused by the formation of a complex salt from Photo Acid Generator (PAG), quencher and developer [4] (see also discussion section 2.3). It is presumed that polymers adhere to this complex salt nucleus and form the satellite defect. As PAG is involving the generation of acidity during development process, we may assume that an increase of acetic acid contributes in an unwanted way to the complex salt formation. Another article [5] describes extended rinse time as a possible solution to the symptom. However, we were interested to understand and stop the root cause.

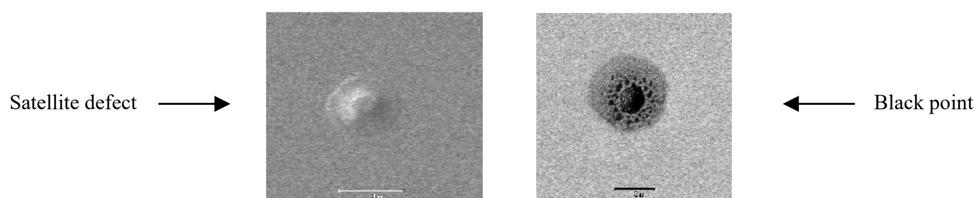


Figure 6. SEM images of a satellite defect and a defect noted as "black point".

As discussed previously, we further had to face the fact that the unwanted release of acetic acid into the DUV-exposure tools / scanners resulted in lens hazing by microscopic salt particles obviously of the nature of an ammonium-acetate salt (no other acid were detected in the clean room at this time). This is notable as the limits for amine content in the tool-inlet air downstream of the inlet filter stack never exceeded the limits set for the tool operation.

At this time, it became clear that the existing tool filters, once be charged with PGMEA, would continuously desorb increasing levels of acetic acid high enough to impact defectivity and damage lenses in 193 nm tools. Furthermore, we expected that new unaccounted leaks would re-initiate this process even on new filters sets. Therefore a stable overall solution was required. The solution chosen to solve this issue was an integrated, analytically supported approach which involved:

- Source identification and tracking of PGMEA by on-line monitoring.
- Acetic acid level reduction on tool level and the prevention of repeat generation of acetic acid by a new filter type.
- Verification of the decrease in defectivity due to the decrease of AMC level.
- Overall stabilization of the system of clean room and tools and spill event prevention by recirculation air treatment.
- Establishment of revised AMC specifications for PGMEA and acetic acid.

We will describe in the following paragraphs the methods and results for the above given 5 steps including the verification of effectiveness on a clean room and wafer level that resulted in the complete restoration of a low defectivity / high yield production environment.

# 2. RESULTS AND DISCUSSION

## 2.1 Source identification and tracking for PGMEA

A standard method for PGMEA sampling and monitoring is the use of actively collected TENAX® tube samples or passive collecting carbograph sampling tubes, followed by TD/GC-MS analysis. Given the necessary accumulation of substance on the sampling tube to perform the analytical procedure and render results in the $ppb_v$ concentration range the required sampling time is between 4 hours and 48 hours, depending on the laboratory methodology.

Although these methods thoroughly applied render good and stable results at a fixed sampling point over time, results are always average values with regard to the sample period. Thus, they will miss peak wise elevated concentrations during the sampling period and only represent them as an increased average value. Moreover, they miss the continuous time course of concentrations that may be dynamic on a 24 hours production basis.

In our case, previous sampling at fixed grid points had not revealed any excursion from our AMC specification. Yet, the level of acetic acid and defectivity increased constantly.

On the background, that use of EC-solvent/PGMEA in production varies over time and related to batch processing it appeared clear that:

- By tube sampling, no excursion / spill can be resolved to its maximum value.
- No correlation of increased AMC levels to the operation or status of a specific tool can be found by bad time resolution.

Therefore, a PGMEA monitoring method was needed that had at least the same or shorter time resolution than batch or service processes. It should have a quantification capability in the lower $ppb_v$ range and is operational on a 24 hours / 7 days basis.

This method was found in the on-line detection and quantification of PGMEA and further compounds with an AMC monitor based on the principle of PTR-MS (Proton Transfer Reaction – Mass Spectrometry), supplied by artemis control AG/Ionicon Analytik GmbH [6]. The instrument (schematic Figure 7) has the following characteristics:

- Identification and quantification of a range of pre-set organic compounds to a detection limit of 0.5 $ppb_v$ per individual compound.
- Parallel quantification of up to 10 compounds.
- Quantification of the concentration of all 10 compounds at a cycle time of 60 seconds per cycle.

For this, the instrument requires a gross sample flow of 500 ml/min from which a splitted partial flow of 7 ml/min is brought continuously to the chemical ionization chamber of the device. Ionization is achieved by transfer of a proton ($H^+$) to the analyte molecules from a $H_3O^+$ discharge source to mainly render M+1 analyte ions. Analyte ions are separated by a quadrupole and detected by a Secondary Electron Multiplier (SEM) detector. The low detection limit in the on-line mode that does not require any pre-concentration is attributable to the complete conversion of analyte molecules to analyte ions and is based on the patented design of the ionization set-up.

Response to analyte / AMC concentration changes expressed as jump-time $t_{90}$ is about 100 ms . Therefore, the time resolution of the measurement was only limited by the programmed scan and count intervals.

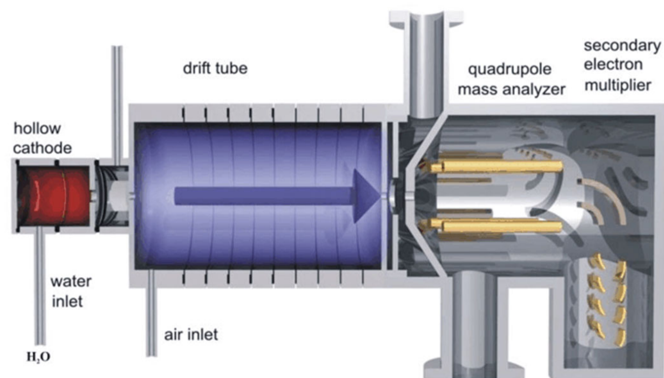


Figure 7. Schematic of PTR–MS: detection relevant parts (Picture provided by Ionicon Analytik GmbH).

In the currently described project, we applied the instrument in a thorough screening of the clean room and subfab level of the photolithography area. This screening was planned and oriented on the basis of the tool clusters and flow direction and regime of the re-circulated fab air. It was soon clearly understood that (Figure 8):

- PGMEA concentrations and emission source strength varied substantially per location even at a special resolution.
- PGMEA concentrations at a fixed spots varied substantially over time by a factor of more than 30 between low level and peak level.
- PGMEA peak concentrations could by correlation analysis be well matched with the batch operations of just 2 tools (tool A) of 20 tracks thus supporting a substantial source reduction by remediation action on those identified tracks.

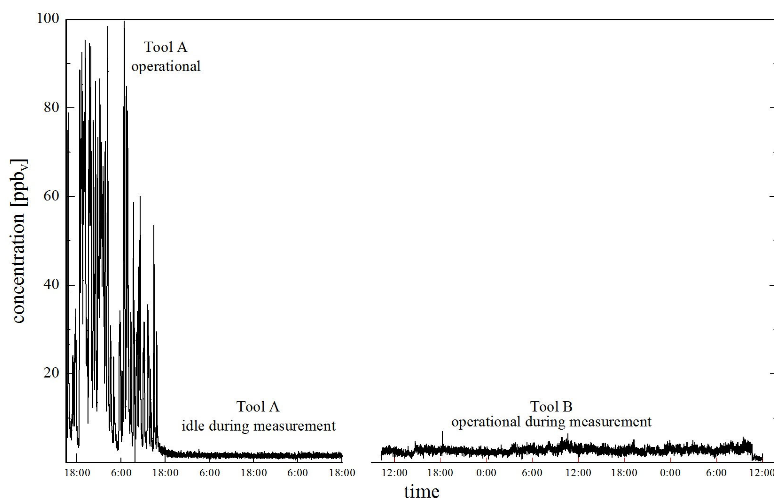


Figure 8. PGMEA peak concentrations at subfab level; problems arising on Tool A whereas Tool B proves to operate normally.

The spill cases are related to solvent vapor emissions from improper exhaust tubes on the two tools and their operation (see details in Figure 9). The remediation action taken was replacement of the exhaust tubing including a minor change of the tubing material. This resulted in a complete vanishing of the peak emissions.

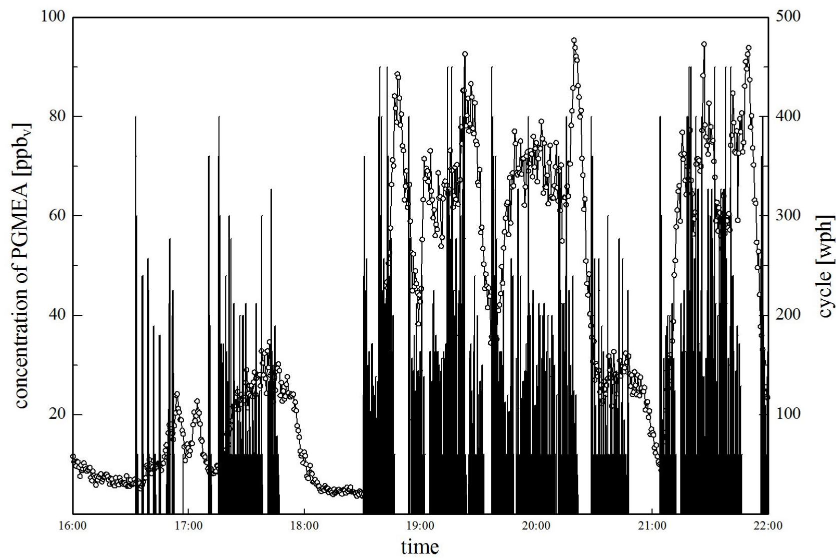


Figure 9. PGMEA peak concentrations at subfab level (o symbols; left axis) plotted together with throughput on problematic tools ( | , right axis); batch processing immediately releases PGMEA which is purged out only slowly.

Identification of the main sources of PGMEA in the clean room was mandatory to be able to deploy the next steps of the project. However, it was also important to track non permanent sources: the tool allowed us to implement several corrective actions that helped to reduce the final baseline of PGMEA. For example, we have discovered that during maintenance on the specific solvent supply system, non negligible leaks were registered by the AMC monitor (Figure 10).

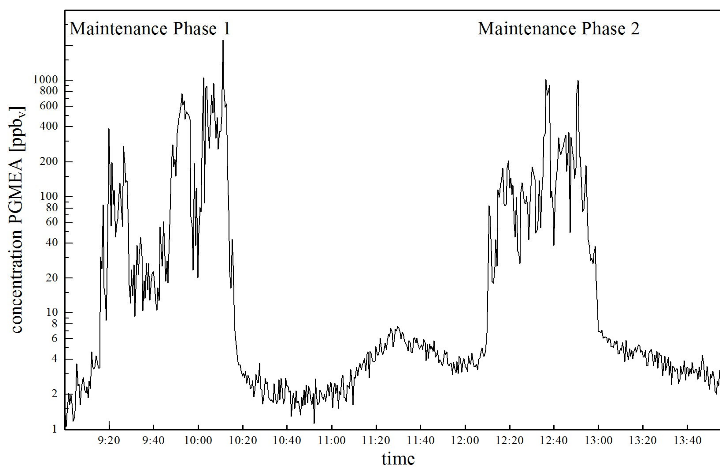


Figure 10. High level of PGMEA detected during solvent system valve maintenance.

The monitoring tool proved to be very useful for the parallel fast and time-resolved quantification of further VOC, in specific solvents, used and emitted in clean room operation (IPA, acetone, and others). Therefore identifying and reducing the overall VOC load to the clean room environment and tool filter is possible.

Moreover the effects of AMC-filtration on the recirculation air handling system (see section 2.3) set operational per zone, could be recorded in real-time and quantified in terms of efficiency (Figure 11).

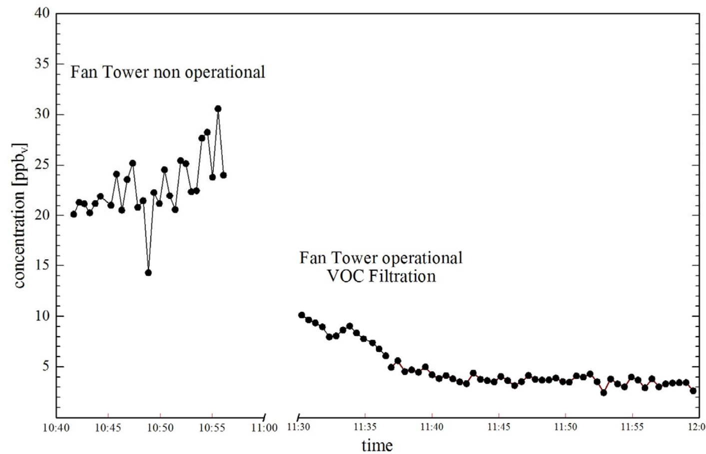


Figure 11. Real-time record of PGMEA reduction in a clean room segment on start-up of a single Fan Tower equipped with VOC filtration.

## 2.2 Acid level reduction on tool level and prevention by application of a new filter type

Although the source of PGMEA as chemical basis for acetic acid generation could be found and stopped, all track and scanner mounted filter systems showed a substantial content of PGMEA due to the extended challenging time. Due to the mixed adsorption/chemisorptions nature of the standard track filters applied, PGMEA was irreversibly adsorbed and yielded a steady freight of acetic acid to the air flow into the tool.

The removal of the challenged filters and replacement with new filters was indicated with the following requirements:

- Removal of ammonia content in the air to or below process specifications.
- No generation of acetic acid from PGMEA traces in the air once it passes the filter units.
- Removal of acetic acid traces from the tool air once acetic acid should be in the clean room air and should pass the filter units.

The solution was found in applying a multifunctional filter stack based on dedicated filter foam media technology (supplied by artemis control AG). The total filter stack of 155 mm height was compiled of two stacked foam media filter CTF-F type of 78 mm height increment.

The media technology applied is a composite of an open-cell polyurethane carrier grid loaded with spherical sorbents. It produces a densely loaded 3-D-filter structure of high sorption efficiency and low flow resistance. The various spherical sorbents (nanobeads®) have different material basis reaching from taylor-made micro-porous carbons to functional porous polymers. The media types are produced in media layers that can be stacked in the most appropriate way to yield a customized site specific performance and operation life time.

In the current case the overall system consists of layers of spherical carbon media as well as bi-functional polymer material for removal of bases and acids.

The performance tests under the harsh conditions of the non-remediated fab revealed the superior performance of the system. In particular no generation of acetic acid from PGMEA challenge to the filter was observed throughout and after the project period. To stop the flow of acetic acid to the clean room air was a key point for project success. Moreover, the results in term of ammonia removal efficiency, which is the primary functionality of these filters, is also really good as it can be seen on Figure 12 (compared to old filters that had lost efficiency due to overpass lifetime).

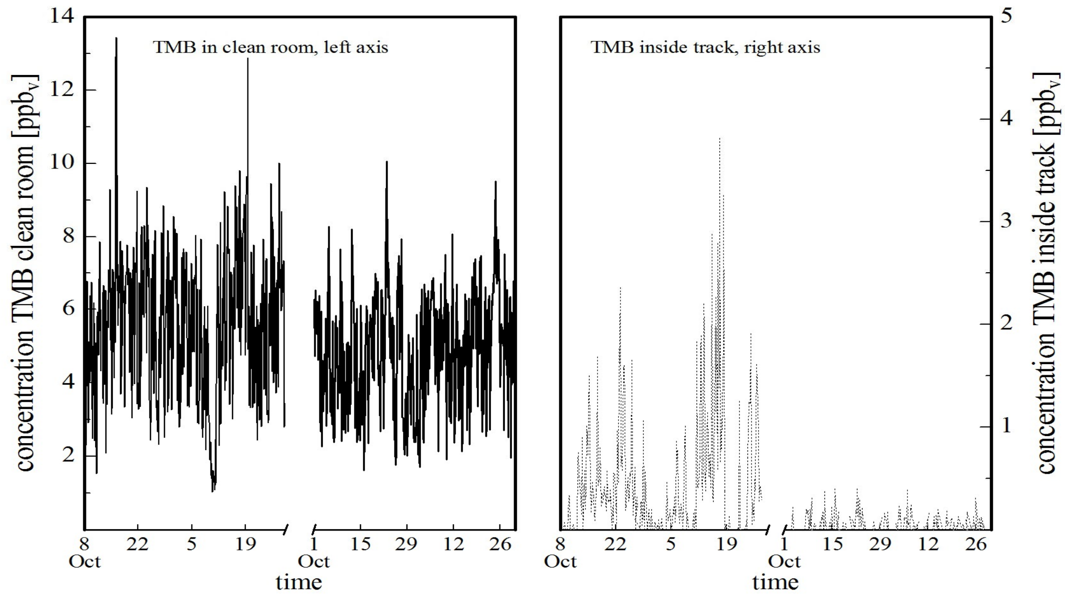


Figure 12. Two months trends of total amine in subsequent years (left and right segment) registered by online Total Base Monitoring (TMB) (5 points per day). TMB is given for clean room level (left diagram) and wafer level /inside track (right diagram). Note different scale of the concentration axis.

### 2.3 Verification of the defectivity decrease on wafer tests

The primary goal to substantially decrease wafer defectivity on DUV-clusters to normal levels was achieved tool by tool in parallel to the change of tool filters and the elimination of the acetic acid discharge to the tools. Figure 13 shows the reduction of the overall defectivity count on a DUV-cluster as well as the defect count variation noted for individual wafers before the tool filter change. It is obvious from the comparison with acetic acid levels found inside the tool clusters at the respective time windows to attribute the DUV-process recovery to the remediation action of filter change / acetic acid exclusion.

These defectivity tests were carried out on 200 mm, CZ, <100> orientation, 725 µm thick Si wafers with p-type boron doped at 1-50 ohm·cm. The wafers were processed on DNS SK2000 Track / ASML750 stepper with the following conditions: BARC DUV30-6 (570 Å) coating / DUV M78T DUV resist (4400 Å) / 90s soft bake at 130° / post exposure bake at 140°C for 90s / developer TMA238 / hard bake 90° for 90s.

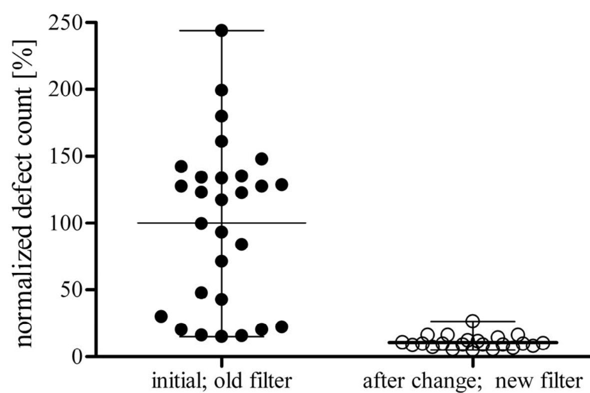


Figure 13. Normalized defectivity counts of wafers in the DUV process; massive reduction and stabilization of wafer defectivity after tool filter change.

The creation of satellite defects has been described [4] as a consequence of a chemical reaction between the PAG used in DUV CAR with the developer. The defect spot should show elevated concentrations of nitrogen and sulfur.

In order to confirm this hypothesis, we have performed some TOF-SSIMS measurements on a monitor wafer where some satellite defects were present after the development process (the size of the defect was here about 2.5 x 2.5 µm²). TOF-SSIMS is a physical and chemical analysis method of material surface. It is based on charged particles detection (secondary ions) which is produced under a bombardment of incident ions (primary ions). The nature of emitted secondary ions is intimately bound to the sample surface nature. Moreover, the secondary ions come from the utmost surface (10 Å), so SIMS is one of the uppermost surface sensitive techniques. The SIMS phenomenon is sufficiently general to analyze all type of samples (ultra-vacuum compatible) and application fields are as large as elemental and molecular analysis, depth profiles, ionic imaging or chemical identification. In the present case, these analysis has been performed on a Time of Flight Static SIMS from IONTOF (TOF 5, tool from CIMPACA characterization platform) using the chemical identification capability. $Bi_3^+$ ions at 25 kV have been used as primary ion beam. The analysis has been performed in static mode (primary ion dose less than $5 \cdot 10^{+12}$ ions/cm²). A pulsed electron flux at low energy (<20 eV) assume the charge neutralization of the surface.

Chemical map of positive secondary ions have been performed in two different settings:

- High spectral resolution and low lateral resolution (HC-Bunched).
- High lateral resolution (BA-image).

One mass spectrum analysis has been performed out of the default, directly on the Bottom Anti Reflective Coating (BARC) whereas a second analysis window (25x25 µm²) was scanned on the default itself. The results show the presence of different masses on the positive spectrum - 28 ($NCH_2^+$), 42 ($NC_2H_4^+$), 58 ($NC_3H_8^+$) and 74 ($NC_4H_{12}^+$) - corresponding to pieces of TMAH (tetra-methyl ammonium hydroxide = $(CH_3)_4$-N-OH, 91 amu), used in the development process. Of course, this mass does not appear in the measurement performed out of the default. On the negative spectrum, we also note pieces of TMAH as mass 26 ($CN^-$) and 42 ($CNO^-$) plus presence of sulfites ($SO_2^-$, $SO_3^-$) which is significant for the PAG presence. These results confirm the initial hypothesis of complex salt creation by reaction of resist with TMAH and PAG. With this assumption, we understand that an increase of acetic acid into the developer ambient should result in an increase of defectivity.

At the same time, we were successful reducing the defectivity of i-line process (black dots) which is – lacking any AMC filtration – directly exposed to the acetic acid levels present in the clean room environment. Comparing defect density levels and acetic acid levels at the same time we proved to relate the black dot defect to the room level of acetic acid (Figure 14 and 15). These tests were carried out on 200mm, CZ, <100> orientation, 725 µm thick Si wafers with p-type boron doped at 1-50 ohm · cm. The wafers were processed on DNS SK200 Track / ASML250 stepper with the following conditions: BARC DUV30-6 (570 Å) / coating i-line SPR955 1.1 (12150 Å) / 90s soft bake at 90° / post exposure bake at 120°C for 90s / developer MF26 / hard bake 135° for 60s.

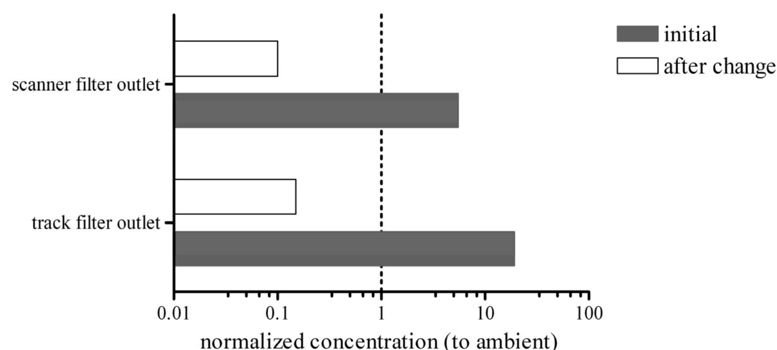


Figure 14. Comparison of acetic acid levels inside the DUV-tool segments before and after tool filter change; reference is made to the clean room ambient acetic acid level (normalized level = 1).

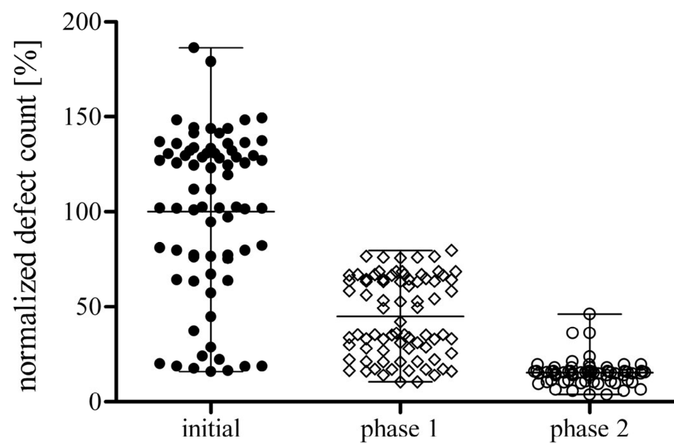


Figure 15. Reduction of the wafer defectivity on i-line clusters. Improvements parallel to the project progress (two phases) exchanging emitting DUV-tool filters and installation of recirculation air filtration.

The reduction of the acetic acid level in the clean room environment parallels the removal of emitting tool filters from DUV-tools and the phases of installation of recirculation air filtration (see section 2.4).

## 2.4 Overall system stabilization by recirculation air treatment

It has been shown in the previous sections that the VOC level in the clean room, in specific the level of PGMEA, is a result of EC-solvent emissions from operations within the photolithography area. Measures to verify that tool flows and pressure status as well as exhaust handling are optimized have been put in place. On-line monitoring is a veritable part of the action scheme. Nevertheless open-tool situations and therefore solvent emissions during maintenance are unavoidable as well as accidental solvent spills can occur.

Taking into account that PGMEA challenges to tool filters are expected to drastically reduce the life time of the balanced multi-purpose interface filters a first-line of defense measure was looked for to minimize the effects of spills to the economy of operation and to stabilize the overall system performance.

We decided to plan to equip the photolithography area with a dedicated VOC removal capability in the path of the recirculation air flow, able to cover 100% of the air flow moved within the clean room zone (Figure 16).

The solution was supposed to involve a minimum of changes to the building structure, the local flows and pressurization of zones and compartments as well as local weights to the inner clean room box and/or the support building structure. Other system requirements were described as:

- No changes or re-commissioning of the fan tower installation
- Operational within existing flow resistance & pressure drop regimes and limits
- Individually serviceable per zone & fan tower for optimized operation cost
- Individually adaptable AMC-removal capability per fan tower according to future demands
- Standardized modular set-up per fan-tower as cost control measure
- No interruption of production

Several options for the recirculation air filtration have been assessed during the planning phase:

- Option (1) consisted of a ceiling based combined AMC + particle filtration.
- Option (2) consisted in the implementation of large area wall-like filtration installation in the plenum segment.
- Option (3) consisted in a sub-fab based suction sided AMC filtration module.

While option (1) could not fulfill the demands on implementation on running production, adaptability to existing building constraints and serviceability and option (2) did not cope with demands on pressure drop limits, modular design and individual service option (3) fulfilled all necessary demands.

Option (3) installation was accomplished on the basis of a filter system that consists of a permanent re-usable filter frame furnished with exchangeable AMC filter panels of a mini-pleat design from activated carbon based composite material. Filter arrangement have been installed on a locally assembled and adapted grid system with a minimum down time of few hours for the individual air handler under refurbishment.

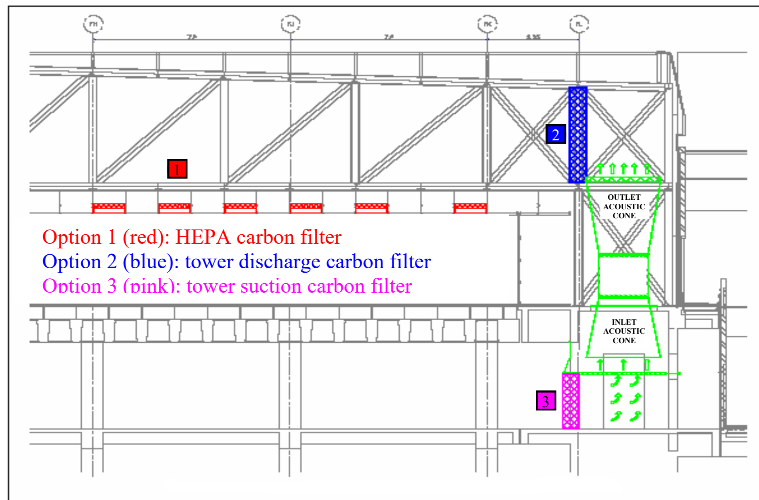


Figure 16. Options for solutions for the recirculation air treatment. Only Option (3) (suction installed AMC filtration per fan tower) was capable to fulfill the system requirements.

The effect of fan tower filters on global AMC level in the clean room was mathematically modeled before installation and was found to match these predictions by finalization of the project (Figure 17). We can see that following the Q5 crisis which initiated the unwanted reaction in the filters, the level was going down to the initial baseline during two quarters (Q6 and Q7). Then it increased again due to the exhaust leak on the two tracks discussed in section 2.1 (Q8 and Q9). The combined action on tools as described, on daily potential leaks during service and the recirculation air treatment led to a drastic diminution of PGMEA in clean room to a level never seen before. At the same time, the level of acetic acid drops systematically too.

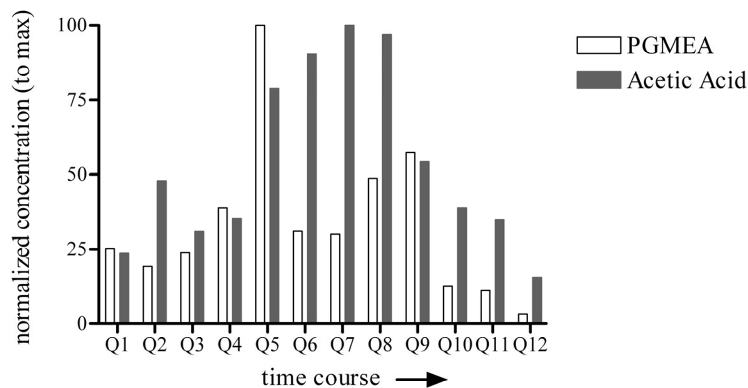


Figure 17. PGMEA & acetic acid trend in the photolithography zone; Q9 – Q12 during AMC control project.

## 2.5 Establishment of a revised AMC-specification for PGMEA and acetic acid

Acetic acid was identified in all cases of hazard creation (wafer defects on i-line process steps, wafer defects on DUV steps and salt induced lens hazing) as key component or relevant defect contributor. Consequently we included both an AMC-limit for acetic acid per compartment into our overall AMC-specifications. With regard to photolithography area AMC

limits we included acetic acid into the limit of overall acids in the zone not to exceed 5 $ppb_v$. This is our specific experience driven extension of ITRS-AMC-limits that leave organic acids as "tbd" both in the 2007 and 2009 guidelines [7].

Limits for the tool interior have been defined as process specific according to our defect evaluation. Consequently acetic acid has become a part of our AMC-monitoring for the photo zone and the tool set. Besides the control of the overall acid limit to < 5 $ppb_v$ the level of acetic acid has specific warning and specification limits as portion of the total allowed value.

Literally all acetic acid is originating from the content of PGMEA in the air of the subfab segment as well as the clean room segment (by the depicted pathways and other pathways under examination). Consequently we established limits for PGMEA in air, which are significantly below 5 $ppb_v$ measured in the stabilized situation of source control and recirculation air treatment (as described in section 2.4). In a next step we will define the limits for the maximum limits of peak excursions and maximum peak duration as measured with the fast time-resolving method as the on-line AMC monitor on the basis of PTR-MS.

## 3. CONCLUSION

We presented in this article our findings on wafer defectivity analysis and scanner lens hazing in photolithography area. We could show that in our case acetic acid was the key component of defect creation with PGMEA releases as the root cause. To our knowledge it is the first time that acetic acid is described as the originator of defectivity on wafer level and lens level.

Excursions of PGMEA level in the clean room, mainly originating from a tool compartment problem together with reactions on acid impregnated activated carbon filters have been identified as the generator for acetic acid in the air. We showed that acetic acid generation is unavoidable and ongoing on filters even after recovery of PGMEA levels to normal due to adsorb quantities on the filters. Fast and time-resolved PGMEA monitoring is capable identifying the sources and leaks to few problematic points that can be solved and controlled. Moreover, we could show that applying the new foam filter media helps avoiding problems of by product generation (acetic acid) which is typical for acid treated activated carbon filtration.

We concluded for our operation that implementing a control of the AMC levels for acetic acid and PGMEA in the photo zone is both feasible and necessary to stabilize yield in the photolithography process.

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