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Application of Hydrosol to Aerosol Based Metrology to Predict Wafer Defects from Process Chemical Contamination

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Abstract. A method for continuous measurement of particle and particle precursor material in high purity liquids using aerosolization and condensation particle courting (A+CPC) is presented. A process for correlating wafter defects with the concentration values measured using A+CPC using Sequential Spin Coating + Surface Enhanced Particle Sizing (SSC + SEPS) is described. The method was applied to monitor the rinsate of an ion exchange resin following the SEMI C93 guide. The results showed correlation between the methods and the concentration values were used to calculate a deposition factor (liquid concentration/surface concentration) on the order of 1E5 (#/ml)/(#/cm^2).

Introduction

Critical feature sizes of modern semiconductor devices have dropped below what can be measured with traditional light-scattering optical-based methods to quantify particle concentrations in process chemicals and on wafers. Without adequate metrology for *in-situ* monitoring of particle and particle precursor[1] concentrations in process chemicals, device makers face challenges correlating device defects with fabrication steps. This is especially true for chemicals used in the final steps of wafer cleaning and drying, which includes ultrapure water and isopropyl alcohol. In this work, we describe a state-of-the-art measurement method capable of detecting native particles and particles formed from dissolved contamination (referred to as particle precursors) in process chemicals down to 3 nm using Aerosolization and Condensation Particle Counting (A+CPC). We also show how this method correlates with defects found on wafers using a novel method for enhancing optical detection of particles on wafers using Surface Enhanced Particle Sizing (SEPS).

Background

Existing methods. State-of-the-art wafer cleaning tools operate with the assumption that they are supplied with process chemicals where the particle and particle precursor concentrations are below a critical threshold. Critical chemicals are typically monitored at the point of distribution using online methods including optical particle counting [1] , acoustic particle counting [2] and total organic carbon measurement [3] along with offline/grab sample methods including Inductively Coupled Plasma – Mass Spectrometry (ICP-MS). While it is generally assumed that the chemical quality level is maintained throughout the distribution system, many manufacturers and users incorporate secondary metrology and/or filtration at, or near, the point of use. Post-process wafer inspection using optical surface scanning is also used to identify critical contamination in process chemicals. These

[1] Particle precursor is defined here as dissolved/dispersed material within the process chemical that is geometrically smaller than a critical particle size; however, when combined with other precursor material forms a particle that leads to a wafer defect.

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methods for contamination monitoring have been sufficient for decades; however, semiconductor manufacturing requirements for modern devices with node sizes approaching 3 nm have surpassed the ability of existing metrology methods to detect defect-causing contamination excursions in process chemicals.

Limitations of existing methods. At the conclusion of a wet-clean process, the wafer is spun at high speeds (Spin-Drying) to remove any remaining liquid. During the Spin-Drying process, small droplets form on the wafer surface which then evaporate, leaving non-volatile material from these droplets deposited onto the surface. The deposited material could lead to a defect if the droplet contained a particle larger than a threshold tolerance or particle precursor at a sufficient concentration that precipitates as a particle, cluster, ring, or "spot". Particle precursor material may also be present as emulsified droplets or loosely formed agglomerates that results in a spatially varying concentration of that material, which can lead to a fraction of droplets with contamination concentrations higher than the bulk liquid. A graphical representation of these particle formation mechanisms is shown in Figure 1. While optical methods can detect native particles with a sufficiently high refractive index ratio and size, these methods are not able to detect particle precursor material and the minimum detected size for commercially available instruments is 20 nm. Bulk property methods like Total Organic Carbon (TOC) measurements can quantify overall chemical quality but are unable to detect medium to high molecular weight organic particles, emulsified material, and other loosely formed agglomerates. To accurately detect contamination in process chemicals that could cause wafer defects, a measurement method should account for the mechanisms of particle formation and deposition.

While wafer inspection tools might provide an indication of changes in the quality of the process chemical, it is often at the expense of contaminated production wafers. Additionally, the limit of detection for wafer state-of-the-art inspection tools (12.5 nm [4]) is not sufficient for modern devices. Physical inspection of particles on wafers is needed in parallel with online metrology to establish a correlation factor that can be used to set control limits. A method for surface particle deposition and detection at sizes below the limits of current tools is needed in parallel to improved on-line metrology.

Novel measurement method

Approach. The formation of liquid-borne surface contamination can be mimicked by dispersing the liquid into small droplets suspended in air (nebulization) and then evaporating the liquid from the droplets to form particles suspended in the gas (aerosolization). These aerosol particles can then be characterized using well-established methods for size classification and detection [5, 6]. To correlate the aerosol particle concentration with the surface particle concentration, the Spin-Drying process can be mimicked using a low-cost wafer coating device and test wafers. After spin drying, the wafers can then be analyzed using a surface inspection method that improves the detection limit by enhancing light scattering.

Instrumentation. The online measurement data presented in this work used a commercially available instrument (Kanomax Model 9010-03). The instrument consists of a proprietary Aerosolizer, which nebulizes and aerosolizes the liquid sample, and an integrated commercially available Condensation Particle Counter (Kanomax Model 3680 Scanning CPC). CPCs operate by condensing a supersaturated vapor onto optically undetectable aerosol particles [7, 8], which increases their size by several orders of magnitude allowing for detection using simple light scattering. Due to the Kelvin effect, the threshold particle diameter that will grow by condensation, D_t, is inversely proportional to the peak supersaturation, S_{max} as shown ion Equation 1.

$$S_{\max} = \frac{P_{\text{vap}}}{P_{\text{sat}}} \propto e^{\frac{1}{D_{\text{t}}}} \tag{1}$$

Where P_{vap} is the actual vapor pressure and P_{sat} is the saturated vapor pressure for the condensing vapor at a given local temperature. Supersaturation is a meta-stable state that is created by exploiting the differences between the mass diffusivity of the working vapor (n-butyl alcohol for this instrument) and the thermal diffusivity of the surrounding gas. To achieve supersaturation in a CPC using n-butyl alcohol, the gas is first saturated with n-butyl alcohol vapor at an elevated temperature (T_{sat}) and is

then directed to a conduit with wet walls at a lower temperature (T_{cond}). Because the thermal diffusivity of air is higher than the mass diffusivity of vapor, the gas will cool faster than it loses n-butyl vapor. The value for S_{max} can be varied by changing the temperature difference between the saturation and condensation regions and varying S_{max} allows for detection of particles above selected critical thresholds as represented in Figure 2 [9]. For this device, the CPC was calibrated at three threshold diameters of 3, 9, and 15 nm using purified proteins as size standards. The combination of an Aerosolization device with a Condensation Particle Counter (A+CPC) allows for Threshold Particle Counting (TPC) of native particles and particles formed from precursor material found in sample liquids.

To correlate the amount of material in the process chemical that is deposited on the wafer, a benchtop spin coating device (Laurel tech. corp. model WS-650Mz-23NPPB) and a home-built applicator and a pinch valve (CT Associates, Inc.) is used to mimic the deposition of the liquid and spin-drying of the wafer used in standard wafer cleaning tools. This deposition step is repeated several times until a steady state surface particle concentration has been reached. This deposition process is referred to as Sequential Spin Coating (SSC) [10]. The resulting particle concentration on the wafer surface is then analyzed using a prototype optical inspection tool (UNISERS) using Surface-Enhanced Particle Sizing (SEPS). SEPS technology is based on creating metallic nanoparticles with a vacuum based coating technique (PVD) on the contamination on wafer. Such metallic nanoparticles have localized surface plasmons. Incident light can couple with these localized surface plasmons at certain resonance frequency, enhancing the optical scattering cross section by orders of magnitude. This optical resonance facilitates the detection of particles down to 8nm on wafer [11, 12].

Instrument performance. The function of instrumentation is to receive an input and provide an output that correlates with a property of the input being measured. To monitor for contamination that could lead to a wafer defect, the output should correlate with changes in the concentration of particles and particle precursors in a process chemical. To validate the capability of this method to detect changes in dissolved precursor material the A+CPC instrument was challenged with potassium chloride (KCl) dissolved in Ultrapure Water (UPW) and then filtered by ultrafiltration to remove native particles. The KCl solution was then introduced to the instrument using online dilution, where the concentrated KCl was injected into the instrument feed using a peristaltic pump at flowrates varying from 10-200 μl/min into 100 ml/min of UPW and mixed using a static helical mixer. To span the dynamic range of the instrument and typical levels of particle precursors in UPW, the KCl is prepared in two concentrations, 10^2 ppb (square markers in Fig. 3 a,b) and 10^3 ppb (diamond markers in Fig. 3 a,b) resulting in a KCl concentrations of 10 to 2000 ppt. Figure 3a shows the total measured CPC aerosol concentration at > 3 nm threshold and Figure 3b shows the net CPC concentration after subtracting the background for varying concentrations of KCl. The results show that the A+CPC instrument can detect changes in particle precursor material as low as 10 ppt above background. To validate the capability of the method to detect changes in native particle concentrations, the instrument was challenged with varying concentrations of 30nm colloidal silica particles. Because the A+CPC method relies on particle counting, the response should show a linear correlation with the concentration of challenge particles in the concentration range of interest. An aerosolization rate (amount of liquid that is aerosolized and measured by the condensation particle counter) can be calculated by finding a scaling value that results in a 1:1 correlation with the challenge concentration. This aerosolization rate can then be applied to the particle concentration measured by the CPC to calculate the particle concentration in the liquid. Figure 4a shows the measured CPC concentration at a > 9 nm threshold (diamond markers from dilutions of a nominal 1 x 10^{11} particles/ml colloidal silica standard and square markers from a comparable 1 x 10^{10} particles/ml standard) and Figure 4b shows the equivalent liquid concentration corrected for the aerosolization rate, which was calculated at 1.2 μl/min.

Defect Correlation. To validate the usefulness of this method the response of the A+CPC instrument should correlate with wafer defects. To demonstrate this, the combination of A+CPC and SSC+SEPS was used to measure the rinse of ion exchange resin following SEMI C93 (Guide for Determining the Quality of Ion Exchange Resin Used in Polish Applications of Ultrapure Water System). The A+CPC

instrument was configured to continually monitor the rinsate at threshold diameters of > 3 nm and > 9 nm, where the CPC concentration was scaled using a calibrated aerosolization rate to calculate the liquid particle concentration. For SSC+SEPS analysis, periodic grab samples of the rinsate were collected and deposited onto test wafers using a pressurized high-purity liquid dispenser (Kanomax Model PV-3260) and the SSC device described above. As shown in Figure 5, both methods correlated with each other throughout the rinsing process. The concentrations measured by the A+CPC (at the > 9 nm threshold) and the SEPS were then used to calculate a deposition factor (particles detected in liquid / particles detected on the wafer), which was on the order of 1E5 (#/ml)/(#/cm^2)[2] [13, 14].

Conclusion

By combining aerosolization with subsequent particle counting, the A+CPC method can be used to quantify both particle precursors and native particles, as demonstrated through the analysis of filtered KCl standards and colloidal silica standards, respectively. Furthermore, this method has been shown to effectively mimic the deposition mechanism for particles and particle precursors across multiple size channels via the evaporation of small droplets remaining on a wafer surface after application and removal of a process chemical through comparison with on-wafer deposition quantified by SSC+SEPS. The *in-situ*, online measurement capabilities of this technique can provide more rapid feedback in comparison to other existing techniques for contamination issues as the critical defect size continues to shrink within the semiconductor industry.

Reference Figures

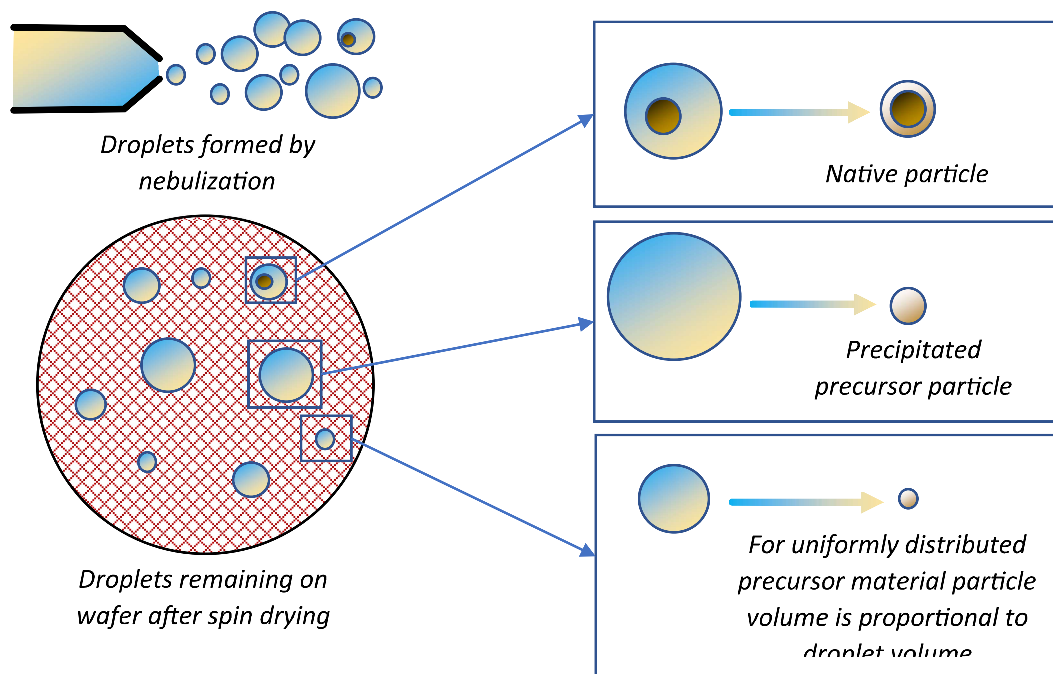

Figure 1: Graphical representation of the relationship between particles generated from droplets via nebulization and by formation on the wafer surface.

[2] To clarify reporting of in-situ versus ex-situ concentrations; ml is used for particles in liquid, ppb is used for precursor in liquid, cm^3 is for particles in gas, and cm^2 for surface defect density.

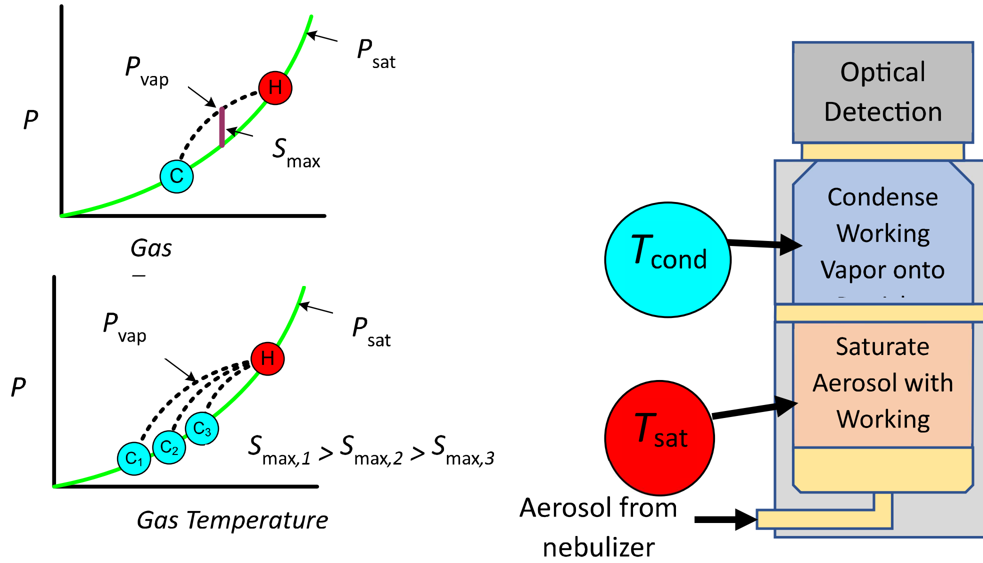

Figure 2: Overview of the principle of operation for a condensation particle counter.

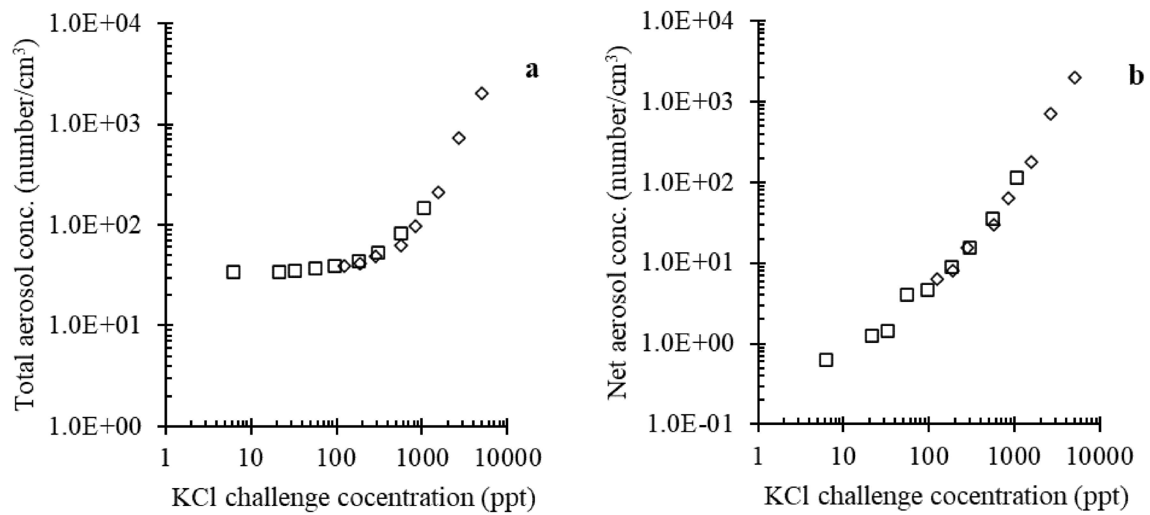

Figure 3: Measured aerosol concentration (a) and background subtracted net concentration (b) measured from different levels of online dilution of a high (diamonds) and low (squares) concentration solution of KCl.

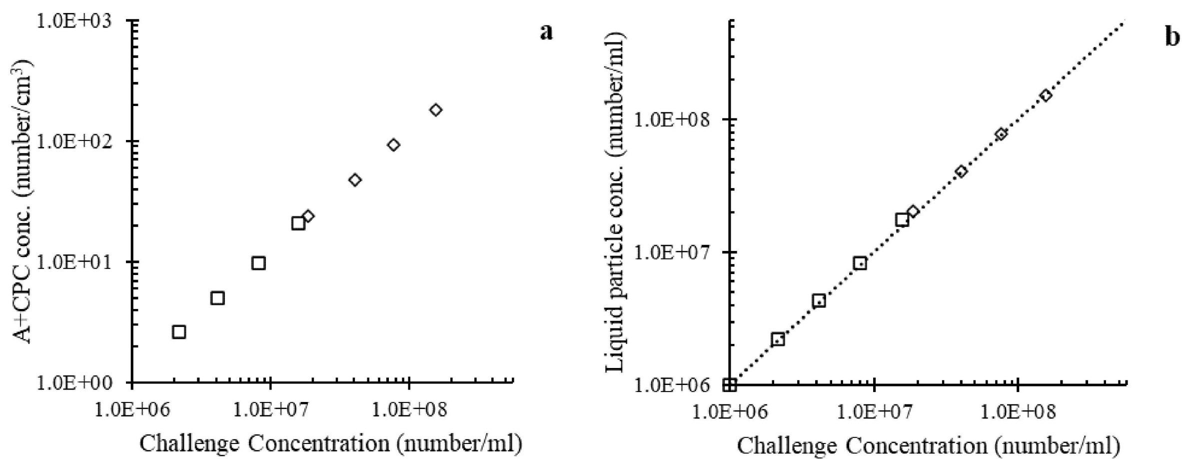

Figure 4: Measured (**a**) and scaled (**b**) colloidal silica challenge results from different levels of online dilution of high (diamonds) and low (squares) concentration standard with a 1:1 line shown on the scaled results.

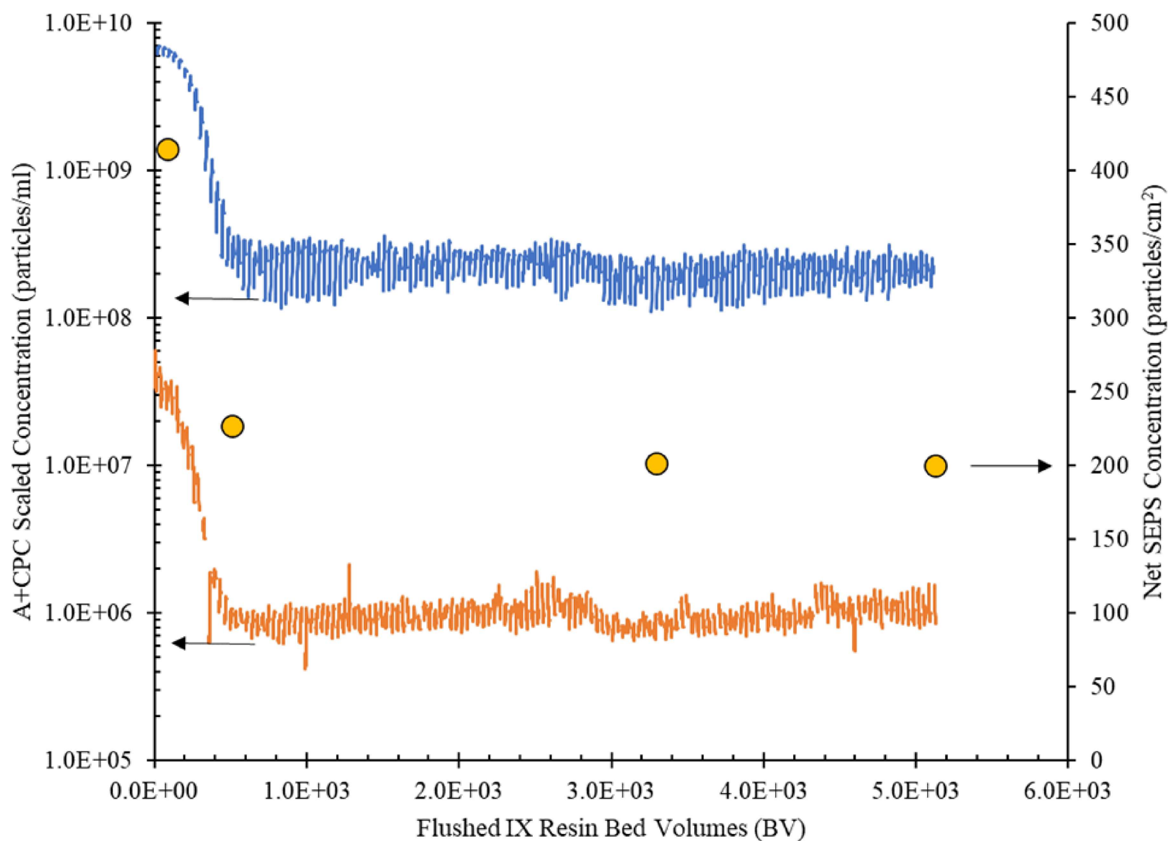

Figure 5: Rinse curve of an ion exchange resin showing corrected volume concentration measured A+CPC at 3nm threshold diameter (top trace) and 9 nm threshold diameter (bottom trace) and particle surface concentration measured using SSC + SEPS > 8 nm (circles), where Bed Volumes (BV) is the total amount of water flushed through the resin divided by the volume of the resin tank. [13]

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