



# Influence of gas type on ultrasound cleaning efficiency

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## ABSTRACT

Acoustic intensity is a very important parameter in high frequency ultrasound cleaning. The intensity variations in a cleaning bath are measured with a raster scanned hydrophone and the results are simulated through the spatial impulse response method. Furthermore, acoustic reflections on the cleaning bath walls are minimized with damping material. The influence of different type of gases (oxygen, argon, nitrogen and carbon dioxide) on the ultrasound cleaning efficiency is investigated. Gas concentrations of oxygen and carbon dioxide are directly measured. The cleaning results indicate that oxygen, argon and nitrogen give comparable cleaning results whereas particle removal with dissolved carbon dioxide gas is completely absent.

## INTRODUCTION

The manufacturing of integrated electronic circuits (IC's) involves numerous cleaning steps in which contaminants need to be removed. These contaminants are often subdivided in organic, metallic and particle contamination. Here, the removal of particulate material is considered. Due to the ever decreasing structure dimensions of IC's, the particles that need to be removed become continuously smaller. The general accepted rule is that the maximum allowed particle size should not be bigger than half of the critical structure dimension. Pure chemical cleaning, which is based on the undercutting of particles, is often used to remove particle contamination. The undercutting decreases the van der Waals interaction force between the particle and the substrate, which facilitates the removal process. In order for this cleaning method to be effective, more than two nanometer of substrate loss should be allowed [1]. However, this amount of substrate loss is not acceptable anymore according to the ITRS roadmap, because today's structure dimensions are too small. To solve this issue, an additional physical cleaning force could be applied in an attempt to lower the material loss. Several physical cleaning methods could be applied, including megasonic cleaning [2], high velocity spray aerosol [3], laser cleaning [4], solid aerosol spray [5]... All those physical cleaning methods exert an additional physical force on nanoparticles which promotes the cleaning process.

Lateral atomic force microscopy measurements are able to determine the collapse force of (semiconductor) structures and the removal force of particles [6,7]. These measurements, performed in air, have shown that a physical cleaning process window exists [7]. In principle, it should be possible to remove all nanoparticles with a size above the critical dimension without impacting fragile structures. However, physical cleaning techniques result today in a rather large distribution of physical forces. As a consequence, some of the generated physical forces are too large and they will not only remove particles but also result in damage [8]. Our goal is to optimize

the physical force distribution and, as a consequence, minimize damage creation. It is important to maintain a reasonable cleaning rate during this optimization process.

In this paper, we will focus on megasonic cleaning, which is a type of acoustic cleaning in a liquid medium. The ultrasound has typically a frequency in the range between 500 kHz and 5 MHz. There are four physical mechanisms in the megasonic cleaning process which are believed to assist in the removal of nanoparticles. First of all, there is the acoustic boundary layer streaming or Schlichting streaming. However, it is experimentally shown that Schlichting streaming alone is unable to remove small particles (< 400 nm) [9]. All other physical forces in megasonic cleaning are related to the presence of small bubbles in the cleaning liquid. These bubbles can be nucleated during the rarefaction part of the sound field. Once the bubbles are present, they will react on the sound field and oscillate. The bubble oscillation causes streaming around the bubble, which might remove nanoparticles. This liquid streaming is generally known as micro-streaming. If the bubbles have the resonant size and the acoustic power is high enough, the microbubbles can collapse near a surface which creates a water hammer pressure on that surface. This water hammer pressure is definitely large enough to remove particles but it might also be responsible for damage creation. The fourth physical force which might be present in megasonic cleaning systems is the emission of a shockwave during bubble collapse. However, this pressure wave has only been observed experimentally in ultrasonic systems. The presence of these shockwaves is currently unclear in higher frequency acoustic cleaning systems.

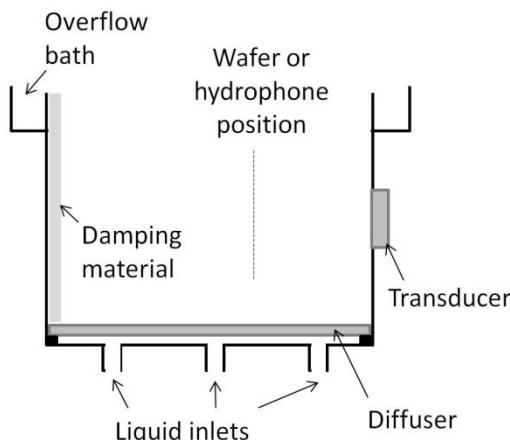
Since it is known that the removal of nanoparticles is associated with the presence of bubbles, the gas concentration and type of gas are very important parameters in this cleaning process [10]. Nevertheless, only very little experiments are known which investigate the influence of the type of gas on cleaning performance [11]. In order to optimize the physical force distribution in megasonic systems, it is important to

control and optimize all parameters that are involved in the cleaning process. These variables include temperature, type and amount of dissolved gas, acoustic field distribution, type of liquid, use of surfactants, frequency of the acoustic field...

## EXPERIMENTAL SETUP

### Megasonic cleaning setup

The megasonic cleaning setup consists of a large bath with inner dimensions of 40 by 40 by 40 cm<sup>3</sup>. The total tank volume is 64 liter, which can be refreshed within two minutes. The liquid temperature is approximately 20.5°C and the large liquid rate minimizes temperature variations during the application of an acoustic field. The transducer mounted at the side of the tank consists of three piezo elements and the active area is ~60 cm<sup>2</sup>. Acoustic reflections can have a large impact on the sound field and, as a result, on the cleaning efficiency [12]. The tank walls and the edges of the transducer are covered with damping material, commercially known as Aptflex F28, which minimizes acoustic reflections. A diffuser is placed at the bottom of the tank in order to optimize the liquid flow in the tank. An overview of the system is given in Figure 1.



**Figure 1** Schematic presentation of the megasonic cleaning bath

Deionized water (DIW) is used plant-wide with guaranteed low concentrations of dissolved O<sub>2</sub> and CO<sub>2</sub> (< 5 ppb), and some uncontrolled concentration of dissolved N<sub>2</sub> from the water storage buffer tanks. This DIW is send through three parallel membrane contractors connected to a vacuum line. This procedure removes all residual gases from the DIW cleaning liquid to a level below our detection limit. Next, three parallel placed liquid membrane contractors are integrated in the gasification system. The contractors are connected to a pressurized gas line (O<sub>2</sub>, N<sub>2</sub>, CO<sub>2</sub> or Ar) which allows us to accurately gasify the cleaning liquid. Oxygen concentrations are measured with a PreSens oxygen sensor. The working mechanism is based on the quenching of sonoluminescence caused by the collision between molecular oxygen and luminescent dye molecules in the excited state. CO<sub>2</sub> concentrations are determined by conductivity measurements [13].

### Particle contamination procedure

First, Si wafers (200 mm) are cleaned with an O<sub>3</sub>-last clean. Next, the wafers are contaminated with monodisperse 78 nm SiO<sub>2</sub> particles using a spin contamination technique. This results in a high amount of particles (~10<sup>6</sup>/cm<sup>2</sup>) on the wafer surface. The wafers are aged for 2 hours in a relative humidity of 40 percent before cleaning in the megasonic bath. A

constant aging time is preferred since strong particle adhesion can be caused by chemical reactions (after initial hydrogen bonding) that take place in presence of moisture and long aging times [14,15]. Each wafer clean is followed by a 1 min spin dry process. Cleaning performance is evaluated by measuring local PRE using light scattering in the haze mode [16,17]. This process performance is quantified with particle removal efficiency  $\eta$  expressed in percentage and is defined as

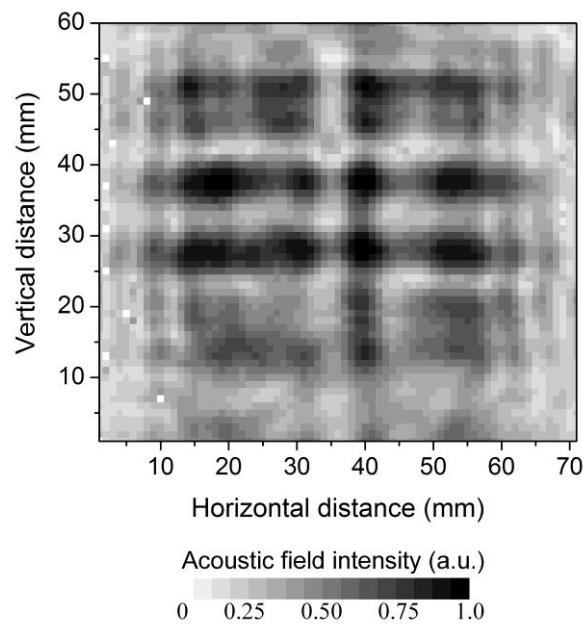
$$\eta = \frac{\sigma_o - \sigma}{\sigma_o} \cdot 100$$

where  $\sigma_o$  is the initial surface concentration after the contamination procedure and  $\sigma$  is the surface concentration after the cleaning procedure.

## RESULTS

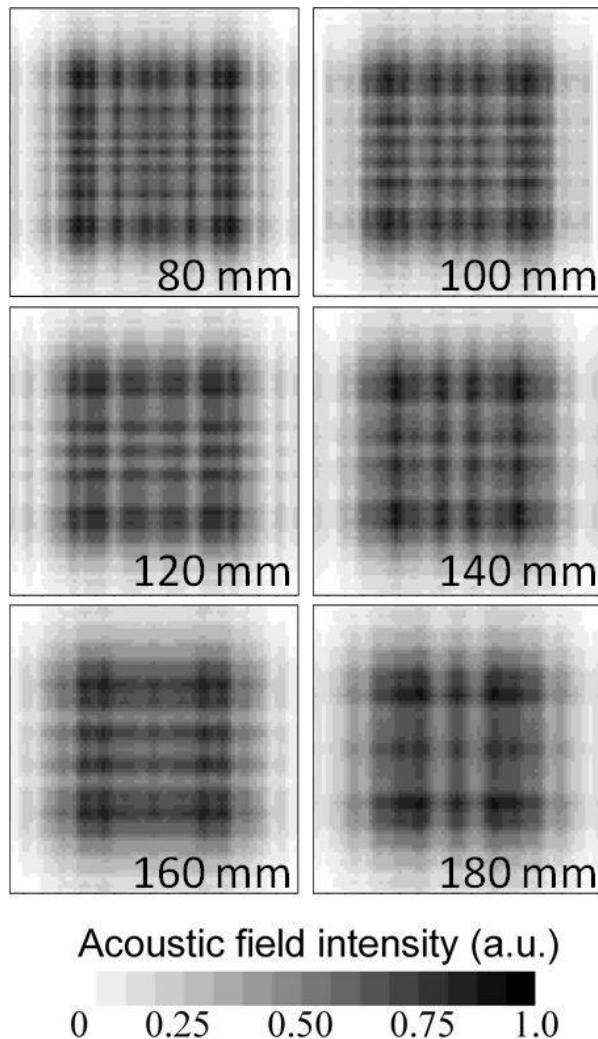
### Acoustic field measurements and simulations

The acoustic field has been measured with a needle hydrophone (Onda HNR-500) with an aperture diameter of 500 μm. The hydrophone was scanned with an Aerotech XYZ positioning system which is controlled with a labview program. The scan area is 60 by 70 mm<sup>2</sup> and the hydrophone is raster scanned in steps of 1 mm. The acoustic field intensity variations are shown in Figure 2.



**Figure 2** Acoustic field intensity variations in front of the transducer at a distance of approximately 145 mm.

The sound field distribution was also calculated with the field II program [18,19]. The simulation is done through the spatial impulse response model. This response gives the emitted ultrasound field at a specific point in space as a function of time, when the transducer is excited by a Dirac delta function. The acoustic field is then calculated by convolving the spatial impulse response with the excitation function of the transducer. The focus of the transducer in the simulations was put to infinity. Figure 3 show the simulated acoustic intensity variations at a transducer-hydrophone distance of 80, 100, 120, 140, 160 and 180 mm. The simulated area is 80 by 80 mm<sup>2</sup> and the simulation is performed in steps of 1 mm. It is clear from the acoustic measurements and simulations that



**Figure 3** Simulated acoustic field intensity variations in front of the transducer at a distance of 80, 100, 120, 140, 160 and 180 mm.

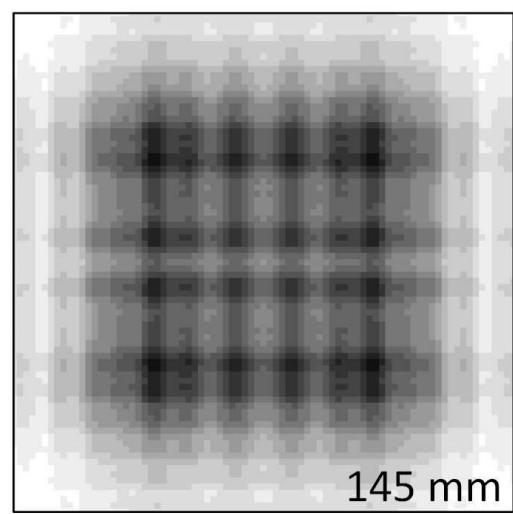
a large variation of the sound field is present, which is typical for the near-field or Fresnel zone of the transducer [20].

Figure 4 shows the simulated acoustic field intensity at 145 mm. The main features in the simulations correspond reasonably well with the hydrophone measurements shown in Figure 2. In the simulations as well as in the experiments, four intensity peaks can be observed around the center of the image and also several peaks are present in the outer regions.

#### Influence of type of dissolved gas

In the particle removal experiments, the water was fully saturated with argon, nitrogen, oxygen or carbon dioxide. A small amount of visible bubbles were present in the connection pipes between the gasification system and the megasonic cleaning bath. These bubbles are blocked by the diffuser and transported along the walls of the tank to the surface of the tank. The saturated condition is confirmed with oxygen concentration and conductivity measurements in the case of oxygen and carbon dioxide, respectively. The total cleaning time in the particle removal tests is 4 minutes.

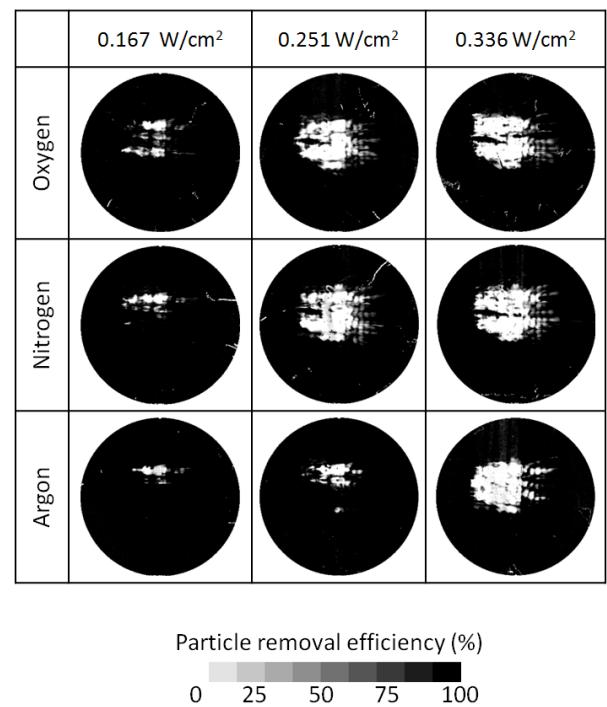
The measurements showed that it was impossible to observe any particle removal when DIW was saturated with carbon



dioxide. An applied electric power of  $2.5 \text{ W/cm}^2$  in saturated

**Figure 4** Calculated acoustic field distribution at 145 mm from the transducer. The main features correspond to the hydrophone measurements.

$\text{CO}_2$  water was not enough to get any cleaning. Even at this high power, no bubbles could be visually observed in the cleaning liquid. This remarkable result can be linked to the experiments of Kumari et al. [21]. It was shown that Ar,  $\text{O}_2$  and  $\text{N}_2$  are capable of generating a sonoluminescence signal, while sonoluminescence was completely absent in  $\text{CO}_2$  gasified water. It was also shown that  $\text{CO}_2$  is an inhibitor of sonoluminescence when the cleaning liquid is gasified with sufficient amounts of oxygen gas.



**Figure 5** Particle removal efficiency maps for oxygen, nitrogen and argon saturated water as a function of power density.

Figure 5 shows the particle removal efficiency maps of oxygen, nitrogen and argon saturated DIW. It can be seen that even at very low power densities of  $0.167 \text{ W/cm}^2$ , which corresponds to a total power of 10 W, megasonic cleaning is feasible. The particle removal efficiency is averaged over an area of 60 by  $60 \text{ cm}^2$ , where the acoustic field is maximized and the averaged results are summarized in Table 1. It can be seen that oxygen, nitrogen and argon are quite comparable in removal efficiencies. The differences which are present might be removed by optimizing gas content for each gas individually.

**Table 1.** Averaged particle removal efficiencies [%]

Power (W/cm <sup>2</sup> )	0.167	0.251	0.336
Oxygen	16.5	65.5	72.5
Nitrogen	21.6	71.5	66.7
Argon	7.7	16.9	69.5

## CONCLUSIONS

The acoustic field of a transducer with an active area of  $\sim 60 \text{ cm}^2$  has been evaluated with hydrophone measurements and simulated with the spatial impulse response model. The data and simulations clearly show near-field acoustic maxima, which influence the uniformity of megasonic cleaning. It was shown that DIW saturated with carbon dioxide gas results in no particle removal, while DIW with saturated argon, nitrogen and oxygen gas can clean already at low powers and their particle removal efficiencies are comparable.

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## REFERENCES

- 1 T. Hattori, *Ultraclean Surface Processing of Silicon Wafers* (Springer-Verlag Berlin Heidelberg New-York, 1998), pp. 453–457
- 2 V. Kapila, P.A. Deymier, H. Shende, V. Pandit, S. Raghavan and F.O. Eschbach, “Megasonic Cleaning, Cavitation, and Substrate Damage: An Atomistic Approach”, *Proceedings of SPIE* **6283**, pp. 628324-1–628324-12 (2006)
- 3 M.T. Andreas, K. Wostyn, M. Wada, T. Janssens, K. Kenis, T. Bearda and P.W. Mertens, “High Velocity Aerosol Cleaning with Organic Solvents: Particle Removal and Substrate Damage”, *Sol. Stat. Phenom.* **145-146**, pp. 39–42 (2009)
- 4 T.G. Park, A.A. Bushnaina, J.M. Lee, S.Y. You, “Substrate damage-free Laser Shock Cleaning of Particles” *ECS Proc.* **26**, pp. 190–194 (2004)
- 5 G.G. Totir, M.M. Frank, R. Vos, S. Arnauts, T. Bearda, K. Kenis, M. Delande, Q.T. Le, E. Kesters, G. Vereecke, G. Mannaert, M. Lux, I. Hoflijk, T. Conard, S. Banerjee, S. Malhouitre, P. Leunissen and P.W. Mertens, “Post Ion Implant Photoresist Removal via Wet Chemical Cleans Combined with Physical Force Pretreatments”, *ECS Trans.* **11**, pp. 219–226 (2007)
- 6 D. Peter, F. Holsteys, M. Dalmer, H. Kruwinus, A. Lechner and W. Bensch, “Collapse Mechanism for High Aspect Ratio Structures with Application to Clean Processing”, *ECS Trans.* **25**, pp. 241–248 (2009)
- 7 T.-G. Kim, K. Wostyn, T. Bearda, J.-G. Park, P. Mertens and M.M. Heyns, “Investigation of Physical Cleaning Process Window by Atomic Force Microscope”, *ECS Trans.* **25**, pp. 203–210 (2009)
- 8 P. Mertens, “Acoustic cleaning in nano-electronics”, *Proceedings of the 155<sup>th</sup> ASA conference, Paris*, pp. 555–560 (2008)
- 9 S. Brems, M. Hauptmann, E. Camerotto, X. Xu, S. De Gendt, M. Heyns and P. Mertens, “The influence of the Angle of Incidence in Megasonic Cleaning”, *Proceedings of UCPSS* (to be published).
- 10 G. Vereecke, R. Vos, F. Holsteys, M.O. Schmidt, M. Baeyens, S. Gomme, J. Snow, V. Coenen, P.W. Mertens, T. Bauer and M.M. Heyns, “Influence of Hardware and Chemistry on the Removal of Nano-Particles in a Megasonic Cleaning Tank”, *ECS Trans.* **92**, pp. 143–146 (2003)
- 11 C. Franklin, “Impact of ionization potentials and megasonic dispersion”, *Sol. Stat. Phenom.* **145-146**, pp. 19–22 (2009)
- 12 S. Brems, M. Hauptmann, E. Camerotto, A. Pacco, S. Halder, A. Zijlstra, G. Doumen, T. Bearda and P.W. Mertens, “Impact of Acoustical Reflections on Megasonic Cleaning Performance”, *ECS Trans.* **25**, pp. 287–294 (2009)
- 13 S. Light, B. Kingman and A.C. Bevilacqua, *209<sup>th</sup> American Chemical Society National Meeting*, Anaheim, CA, April 2–6 (1995)
- 14 T.-G. Kim, Y.-S. Yoo, S.-H. Lee and J.-G. Park, “Effects of size, humidity, and aging on particle removal from Si wafers”, *Microelectron. Eng.* **86**, pp. 145–149 (2009)
- 15 A.A. Busnaina, H. Lin, N. Moumen, J.-W. Feng, and J. Taylor, “Particle Adhesion and Removal Mechanisms in Post-CMP Cleaning Processes”, *IEEE Trans. On Semicon. Manuf.*, **15**, pp. 374–382 (2002)
- 16 K. Xu, R. Vos, G. Vereecke, M. Lux, W. Fyen, F. Holsteys, K. Kenis, P. Mertens, M. Heyns and C. Vinckier, “Relationship between Particle Density and Haze on a Wafer: a New Approach to Measuring Nano-Sized Particles”, *Sol. Stat. Phenom.* **92**, pp. 161–164 (2003)
- 17 F. Holsteys, J. Roels, K. Kenis, Q. Le and P.W. Mertens, “Monitoring and Qualification Using Comprehensive Surface Haze Information”, *IEEE Inter. Symp. on Semicon. Manuf. Conf. Proc.*, pp. 378–381 (2003)
- 18 J.A. Jensen and N.B. Svendsen, “Calculation of pressure fields from arbitrarily shaped, apodized, and excited ultrasound transducers”, *IEEE Trans. Ultrason., Ferroelec., Freq. Contr.* **39**, 262–267 (1992)
- 19 J.A. Jenson, “Field: A program for simulating ultrasound systems”, *Med. Biol. Eng. Comp.* **4**, 351–353 (1996)
- 20 L. Medina, E. Moreno, G. González and L. Leija, “Circular ultrasonic transducer characterization: theoretical and experimental results”, *Rev. Mex. Fís.* **49**, pp. 511–518 (2003)
- 21 S. Kumari, M. Keswani, S. Singh, M. Beck, E. Liebscher, P. Deymier and S. Raghavan, “Control of Sonoluminescence Signal in DI Water Using Carbon Dioxide”, *Sematech Surface Preparation and Cleaning Conference (SPCC)*, Austin , Texas (2010)