The suitability of ultrasonic and megasonic cleaning of nanoscale patterns in ammonia hydroxide solutions for particle removal and feature damage

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Abstract
In this study, usually, arrays of square or rectangular piezo devices bonded to a substrate and spaced as close together as possible for transduction for efficient megasonic cleaning, megasonic cleaning is applied to removing sidewall particles during post-silicon etch cleaning. A system was developed containing different measures of ultrasonic and megasonic processes, generated by adjusting the power in NH4OH. Megasonic waves in the gap between the wafer and the megasonic transducer ensure uniform sonic energy across the whole wafer. The generation of radicals is promoted by megasonic energy, such that residue removal occurs in dilute solution. The feature damage for the megasonic cleaning decreased in the NH4OH solution compared to ultrasonic. Moreover, the mechanical force of the bubble cavitation generated by megasonic cleaning also improves residue removal and enhances the mass transfer rate. Extreme cavitation is beneficial for removing particles but can damage wafer features. The damage caused by ultrasonic cleaning is significantly larger than megasonic cleaning. Compared with typical methods of silicon oxide residues removal, this megasonic process has a low material loss and a high residue removal efficiency for via, with a high aspect ratio. In addition, it minimizes defects to structures. These results suggest that megasonic cleaning can be applied to the nanoparticle cleaning process. Megasonic cleaning is quickly becoming an efficient method for post process cleaning in IC fabrication processes.

Keywords: megasonic clean, TSV clean, blind hole clean

(Some figures may appear in colour only in the online journal)

1. Introduction
Particulate residue is a major concern in IC, both owing to yield loss and owing to the failure to control defects; this limits product performance for the manufacturing of semiconductors [1]. However, through chemical solutions aided by sound fields (in the MHz range), particle removal can generally be achieved [2]. Megasonic cleaning is a gentler cleaning mechanism than acoustic streaming or acoustic cavitation [3, 4] and is broadly used for wafer surface cleaning during device fabrication, usually arrays of square or rectangular piezo devices bonded to a substrate and spaced as close together as possible for transduction for efficient megasonic cleaning [5]. Processes such as front-end-of-line, back-end-of-line, and high particle removal efficiency (PRE) can be achieved [6, 7]. Recently, horizontally-single or stacked Gate-All-Around (GAA) Nanowire/Nanosheet...
structures have been investigated intensively, owing to their excellent short-channel control and electrostatics [8, 9], which are effective for sub 5 nm technology nodes [10]. Si, GeSn, InGaAs, and SiGe stacked channel Field-effect transistors (FETs) have been studied previously [11], and pure-Ge stacked nanosheet FETs have been successfully demonstrated for the first time, through the assistance of megasonic agitation [12].

Cavitation involves oscillations lasting several acoustic cycles, and features implosive releases of energy that ultimately lead to bubble collapse [3]. Cavitation intensity is considered to be a crucial index [13, 14] and a significant indicator of feature damage on the wafer’s surface [15, 16]. Extreme cavitation is beneficial for removing particles but can damage wafer features [17, 18]. The particles are removed from the patterned wafer surface owing to stable cavitation and microstreaming, by means of the acoustic streaming of oscillating bubbles [19]. Maintaining stable cavitation and smaller nodes while keeping a high removal efficiency and avoiding damage to the wafer surface is the main challenge for megasonic cleaning [20, 21].

Particle removal has been proposed using many kinds of forces, including microcavitation [22], acoustic pressure gradients [23], and acoustic streaming [24] as induced by megasonic waves. This streaming is deemed to be the active process in the megasonic cleaning of substrates [18]. Owing to its importance in surface cleaning, the shear stress appears to near-wall cavitation through both transient and steady impinging jets on solid walls [25, 26]. This study aims to better determine the beneficial effects of megasonic cleaning. Here, a megasonic waveguide, assembled with a megasonic transducer, was fabricated into the megasonic system of the power supplier. To determine the megasonic cleaning performance, the acoustic pressure was measured, and comparisons were made between both megasonic and ultrasonic techniques.

2. Experimental details

Figure 1(a) shows a schematic drawing of the megasonic cleaning process featured in this study, and also shows the wafer’s location. Megasonic cleaning was performed on both sides of the wafer, with full power on the backside and attenuated power on the frontside of a single wafer cleaning plate. The spin cleaning tool fitted with a radial transducer array for megasonic wet cleaning of single wafers. The megasonic transducer (area 323 mm) is designed to apply uniform acoustic energy to a spin substrate (typically 0–50 rpm) at a frequency 0.98 MHz. The spin chuck is designed to hold 200 mm wafers. The size of silicon oxide particles, determined using dynamic light scattering, was found to be 250 ± 50 nm. All experiments were performed at the Taiwan Semiconductor Research Institute (TSRI) in a Class 100 Micro/Nano fabrication center. Both patterned and blanket samples were exposed to megasonic agitation in a wafer spin cleaning assembly on 200 mm wafers. A uniform acoustic energy was applied, consisting of a full wafer megasonic plate at a frequency of 0.98 MHz. Ultrasonic cleaning at a nominal frequency of 80 kHz is investigated experimentally. Point-of-use dilution was used to ensure precise chemical delivery, improve process repeatability, and ensure fresh chemistry for every wafer. Dual-side cleaning was performed, with different frontside and backside chemistry capabilities. Airflow was optimized for low particles. A system containing different measures of ultrasonic and megasonic processes, generated by adjusting the power by NH4OH, was developed.

Figure 2 shows images of the cleaning results at different residue concentrations, at an acoustic power density of 70 W. A solution of NH4OH with a pH of 8.2 was examined for both (a) megasonic cleaning after 30 s, and (b) ultrasonic cleaning after 30 s, using an optical microscope. Cleaning was deemed to be
sufficient where plenty of areas covering an indicative site on the wafer had been destroyed. Contamination sites were identified through small particles. For the megasonic cleaning, the PRE was performed to determine the cleaning capability. Firstly, a large number of particles over 1 μm were counted and placed onto an eight-inch wafer. The wafer was then placed into the transducer equipment, which was erected through megasonic agitation. The wafer was then cleaned of particles to avoid contamination, using a supplied power of 70 W. The solution was spread across the wafer at a flow rate of ∼5.5 to 6 μm min⁻¹; the sample was then put into a particle counter to count the number of remaining particles after cleaning.

3. Results and discussion

The results for both the megasonic and ultrasonic experiments are shown in figures 2(a) and (b). Roughly 10,000 particles were counted after ultrasonic cleaning, compared to 753 particles after megasonic cleaning. The cleaning efficiency of
the developed megasonic cleaning system was therefore calculated to be 97%. Figure 3 shows a plot of the acoustic power density compared to the damage density occurring in NH₄OH at pH 8.2. Strong damage was observed at sites following increases in the power of the solution, clearly demonstrating that the megasonic system resulted in significantly lower damage than the ultrasonic system. Figure 3 shows the damage sites on a patterned fragment after cleaning in NH₄OH. The defect density was enlarged in NH₄OH at pH 8.2 following an increase in power from 20 to 70 W. The feature damage for the megasonic cleaning decreased in the NH₄OH solution compared to that in ultrasonic and megasonic cleaning exposure times of 30 s. Table 1 appears to show that the contamination on patterned wafer was not the dominant defect, and accounted for the defect results on both the megasonic and ultrasonic samples cleaned in NH₄OH at pH 8.2.

Figure 4 shows the PRE function of the power input in SC1. The cleaning efficiency of the megasonic system was higher than in the ultrasonic system. This would be expected, as the ratio of removal force decreases as the particle size decreases, therefore removal efficiency decreases with decreasing particle size. Figure 4 shows the PRE increased as the power increased; for each particle size the maximum removal efficiency was obtained at 70 W. The increase in removal efficiency in response to increasing power was likely due to the resulting increase in streaming velocity. Streaming velocity was proportional to power in PRE results on a SiO₂ blanket using NH₄OH. However, a cleaning time of 30 s in NH₄OH at pH 8.2 achieved a PRE of nearly 98% on blanket SiO₂.

Figure 5(a) shows Scanning Electron Microscope (SEM) images of patterned samples taken before megasonic cleaning. Before the megasonic cleaning, the polymer residues can clearly be observed on the sidewall of the SiO₂. Figure 5(b) shows the same samples after cleaning, demonstrating good results for residue removal following megasonic cleaning and immersion in NH₄OH. Full wafer tests were performed on 200 mm wafers, resulting in removal of the entire residue as well as the particles. To extend processes for nanotechnology process flows in order to obtain results of megasonic agitation. Megasonic help remove residues from device wafers in several process flows that evaluated the ability of megasonic agitation. For interconnects, processing with physical agitation appears to be preferential, providing that it is possible to limit the amount of etching required for residue removal, such that the dielectric is not damaged. Figure 5(b) shows that enhancing the process can achieve the desired result, i.e. that megasonic agitation can assist in removing the residue.

Figure 6 shows a comparative study of these cleaning methods. Each wafer was scanned before and after the cleaning process. Switching from plasma etch conditioning to megasonic conditioning on the surface effectively reduced the amount of debris, and number of scratches; the number of defects also decreased slightly. Megasonic cleaning therefore appears to reduce both scratches and defects when replacing the standard conditioner. In the sidewall and via bottom, post-ultrasonic cleaning resulted in heavy debris, which can adhere onto the wafer surface. The contamination was completely removed from the sidewalls and bottom of the via and the surface of the wafer, after megasonic cleaning. The contamination was still clearly visible on the sidewall of the metal gate etch after ultrasonic cleaning. Megasonic cleaning is less damaging than ultra-sonic cleaning, however, figure 7 shows that megasonic cleaning can clean well in the
nanoparticle cleaning process. Therefore, more residues will stick to a surface following ultrasonic cleaning, relative to megasonic cleaning. In addition, the megasonic cleaning across the via effectively removed particles from the wafer’s surface. Figure 8(a) shows that the residue was still clearly visible on the bottom of via etch after ultrasonic cleaning. Ultrasonic cleaning resulted in heavy residues in the sidewall and via bottom which can adhere onto the wafer surface. Figure 8(b) shows the contamination was completely removed from the sidewalls and bottom of the via etch after megasonic cleaning.

Figure 9 shows the removal of residue after four megasonic cleaning processes of four different immersion times, 0, 15, 30, and 60 s, at 70 W. The SEM images show varying numbers of defects across the different times. The sizes of the residue sites were found to be in the range of ~200 nm in the NH₄OH solution. The post-etch residue was not removed by ultrasonic cleaning but was by megasonic cleaning. After megasonic cleaning for 60 s, the residue had been removed from the sidewall of the metal lines. The effects of megasonic agitation were also applied to solution for PR removal particles. The use of megasonic agitation can also advantageous in preventing deposition. In addition, megasonic agitation is not only appropriate for reducing defects, but can also increase PR without the need for excessively strong forces, which could otherwise lead to structural damage. Therefore, Megasonic cleaning is less damaging than ultra-sonic cleaning.

Figure 10 shows that megasonic cleaning is four times faster than ultrasonic cleaning. Megasonic cleaning of patterned wafers was both efficient at removing particles and resulted in a reduction of damage. Figure 10 reveals that ultrasonic cleaning showed higher numbers of defect adders than megasonic cleaning. The ultrasonic cleaning required to enhance removal rates was higher than that of megasonic cleaning. Therefore, ultrasonic cleaning needs to work completely to clean off the particles. Consequently, the megasonic cleaning performed in SC1 could be adopted as a committed, step-critical process to clean defects.

Figure 11 shows the effects of acoustic streaming and cavitation upon a nearby wafer feature during the acoustic irradiation of liquids. When bubbles oscillate, microstreaming forces that aid in particle removal can be generated. Figure 11 shows a schematic drawing of a substrate feature of length L, height H, and thickness W, that experiences shockwaves generated by a megasonic transducer. The bubble collapse of the maximum velocity of the liquid (V) and the pressure wave (Press Shock) generated by the shock wave, m, is 1500 m s⁻¹ if the liquid is water, where ρ is the density of the liquid. The force creates a shear fracture along the plane LW, and the shock wave works in the plane of area HL. Maximum liquid velocities have been reported in the range 10–50 m s⁻¹ after
bubble collapse, for an initial bubble radius in the range of 0.25–2.5 μm in water, at pressure amplitudes of 3–5 atm. The pressure generated by a shockwave, taking a minimum value of 10 m s\(^{-1}\) for the liquid velocity, can be computed as follows:

\[
\text{Press Shock} = \rho V = 1000 \times 1500 \times 10 = 0.015 \text{ GPa}
\]

\[
\text{Force Shock} = \text{Force Shear} = \frac{(\text{Shock Press})(HL)}{(\text{Shear Strength})(LW)}
\]

\[
\text{Force Shock} = \text{Force Shear} = \left(\frac{2 \text{ cm}}{500 \mu m}\right) \left(\frac{0.015}{0.1}\right) = 6,
\]

where \(L\) and \(H\) are both 2 cm in the substrate, \(W\) is 500 μm in the substrate, and the shear strength is 0.1 GPa. In addition, the force shock can exceed the shear strength force in the structures.

Figure 12 shows that horizontally three stacked pure-Ge nanosheet GAA FETs. In this device process, we intentionally
grow large mismatch Ge/Si multilayers rather than Ge/GeSi multilayers as the starting material, because the large difference of material properties between Ge/Si is beneficial to the selective etching process. In order to avoid island growth, the flat Ge/Si multilayers are grown at a low temperature. Due to the excellent selective etching, the shape of Ge nanosheets almost keeps unchanged after etching. Additionally, we found the dislocations in suspended Ge sheets are more easily to remove than the case that Ge layers are still tied with Si layers. Regarding selective etching, this study demonstrated that at a suitable temperature, and with the assistance of megasonic agitation, the Si layers can be easily etched away over Ge layers with good selectivity, using a Tetramethylammonium hydroxide (TMAH) solution. Three-step B implantations were sequentially performed with doses and energies of $2 \times 10^{15}$ cm$^{-2}$ and 30 KeV, $1 \times 10^{15}$ cm$^{-2}$ and 20 KeV, and $1 \times 10^{15}$ cm$^{-2}$ and 10 KeV, respectively, for S/D p-type uniform doping. B activation achieved by RTA at 550 °C for 60 s. A schematic of the whole process is shown in figure 12(a). In order to achieve a good selective etching between Ge/Si, the solution must be kept at a temperature of $\sim$60 °C and must be assisted by megasonic agitation. The stacked Ge nanosheets have a length of 1.0 μm; their appearance following the etching away of the Si interlayers is shown in figure 12(b) The remaining Ge sheets appear to be perfect, as shown by the measured $I_D-V_G$ curves for a three-stacked Ge nanosheet P-FET and a single Ge nanosheet P-FET with a short channel of 180 nm. The three sheets device has an $I_{on}$ of 1030 μA μm$^{-1}$ at $V_D = V_G = 1.0$ V.

4. Conclusions

In this paper, megasonic cleaning was shown to reduce feature damage while providing high PRE in ammonium hydroxide
Figure 12. (a) Stacked Ge nanosheet structures formation by megasonic-agitation-assisted TMAH selective etching at 60 °C. (b) The SEM tilting view images for the stacked Ge nanosheets long structure of 1.0 μm and $L_d$–$V_g$ curves for a three stacked Ge nanosheet and single nanosheet.
solutions. The mechanical force of the bubble cavitation generated by megasonic cleaning also enhances the mass transfer rate and improves residue removal. Compared with typical methods of silicon oxide residues removal, this megasonic process has a high residue removal efficiency and a low material loss for via, with a high aspect ratio. In addition, the results of a PRE test in a NH₂OH solution was close to 98% for blanket SiO₂. Extreme cavitation is helpful for removing particles but can damage wafer features. The damage caused by megasonic cleaning is significantly lower than for the ultrasonic cleaning system. Finally, a pure-Ge stacked nanosheet FETs was successfully demonstrated through megasonic cleaning over Ge using TMAH at 60 °C with megasonic agitation. These results suggest that megasonic cleaning can be applied to the nanoparticle cleaning process. Megasonic cleaning is quickly becoming an efficient method for post process cleaning in IC fabrication processes, and there is literature available.

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