MICRO
ADVANCED PROCESS CONTROL, DEFECT REDUCTION, AND YIELD ENHANCEMENT FOR SEMICONDUCTOR MANUFACTURING

Using cost-effective dilute-acid chemicals to perform postetch interconnect cleans

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Tests demonstrate the efficacy of using an optimized POU dilute-acid solution in a batch spray tool to remove residues from M1 and M2 lines, vias, and bond pads.

W*hile leading-edge logic chip* manufacturers have made the conversion to copper conductors and low-k dielectrics for advanced devices at the 90-nm technology node, aluminum and deposited silicon oxide continue to be used for noncritical layers and in memory and other logic devices.[1] An important step in the fabrication of copper and aluminum interconnect structures is postetch residue removal. Anisotropic plasma etching of photoresist-patterned aluminum leaves a mixture of organic polymer and back-sputtered metal oxides on the sidewalls of the remaining aluminum lines.[2–5] Subsequent removal of the photoresist mask by oxygen plasma or ozone ashing leaves additional residues and affects the composition and adherence of the etch residues.[6] To prevent particle defects, electrical leakage, and subsequent corrosion, these residues must be removed from the patterned aluminum lines before deposition of the next dielectric layer.

Anisotropic plasma etching of the dielectric layer to form vias also leaves etch residues on the bottom, sidewalls, and top edge of the pattern.[5–8] The dielectric layer is commonly formed by depositing silicon oxide using tetraethoxysilane (TEOS) as a precursor.[9,10] Residues formed during via etch are different from those formed during aluminum line etch because of the nature of the material being etched and the plasma

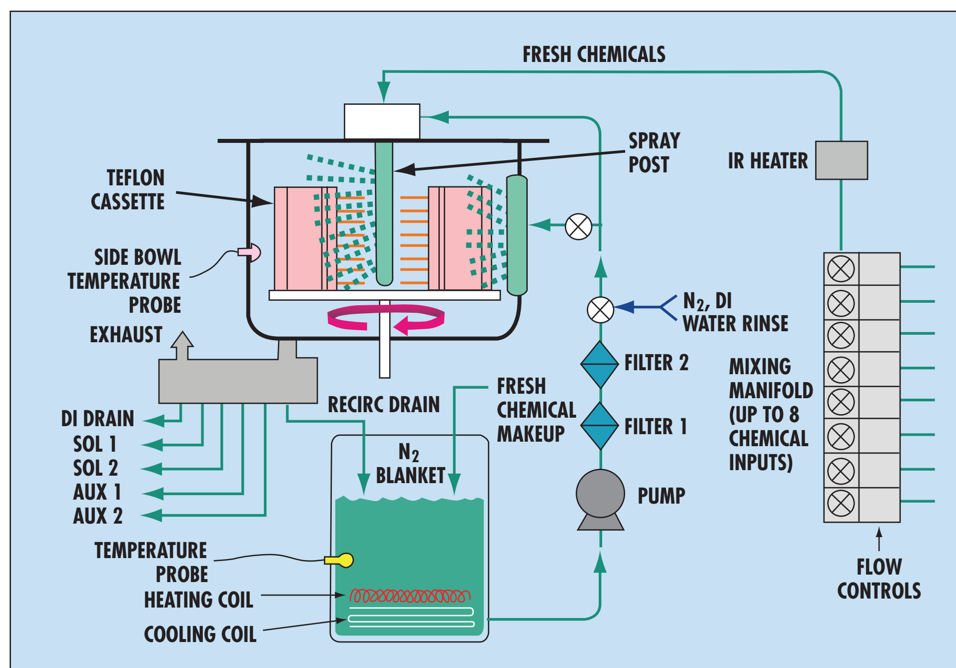

Figure 1: Schematic diagram of a 200-mm batch spray cleaning tool.

chemistry. In addition, etch residues are formed during the patterning of the final aluminum bond-pad contact through the top dielectric layer, which is a combination of TEOS-deposited silicon oxide and plasma-deposited silicon nitride.

To remove these residues effectively, careful consideration must be given to the etching and ashing processes that form the residues and the materials that are patterned. Differences in the composition of metal layers can affect the nature of the etch residue and the removal process. This article focuses on the use of dilute-acid chemicals for removing residues from postetch interconnects in logic devices.

Chemical Solutions Used to Remove Postetch Residues

To prevent subsequent corrosion, postetch residue removal is performed to remove all "visible" residues from the sidewalls and surfaces of the patterned features and "molecular" contaminants such as chlorine. The cleaning process must also minimize etching of the desired aluminum line and underlying dielectric in the case of M1 and M2 and minimize lateral etching of the final dielectric in the case of bond-pad contacts. In addition, postetch cleaning processes must avoid attacking the tungsten studs that are deposited into the vias to electrically connect M1 and M2 layers, since these studs can be exposed at the edge of the aluminum line.[11] Finally, it is always desirable to carry out the cleaning process in the most economical way possible by maximizing equipment throughput while lowering chemical consumption and costs.

For many years, the predominant method for removing postetch residue from aluminum lines and from the vias formed in TEOS-deposited oxide has been the use of amine-based chemistries in immersion baths or wafer-surface sprays.[12] Many variations of amine-based chemistries are available from different chemical suppliers. The most common ones include a hydroxylamine (HDA) as the main active component. These chemistries also include chelating agents, surfactants, and other solvents.

While HDA-based chemical solutions are reliable and offer a relatively large process window, they are expensive and require special waste treatment. These solutions usually must be collected and returned to the manufacturer for disposal. In addition, HDA-based solutions produce a pH spike during the initial water-rinsing steps, etching and pitting exposed aluminum. To counteract this phenomenon, an intermediate rinsing step using isopropyl alcohol (IPA) or a buffered solution is required before final water rinsing. Finally, HDA-based solutions require elevated temperatures (60–70°C) and long exposure times (10–20 minutes) to achieve effective residue removal.

A few years ago, semiaqueous chemistries (SACs) were introduced. Marketed by several chemical manufacturers, SACs are usually fluorine-based solutions that include ammonium fluoride or other fluoride species to attack and remove

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residues.[11,13] These solutions are also composed of a variety of chelating agents and surfactants. Because SAC solutions do not produce a pH spike during rinsing, they can be rinsed directly with water. In addition, they can be formulated to work more quickly than HDA-based chemistries. SAC solutions can be used very effectively in batch spray systems.[14]

More recently, dilute acid formulations have been used increasingly for postetch cleaning of aluminum lines and silicon oxide vias. The most common formulations include dilute sulfuric acid (H_2SO_4) and dilute hydrogen peroxide (H_2O_2) in the concentration range of 1–15% by weight.

Acid cleaning solutions have been used for years in IC manufacturing. Relatively concentrated solutions of ammonium

hydroxide (NH_4OH) and H_2O_2 (SC-1) and solutions composed of hydrochloric acid (HCl) and H_2O_2 (SC-2) were first introduced to the industry in 1970.[15] Concentrated mixtures of H_2SO_4 and H_2O_2 (SPM) were also commonly used for removing photoresist and organic contaminants. In addition, more-dilute mixtures of strong acids and H_2O_2 have commonly been used in metal finishing and passivation.

Levels	Time (sec)	Temp (°C)	HF (ppm)	H_2O_2 (wt %)	H_2SO_4 (wt %)
M1, M2, Vias	60–150	20–35	0–150	2–20	5–15
Bond Pads	420–450	20–35	0–150	2–4	2–15

Table I: Dilute-acid-mixture ranges and conditions.

Dilute-acid mixtures have also been used in IC cleaning applications for several years.[16,17] Starting in 1996, dilute H_2SO_4 and H_2O_2 and very dilute HF acid were used.[18] Premixed versions of these solutions, which include chelating agents and surfactants, have been used for postetch cleaning of aluminum lines and silicon oxide vias.[19–22] While chemical manufacturers supply rather expensive premixed versions of these solutions, dilute-acid chemistries can be accurately mixed and diluted at the point of use (POU) using readily available and relatively inexpensive concentrated solutions.[22] Using POU-mixed dilute-acid solutions in a batch spray tool provides an effective and low-cost way to clean aluminum lines, vias, and bond pads.

Test Equipment and Processes

The dilute-acid cleaning tests discussed in this article were performed using 200- and 300-mm wafers processed in two different Zeta spray cleaning systems from FSI International (Chaska, MN). A schematic diagram of the 200-mm system is illustrated in Figure 1. The 200-mm system can hold four 25-wafer PFA cassettes, while the 300-mm system can hold two 25-wafer cassettes. In both cases, the wafers are held horizontally in the cassettes. The cassettes rest on a PFA turntable that can be rotated at speeds up to 500 rpm. The cassettes and turntable are held in a nitrogen purged chamber with a sealed lid.

Chemicals, rinsewater, and nitrogen are dispensed from a central spray post that extends from the chamber lid. Fluids can also be dispensed from a side spray post mounted in the wall of the chamber. Chemicals are mixed and diluted using a mixing manifold. Mixture ratios are set by the process recipe and are controlled by precision flow controllers. An infrared heater can be used to control the temperature of the solution before it is dispensed into the chamber. The temperature of the solution as it flows from the wafer surface can be monitored using a temperature probe that is mounted in the chamber wall. Temperature measurement can be used to optimize the process or, in the case of precision etching applications, to control the etch time to achieve a desired etch amount.

Using POU-mixed dilute-acid solutions in a batch spray tool provides an effective and low-cost way to clean aluminum lines, vias, and bond pads.

The work described here employed dilute mixtures of H_2SO_4, H_2O_2, and HF to remove etch residues from aluminum lines (M1 and M2), oxide vias, and open bond pads.

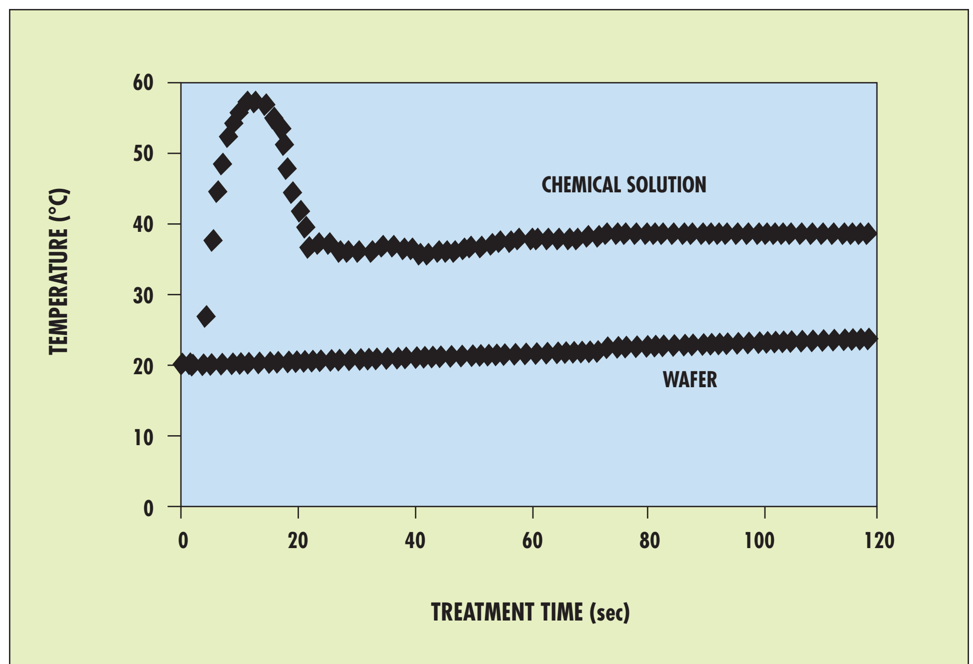

Figure 2: Chemical solution temperature and wafer temperature (measured when the fluid left the wafer surface) as a function of processing time. The as-mixed dilute solution was composed of H_2SO_4, H_2O_2, and HF.

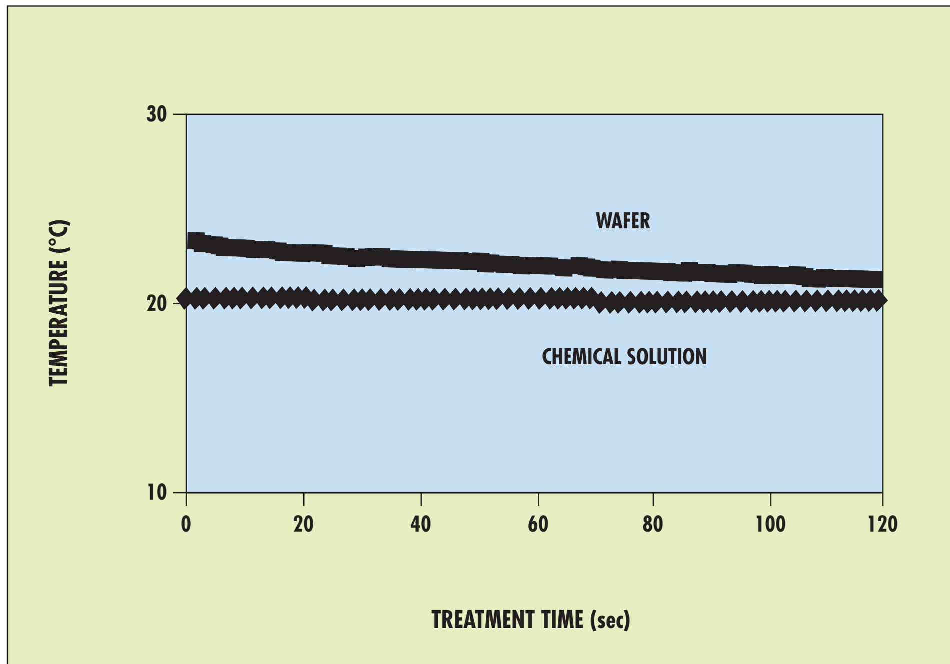

Figure 3: Chemical solution temperature and wafer temperature (measured when the fluid left the wafer surface) as a function of processing time. The chemical was a precooled dilute solution of H_2SO_4, H_2O_2, and HF.

In all cases, good results were obtained without active temperature control. Chemical solutions typically reached about 35°C after mixing. Cooling occurred during atomizing dispense into the chamber, and the chemical solution's on-wafer temperature rose slowly from room temperature to about 25°C during a 3-minute treatment. For M1 and M2 lines, precooling of the blended solution to 25°C before wafer processing was also tested using a run tank. Table I lists the dispense times, solution temperatures, and chemical concentration ranges that were explored in this work.

The M1 metal stack included a bottom layer of Ti/TiN under the aluminum-copper-silicon alloy and a cap layer composed of Ti/TiN. The M2 metal stack, while similar, contained a cap layer that was composed solely of TiN. Both metal layers were patterned on top of a 7000-Å silicon oxide layer that had been deposited in a plasma CVD tool using a TEOS precursor. Vias were etched in the silicon oxide. The aluminum bond pads, which consisted of a metal stack that was very similar to the M2 stack, were exposed during plasma etching through the final passivation layer of 14,000 Å of plasma-deposited nitride on top of 12,000 Å of plasma-deposited TEOS silicon oxide. After undergoing patterning, dry etching, and ashing, the wafers were cleaned with a dilute-acid solution.

Controlling Process Variables

Liquid Flow. To achieve proper residue removal, chemical flow must be controlled. The total flow rate was held in the range of 2000–3000 ml/min to provide proper spray atomization and uniform chemical coverage over the wafer surface. Individual chemical flows were controlled at flow rates as low as 20 ml/min for prediluted HF to as high as 250 ml/min for H_2O_2. To ensure proper dilution, DI-water flow was typically controlled at approximately 1750 ml/min but was adjusted to keep the total flow rate in the desired range of 2000–3000 ml/min. The wide variety of chemical solutions used in this work was achieved using the mixing manifold.

Liquid Temperature. Temperature is another variable for controlling residue removal and material loss in the dilute-acid process. Because of the exothermic nature of mixtures containing H_2SO_4 and DI water, solution temperature increases from ambient to nearly 35°C depending on the concentration of the solution. When dispensed on the wafer, solutions with temperatures in that range cause the wafer temperature to rise from 20° to 25°C, as shown in Figure 2.

Good results were obtained in this work without resorting

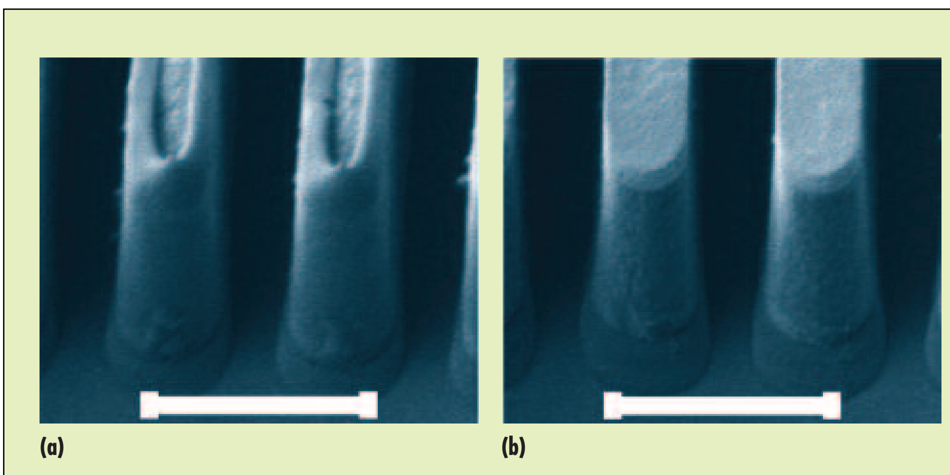

Figure 4: SEMs of patterned M1 aluminum lines before and after residue was removed using an optimized dilute-acid solution of H_2SO_4, H_2O_2, and HF at an as-mixed solution temperature of approximately 35°C and a processing time of 120 seconds. The white bars represent a length of 1 μm.

to active temperature control. However, if close temperature control is desired to expand the process window, the dilute-acid solution is first sent to a run tank where the temperature is adjusted and controlled using a heating element and/or a chiller coil located inside the tank. The run tank holds approximately 20 L of solution and contains a recirculation loop in which the mixture can flow continuously over a filter while its temperature is maintained at the desired setpoint until needed by the process.

The use of dilute solutions allows cost-effective single-pass processing in which the chemical solution flows to a waste drain after being sprayed onto the wafers. After each process run, the run tank is refilled with POU-blended chemical solution and heated or cooled as required to reach the desired setpoint temperature. Figure 3 presents a case in which the mixed acid solution was precooled to 20°C before it was dispensed onto the wafers. The wafer temperature started at about 22°C and decreased to about 21°C during processing.

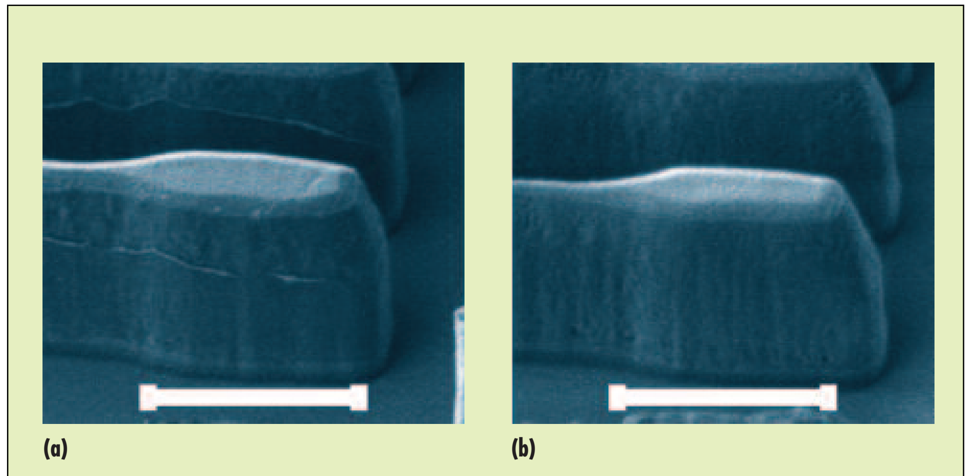

Figure 5: SEMs of patterned M2 aluminum lines before and after residue was removed using an optimized dilute-acid solution of H_2SO_4, H_2O_2, and HF at an as-mixed solution temperature of approximately 35°C and a processing time of 90 seconds. The white bars represent a length of 1 µm.

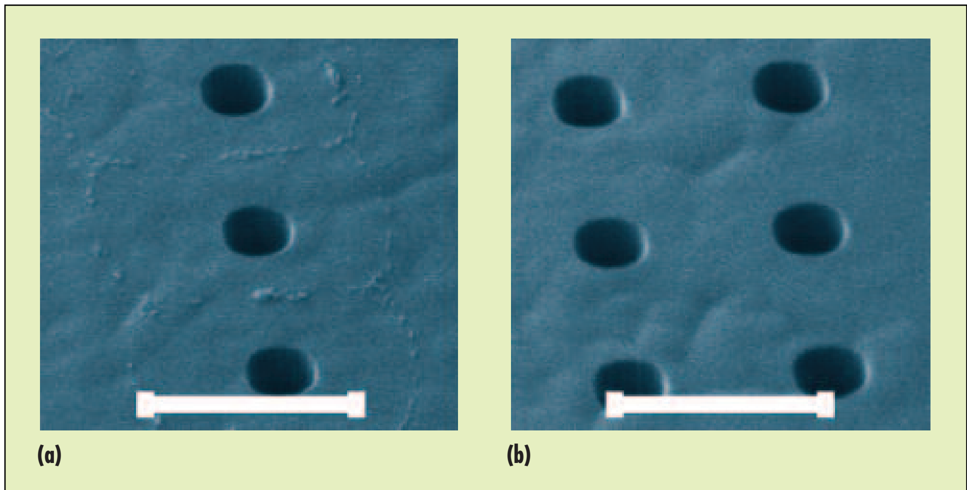

Figure 6: SEMs of patterned vias before and after residue was removed using an optimized dilute-acid solution of H_2SO_4, H_2O_2, and HF at an as-mixed solution temperature of approximately 35°C and a processing time of 150 seconds. The white bars represent a length of 1 µm.

Process Window. The range of parameters listed in Table I was investigated to determine both the size of the process window and the optimal process conditions. A wide process window not only protects against parameter fluctuations during the cleaning process but also guards against fluctuations in upstream processes that affect the amount of residue or the difficulty in removing it. In this work, the process window was determined by the need to remove etch residues completely while preventing the metal lines and pads from being attacked and corroded and the oxide dielectric from being etched. (Residue removal rates were measured both visually and electrically.)

In addition, it is desirable for the process to be selective enough to allow repeat cleaning. Occasionally, wafers must be re-ashed if initial ashing is incomplete, a reprocessing step that also includes a postash residue-removal step. Therefore, process selectivity must allow for a second cleaning sequence while ensuring that neither the metal lines, the pads, nor the oxide dielectric are damaged.

Results and Discussion

M1 and M2 Lines. Etched and ashed M1 and M2 wafers were first processed at the as-mixed solution temperature of ~35°C and with a fixed H_2SO_4 concentration, an HF concentration ranging from 50 to 150 ppm, and an H_2O_2 concentration ranging from 2 to 20%. Processing time was 120 seconds. Representative scanning electron micrographs (SEMs) of M1 lines before and after residue removal are shown in Figure 4. The test results show that residues can be removed without causing corrosion or significant line loss. However, at the highest HF concentration, the metal lines were attacked somewhat. The process window for H_2O_2 concentration appears to be very wide, since concentration levels ranging from 2 to 20% resulted in acceptable residue removal without causing pitting or corrosion. By optimizing the

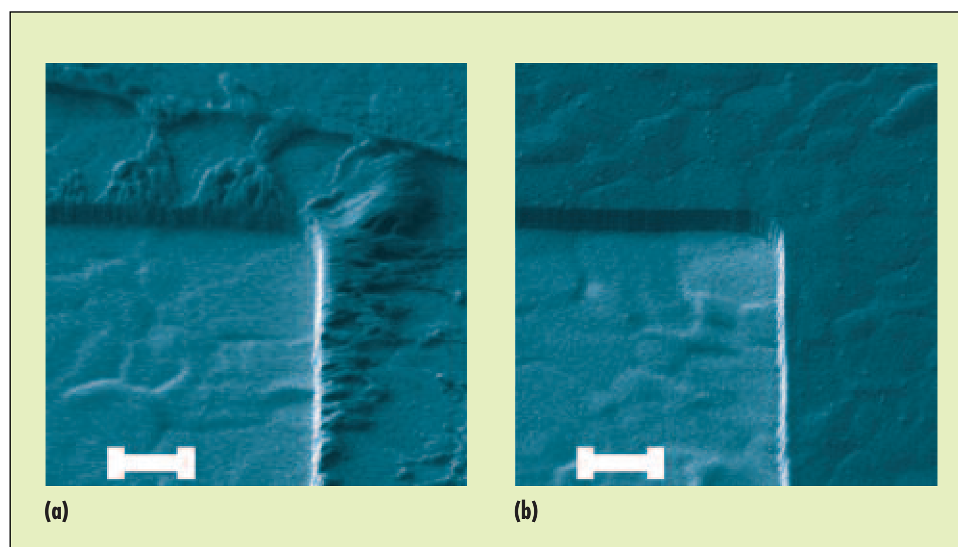

Figure 7: SEMs of patterned bond pads before and after residue was removed using a reoptimized dilute-acid solution at an as-mixed solution temperature of 35°C and a processing time of 450 seconds. The white bars represent a length of 1 µm.

Performance Parameter	Four-Chamber Single-Wafer Tool	Semiautomated Batch Spray Tool	Automated Batch Spray Tool
Batch size (wafers/run)	1	100	100
Throughput (wafers/hour)	130	250	250
Footprint (m^2)	4.6	3.1	6.3
Total chemical usage (ml/wafer)	127	5.1	5.1
Typical chemical cost ($/wafer)	0.46	0.02	0.02

Table II: Performance comparison between 200-mm single-wafer and batch tools used to clean M1 lines with an optimized dilute-acid solution composed of H_2SO_4, H_2O_2, and HF. Processing time was 60 seconds for the single-wafer tool and 120 seconds for the batch spray tool. Total chemical use includes the combined volumes of 96% H_2SO_4, 31% H_2O_2, and 100:1 HF.

Performance Parameter	Four-Chamber Single-Wafer Tool	Automated Batch Spray Tool
Batch size (wafers/run)	1	50
Throughput (wafers/hour)	130	160
Footprint (m^2)	11.6	6.3
Total chemical usage (ml/wafer)	127	10.2
Typical chemical cost ($/wafer)	0.46	0.04

Table III: Performance comparison between 300-mm single-wafer and batch tools used to clean M1 lines with an optimized dilute-acid solution composed of H_2SO_4, H_2O_2, and HF. Processing time was 60 seconds for the single-wafer tool and 120 seconds for the batch spray tool. Total chemical use includes the combined volumes of 96% H_2SO_4, 31% H_2O_2, and 100:1 HF.

dilute H_2SO_4/H_2O_2/HF solution, processing time for M1 lines can be extended to 150 seconds.

Figure 5 presents SEM images of M2 lines before and after being processed in the same optimized solution as the M1 lines at the as-mixed temperature of ~35°C and a processing time of 90 seconds. As the processing time exceeded 90 seconds, however, the M2 lines began to suffer from pitting. When the chemical solution was cooled in the run tank to 25°C before wafer processing, cleaning time could be extended to 120 seconds. When it was cooled to 20°C, the cleaning time could be extended to 150 seconds. While dilute solutions of H_2SO_4 and H_2O_2 without HF did not etch or corrode the metal lines, they were also unable to remove the etch residue.

Vias. Postetch vias were cleaned using the same optimized solution as in the M1 tests and at the same as-mixed temperature of ~35°C. Processing time was varied from 60 to 150 seconds. The tests found that a processing time of at least 120 seconds was required to remove all visible via residues. Processing time could be extended to 150 seconds without etching the oxide measurably and increasing the via hole diameter. Representative SEM images of vias before and after cleaning are shown in Figure 6.

Bond Pads. Cleaning the etch residues at the bond-pad level is more difficult than at the metal or via levels because the conditions for etching the thick nitride passivation layer produce an etch residue that takes longer to remove completely. When the solution concentrations that were successful for metal lines and vias

were tested for the bond pad, treatment times greater than 180 seconds were required for complete residue removal, but that processing time caused the bond pad at the edges of the passivation layer to be attacked. By reoptimizing the solution concentration, damage to the bond pad was avoided, but a minimum treatment time of 420 seconds was required to achieve complete residue removal. Figure 7 shows typical bond-pad cleaning results using the reoptimized solution with a treatment time of 450 seconds at the solution's as-mixed temperature of ~35°C.

Chemical Costs and Use

While the cost of using HDA solutions in typical 200-mm wet benches or batch spray tools to remove residues from interconnects can exceed $1 per wafer, even when the chemical is recirculated, the use of POU-blended dilute acids is relatively inexpensive. Table II compares the performance of a four-chamber single-wafer tool, a semiautomated batch spray tool, and an automated batch spray tool using an optimized dilute-acid process to clean M1 lines on 200-mm wafers. The table also compares total chemical costs. Although such costs vary from region to region, they can be as low as 2 cents per wafer when used in a 200-mm batch spray system. In contrast, a four-chamber single-wafer tool consumes 10–20 times more chemical per wafer than a batch spray system, amounting to 46 cents per wafer. Also, a batch spray system has overall higher throughput than a single-wafer tool.

While the cost of using HDA solutions can exceed $1 per wafer, the use of POU-blended dilute acids is relatively inexpensive.

Table III compares the performance of a four-chamber single-wafer tool and an automated batch spray system using an optimized dilute-acid process to clean M1 lines on 300-mm wafers. The batch spray tool running a POU-blended dilute-acid process is more effective than the single-wafer tool.

Conclusion

Postetch cleaning of aluminum lines and silicon oxide vias has traditionally been performed using proprietary solvent mixtures containing amines, chelating agents, surfactants, and other components. These mixtures, which generally contain HDA, are designed to achieve effective postetch residue removal without excessive etching of the silicon oxide dielectric and without attacking or subsequently corroding the aluminum. However, the supply, cost, and residual defect issues associated with HDA have prompted IC manufacturers to search for alternative cleaning chemistries.

Concentrated and dilute-acid mixtures have been used for decades to clean silicon wafers during IC manufacturing when only silicon and silicon oxide are exposed. However, concentrated acids have not typically been used when aluminum and other metals are exposed because of the risk of damage to the metal. In contrast, dilute-acid mixtures have been employed successfully to achieve effective etch-residue removal in aluminum lines and silicon oxide vias. Typically containing sulfuric acid, hydrogen peroxide, and hydrofluoric acid, these chemicals are readily available in fabs and can be easily blended and diluted at the point of use. POU-blended dilute-acid chemical solutions can remove residues at the M1, M2, and via levels in as little as 2 minutes. Aluminum bond pads can be cleaned with a dilute solution of sulfuric acid and hydrogen peroxide.

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