

Oxygen-Plasma Cleaning of Hybrid Integrated Circuits

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The immediate need to eliminate the use of ozone-depleting chlorofluorocarbons (CFCs) is the driving force behind efforts to develop new methods for cleaning hybrid integrated circuits (HICs) without using CFC-113 based solvents. Oxygen-plasma cleaning is a cost-effective method for replacing processes that use these solvents. The performance of oxygen-plasma cleaning has improved over the years, but it has never worked successfully as a method for cleaning high-voltage circuits before they are encapsulated. However, results from a fractional-factorial designed experiment showed that an improved oxygen-plasma cleaning process can achieve cleanliness levels that equal or surpass those using a CFC-113 based solvent. This paper describes how oxygen-plasma substitution for a CFC-113 based solvent was developed and implemented at AT&T's Merrimack Valley Works.

Introduction

Since the mid-1960s, the electronics industry used CFC-113 based solvent degreasing to clean hybrid integrated circuits (HICs). (See Panel 1 for a list of abbreviations, acronyms, and terms.) Today, faced with evidence that CFCs are a major factor in depleting the earth's ozone layer, engineers are developing new processes that eliminate the use of CFCs.

The standard pre-encapsulation cleaning process, known as Method VI, is used to clean HICs. Liquid and vapor degreasing in a CFC-113 based solvent is followed by immersing the HICs in a heated hydrogen peroxide bath, rinsing them in de-ionized (DI) water, and then drying them. The degreasing step dissolves organic contaminants (e.g., fingerprints and trapped photoresist) from circuits. Hydrogen peroxide and water rinsing oxidize and remove other ionic contaminants, such as sodium and chlorine. Then, dielectric or encapsulating materials, including room temperature vulcanizing (RTV) rubber and photodefinable polymer, are applied. Residues that remain during encapsulation or other coating processes can create current leakage paths, which compromise reliability, especially in the presence of high temperature, humidity, and bias (THB) voltage.

Because CFC-113 based solvents are

no longer an option, we use screening procedures to evaluate whether a particular replacement process can ensure a residue-free current path without leakage. One effective screening method measures leakage current between biased conductors at elevated temperature and humidity. Known as a double track, this simple test vehicle consists of a pair of conductors that loop back and forth, covering a large area of the circuit.

While many reliable CFC-113 replacements exist that comply with environmental and safety regulations, oxygen plasma may be a more cost-effective cleaning method because associated raw material and waste disposal costs are low. This paper describes how the results of fractional-factorial designed experiments reduced the leakage in circuits cleaned with oxygen plasma.

Oxygen-Plasma Cleaning

Oxygen-plasma cleaning takes place in a continuously evacuated processing chamber backfilled with oxygen gas. A radio-frequency (RF)-driven set of electrodes generates a glow discharge, converting the input gas to monatomic oxygen and oxygen radicals. These react with organic contaminants to form low levels of carbon monoxide, carbon dioxide, and water vapor. The byproducts are then

removed from the chamber through the pumping system. This process generates only low levels of exhaust gas and requires no costly waste handling or disposal.

Many phases of integrated circuit fabrication use plasma etching extensively to remove numerous materials, including metals, metal oxides, and polymers. Figure 1a shows the configuration described above, referred to as a capacitively coupled plasma system because it resembles a parallel-plate capacitor.

Figure 1b shows the plasma being generated in an inductively coupled system. This type of system is commonly used to strip photoresist or remove photoresist residues. Oxygen plasma also effectively removes bleed-out (epoxy resin that migrates away from device sites) and organic residues from die-attach assembly operations. However, radiation from the plasma in parallel-plate or inductive systems causes catastrophic breakdown of silicon devices, rendering this technique useless for pre-encapsulation cleaning.

Placing the circuit boards between the plasma region and the vacuum system can prevent damage to silicon devices. This configuration, known as downstream plasma, removes the circuits from the effects of the energetic plasma, but not from the reactive species themselves. In a variation of this concept, shown in Figure 1c, the plasma is placed between a powered cage and the chamber wall. Although downstream plasma is effective in eliminating damage to silicon devices, the rate of organic-residue film removal drops off dramatically, causing a corresponding drop in productivity.

Nevertheless, the development of downstream plasma systems has spurred a more comprehensive set of applications, including pre-encapsulation cleaning. In their paper, Cook and Hannon¹ described the feasibility of replacing Freon®-TMS degreasing followed by hydrogen peroxide as a pre-encapsulation cleaning method with downstream oxygen plasma. (Freon is a registered trademark of E. I. Du Pont de Nemours and Company.) Performing downstream plasma cleaning before a circuit is encapsulated improves leakage current results on triple-track circuits tested at 85°C, 85-percent relative humidity, and 180 volts (V) bias. However, when we applied the same cleaning approach to high-voltage circuits, we found that raising the bias voltage to 1000V produced a high failure rate for plasma-cleaned circuits, but not for circuits cleaned in a CFC-113 based solvent and hydrogen peroxide.

Panel 1. Abbreviations, Acronyms, and Terms

CFC	— chlorofluorocarbon
DI	— de-ionized
HIC	— hybrid integrated circuit
PTFE	— polytetrafluoroethylene
RF	— radio frequency
RTV	— room temperature vulcanizing
THB	— high temperature, humidity, and bias

Despite repeatedly disappointing results on high-voltage reliability test vehicles, efforts to improve plasma cleaning continued, using photoresist removal measurements to gauge performance. Fortunately, the design of downstream plasma chambers has improved over the years. Recent work shows that small additions of a dopant gas enhance removal rates and uniformity significantly. Thus, the downstream plasma-cleaning process required extensive recharacterization and optimization before it could be considered a possible replacement for a CFC-113 based solvent. However, poor high-voltage THB performance persisted, even after removal rates and uniformity were significantly improved by optimizing power, pressure, etch time, and dopant levels, possibly because:

- Little or no correlation exists between photoresist removal rates and circuit performance during high-voltage THB testing.
- We did not select settings or parameters that were sensitive enough to correlate photoresist removal to pre-encapsulation cleaning.
- We may not have properly selected important factors in plasma cleaning or other related processing that affect leakage. Later in this paper, we will discuss a factor that has a significant effect on THB leakage current.

Reliability Testing

As circuit density increases, the allowable concentration of contaminants decreases, making them more difficult to detect. While many analytical tools such as Auger electron spectroscopy, ultraviolet (UV)-fluorescence, electron spectroscopy for chemical analysis, contact angle measurement, and ionography are useful for evaluating overall circuit cleanliness, none of these can reliably and quickly scan large circuit areas for localized contamination. Consequently, we must use a

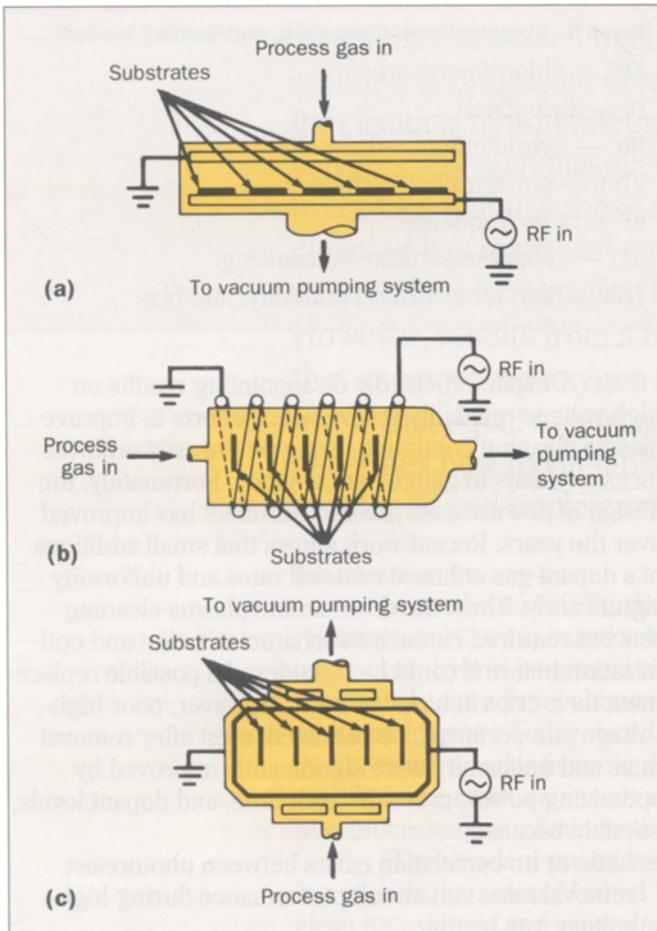


Figure 1. (a) A capacitively coupled plasma system. A regulated flow of oxygen gas is introduced to the evacuated chamber and ionized between a grounded and RF-driven electrode. The ionized oxygen reacts with organic contaminants on the substrate surfaces, forming volatile byproducts that are removed by the vacuum pump. (b) An inductively coupled plasma system. Oxygen gas introduced into the evacuated chamber is ionized inductively by an RF-driven coil, reacts with organic contaminants, and is removed by the vacuum pump. (c) Downstream plasma, a variation in which the circuit boards are placed away from the RF field, which is confined between a powered cage and the chamber wall. Oxygen gas introduced into the evacuated chamber is ionized between an RF-driven cage and the chamber walls. The ionized gas reacts with organic contaminants on the substrates in the absence of RF radiation, preventing silicon device damage.

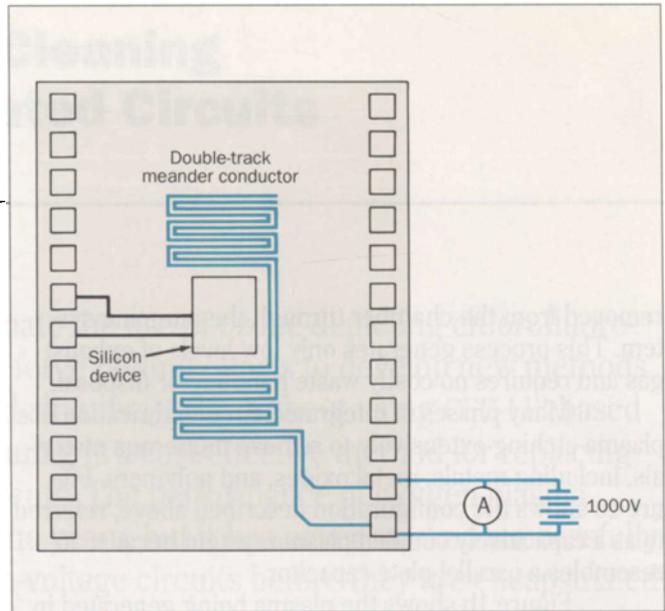


Figure 2. A double-track meander conductor patterned on ceramic using Ti-Pd-Cu-Ni-Au metallization. This circuit also features a dummy silicon device to simulate the solder attachment operation during typical HIC assembly processing.

more sensitive test to measure contamination levels. THB testing is sensitive over a range of contamination levels, and it can detect contaminants over a large circuit area.

During a THB test, we measure leakage current between oppositely biased adjacent conductors in an environmental chamber at higher temperature and humidity, typically 85°C and 85-percent relative humidity. Bias voltage can range from as little as 1V or 2V to as much as 1000V, depending on the circuit type being tested. Although we can use THB tests on production circuits, the most efficient way to evaluate new processing methods is to measure leakage current on specially designed test vehicles. The test results can be related to circuit life by introducing acceleration factors.² Acceleration factors are a function of track length, spacing, temperature, humidity, and bias voltage. These parameters are usually selected to simulate up to a 40-year life during a 1000-hour test.

Test Vehicles

Typical test vehicles for THB testing are double- or triple-track conductors patterned on ceramic. They are designed to detect small, isolated concentrations of contaminants over a large circuit area. These contaminants are produced during circuit fabrication by photoresist processing, wet chemical operations, and handling.

For the fractional-factorial designed experiment, we patterned a double-track meander conductor on ceramic using titanium-palladium-copper-nickel-gold (Ti-Pd-Cu-Ni-Au) metallization. This circuit also featured bond pads for solder attachment of a silicon device (see

Table I. Experimental Design Matrix

Run	Pressure (torr)	Power (watts)	Dopant (percent)	Time (minutes)	DI rinse method	Fixture material
1	0.5	100	2.5	5	Immersion	Aluminum
2	0.5	100	2.5	5	Spin	PTFE
3	0.5	100	7.5	20	Immersion	Aluminum
4	0.5	100	7.5	20	Spin	PTFE
5	0.5	450	2.5	20	Immersion	PTFE
6	0.5	450	2.5	20	Spin	Aluminum
7	0.5	450	7.5	5	Immersion	PTFE
8	0.5	450	7.5	5	Spin	Aluminum
9	2.0	100	2.5	20	Immersion	PTFE
10	2.0	100	2.5	20	Spin	Aluminum
11	2.0	100	7.5	5	Immersion	PTFE
12	2.0	100	7.5	5	Spin	Aluminum
13	2.0	450	2.5	5	Immersion	Aluminum
14	2.0	450	2.5	5	Spin	PTFE
15	2.0	450	7.5	20	Immersion	Aluminum
16	2.0	450	7.5	20	Spin	PTFE

Figure 2). This device was used to simulate the features of a real circuit; it had no electrical significance. After replicating all standard circuit assembly steps, we subjected cells of the test circuits to various cleaning parameters and encapsulated them in RTV silicone rubber.

We measured leakage current between the encapsulated conductor paths at 85°C and 85-percent relative humidity with a dc bias voltage of 1000V for 1000 hours. Leakage currents exceeding 1 microampere (μA) signified failure.

Experimental Approach

Plasma cleaning is a complex process affected by many factors. For the fractional-factorial designed experiment, we selected factors that affect bulk organic film-removal rates, even though prior THB tests had failed to relate these factors to leakage current:

- Process pressure — A function of total gas flow rate, pumping speed, and chamber size. Prior studies show that process pressure significantly affects etch uniformity and removal rates.
- RF power — RF energy ionizes the process gas to form highly reactive ionic species.
- Dopant — Small additions of a dopant gas stabilize the plasma and increase the concentration of oxygen radicals, which, in turn, increases cleaning rates.
- Process time — Duration of the plasma process. This does not include the time it takes to evacuate the chamber or vent it to atmospheric pressure when the process is complete.

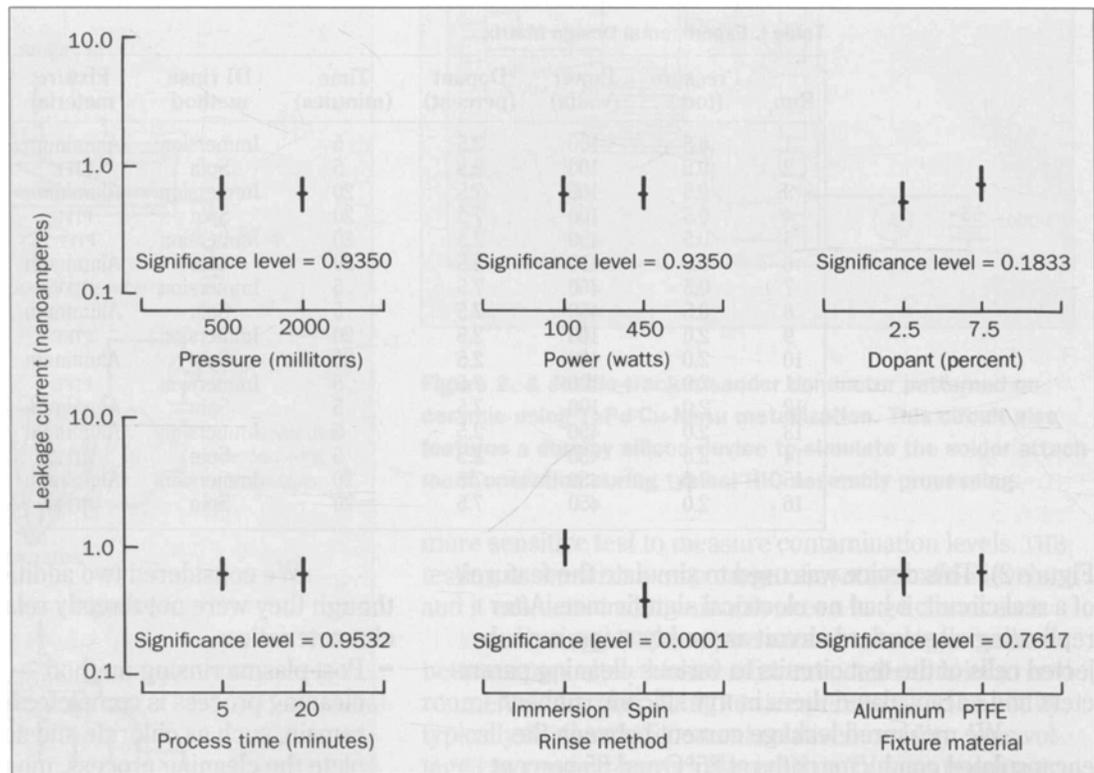
We considered two additional factors, even though they were not directly related to the plasma glow characteristics:

- Post-plasma rinsing method — After the plasma-cleaning process is complete, some ionic residues remain, such as chloride and sodium ions. To complete the cleaning process, most automated circuit-rinsing equipment uses immersion rinsing, although spray rinsing may be more effective.
- Fixture tool material — Typical fixtures that hold hybrid circuits inside the plasma chamber are made from aluminum and polytetrafluoroethylene (PTFE). We conducted earlier reliability tests using PTFE fixtures in the plasma reactor to determine whether this type of fixture material might be a source of contamination.

To examine the main effects on leakage current for each factor, we could have used a full-factorial experimental design, where all combinations of settings for each factor are examined. To study 2 settings for each of the 6 factors, we would need 2^6 , or 64, cells, providing estimates of all main effects and interactions on leakage current.

With the THB test chamber capacity limited to 120 circuits, even a cell size of 2 circuits would be too large, making a full-factorial experiment an impractical choice unless some factors were eliminated. Fortunately, when certain interactions are known to be unlikely, a fractional-factorial design simplifies the experiment without eliminating factors.³ Confounding unlikely interactions with main effects so that all combinations of

Figure 3. Plots of the t-distribution 95-percent confidence limits for the main effects, along with corresponding significance levels. The significance level for pressure, power, and process time are almost unity, so their effects are not significant. The significance level for fixture material is also high, so this effect is insignificant as well. Rinse method is significant at a level of 0.0001. To a lesser degree, the effect of doping gas is significant at a level of 0.1833.



settings and factors are no longer required reduces the size of the experimental matrix without sacrificing necessary information. Limiting the experiment to a study of 6 factors and 2 of the possible 15 interactions reduced the experiment to 16 runs. This raised the number of circuits per cell to 7, leaving space for 8 control circuits. Because process time, rinse method, and fixture material were not expected to interact with other processing parameters, only the interactions between power and pressure, along with pressure and percent dopant gas, were considered to be likely.

The levels selected for power, pressure, and dopant gas generally represented the upper and lower limits of the process. The plasma reactor was capable of operating at pressures ranging from 500 to 2000 millitorrs, so the limits of this range were used for the experiment. The operating range of the mass flow controller, an apparatus that controls the process gas flow, limited gas-doping levels at the higher process pressure range. With an RF power supply rating of 500 watts (W), settings of 100W and 450W assured a stable plasma without overloading the power supply. The levels for rinse method

and fixture tool material represented methods and materials associated with typical production techniques, i.e., circuits could be rinsed by immersion in successive overflow DI water tanks, or by high-pressure spraying in a spin-rinsing apparatus. Aluminum and PTFE are the most common materials used for plasma fixturing.

Table I shows a run-by-run array of experimental variables and their settings for each run.

Results

We combined leakage current values recorded at 1, 6, 25, 50, 100, 170, 265, 504, 770, and 1005 hours for each circuit into a single value by calculating the average of the logs of all readings and taking the antilog of the average using Equation 1.

$$\bar{i} = 10^{\frac{\sum \log_n i}{n}} \quad (1)$$

Since only 5 of 120 circuits exceeded the $1 \mu\text{A}$ failure criteria, response based on circuit failures or time to failure was not appropriate, although cells with fail-

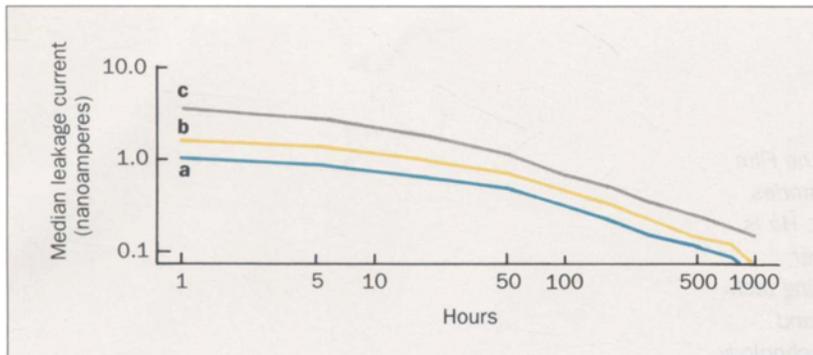


Figure 4. (a) Leakage current for the control cell cleaned in CFC-113 based solvent and hydrogen peroxide, where none of the 8 circuits failed. (b) The combined median leakage current for circuits processed in cells 2, 4, 6, 8, 10, 12, 14, and 16, all of which were spin rinsed. None of the 56 circuits tested failed. (c) The combined median leakage current for circuits processed in cells 1, 3, 5, 7, 9, 11, 13, and 15, all of which were immersion rinsed. Five of the 56 circuits tested failed.

ures had higher median leakage current. Figure 3 shows plots of the t-distribution 95-percent confidence limits for the main effects, along with their corresponding significance levels.

Low significance values suggest strong effects. When examined, these plots show that rinsing and, to a lesser degree, the level of doping are the only factors that have a significant effect on leakage performance. The exceptional results achieved with spin rinsing explain why previous studies failed to produce acceptable THB performance for high-voltage circuits. Figure 4 shows median leakage currents for circuits cleaned in CFC-113 based solvent, followed by hydrogen peroxide and DI rinsing; plasma cleaning, followed by immersion in DI water; and plasma-cleaned circuits spin-rinsed in DI water. In earlier studies, we assumed that immersion rinsing could remove the remaining ionic contamination from the plasma process. The most recent result indicates that acceptable cleanliness is only guaranteed if plasma cleaning is followed by the high impact from the water spray nozzles. Lower levels of doping gas may also improve leakage performance, but the significance of this effect is low relative to the rinsing effect. All other main effects show no difference between parameter settings. Similarly, the interactions between power and pressure and dopant gas and pressure had insignificant effects on leakage.

Summary

Oxygen plasma offers a clean, safe, reliable, and inexpensive alternative to CFC solvents for pre-encapsulation cleaning of HICs. Raw material cost for this process is insignificant compared to CFC-113 based solvents and replacements such as isopropyl alcohol. There is also no waste disposal cost associated with oxygen

plasma, because there are no spent materials to handle. However, if high-voltage circuits undergo oxygen-plasma cleaning before encapsulation, careful spray rinsing in DI water should follow. Accelerated THB testing proves that this method is comparable to any other standard cleaning method.

Fractional-factorial designed experiments can be used to select cleaning parameters for a process in which six-week accelerated age tests are required to guarantee long-term reliability. To reduce the sensitivity of the process to variation in levels of contamination, we dismissed all unimportant effects and focused on relevant parameters. The factorial experiment screened out all plasma-processing parameters and identified rinsing as the key factor that affects leakage current. Doping has a small effect on leakage current, while power, pressure, etch time, and fixture material have no significant effects.

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