

Title:

C a r b o n D i o x i d e - B a s e d S u p e r c r i t i c a l
F l u i d s a s I C M a n u f a c t u r i n g S o l v e n t s

Author(s):

J . B . R u b i n	C S T - 1 2	L A N L
L . B . D a v e n h a l l	C S T - 1 2	L A N L
C . M . V . T a y l o r	C S T - 1 2	L A N L
L . D a l e S i v i l s	C S T - 1 2	L A N L
T . P i e r c e	C S T - 1 2	L A N L

K a r l T i e f e r t
E n v i r o n m e n t a l P r o j e c t M a n a g e r
T h e H e w l e t t - P a c k a r d C o . , I n c .
S a n t a C l a r a , C A

Submitted to:

1 9 9 9 I E E E I n t e r n a t i o n a l S y m p o s i u m o n
E l e c t r o n i c s a n d t h e E n v i r o n m e n t

1 1 - 1 3 M a y 1 9 9 9 , D a n v e r s , M A

RECEIVED
AUG 19 1999
OSTI

Los Alamos

NATIONAL LABORATORY

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the University of California for the U.S. Department of Energy under contract W-7405-ENG-36. By acceptance of this article, the publisher recognizes that the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy. The Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; as an institution, however, the Laboratory does not endorse the viewpoint of a publication or guarantee its technical correctness.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Carbon Dioxide-Based Supercritical Fluids as IC Manufacturing Solvents

J.B. Rubin, L.B. Davenhall, C.M.V. Taylor, L. Dale Sivils and T. Pierce

Physical Organic Chemistry Group (CST-12), Mail Stop E-537
Los Alamos National Laboratory, Los Alamos, New Mexico 87545

Karl Tiefert
Environmental Project Manager
The Hewlett-Packard Co., Inc.
Santa Clara, California 95054-3292

Abstract:

The production of integrated circuits (IC's) involves a number of discrete steps which utilize hazardous or regulated solvents and generate large waste streams. ES&H considerations associated with these chemicals have prompted a search for alternative, more environmentally benign solvent systems. An emerging technology for conventional solvent replacement is the use of supercritical fluids based on carbon dioxide (CO₂).

Research work, conducted at Los Alamos in conjunction with the Hewlett-Packard Company, has led to the development of a CO₂-based supercritical fluid treatment system for the stripping of hard-baked photoresists. This treatment system, known as Supercritical CO₂ Resist Remover, or SCORR, uses a two-component solvent composed of a non-hazardous, non-regulated compound, dissolved in supercritical CO₂. The solvent/treatment system has been successfully tested on metallized Si wafers coated with negative and positive photoresist, the latter both before and after ion-implantation. A description of the experimental data will be presented.

Based on the initial laboratory results, the project has progressed to the design and construction of prototype, single-wafer photoresist-stripping equipment. The integrated system involves a closed-loop, recirculating cycle which continuously cleans and regenerates the CO₂, recycles the dissolved solvent, and separates and concentrates the spent resist. The status of the current design and implementation strategy of a treatment system to existing IC fabrication facilities will be discussed.

Additional remarks will be made on the use of a SCORR-type system for the cleaning of wafers prior to processing.

1. INTRODUCTION

The temperature at which the vapor pressure above a pure liquid reaches one atmosphere is known as the normal boiling point. For water, the normal boiling point at one atmosphere is 100°C. In an open container, Figure 1, the temperature of liquid water cannot be raised above 100°C since this would cause the vapor pressure of water to rise above one atmosphere, which would exceed the ambient pressure conditions. If we place a quantity of liquid water in a *sealed* container, however, then we may heat to higher temperatures, since the vapor pressure of the water can now exceed one atmosphere. As we uniformly heat the sealed container, the density of the liquid water decreases through thermal expansion. Simultaneously, the density of the water vapor increases. We can continue this heating process until the density of the liquid has been so reduced, and the density of the vapor phase has been so increased, that the two densities become equal. The temperature where the liquid and vapor densities become equal is called the critical temperature. Since the temperature inside the sealed container is everywhere equal, and the density is everywhere equal, thermodynamics dictates that the pressure inside the container be everywhere equal. This pressure is called the critical pressure. A fluid that has been brought to conditions above its critical temperature and pressure is known as a supercritical fluid.

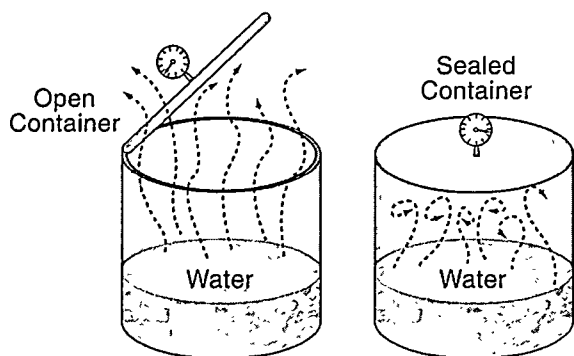


Figure 1. Schematic illustration of supercritical fluid formation by heating of a liquid in a sealed vessel.

This physical description of the formation of a supercritical fluid suggests that all simple fluids can be made supercritical by generating the appropriate conditions of temperature and pressure. This is indeed correct, and Table 1 gives the critical temperatures and pressures of some common fluids.

The attractiveness of supercritical fluids as solvents stems from their unique combination of gas-like and liquid-like properties. Table 2 gives a comparison of some physico-chemical properties of a typical organic fluid in the liquid, supercritical, and gaseous states. It can be seen that the supercritical fluid state possesses *gas-like* values of viscosity and diffusivity. Table 2 also shows that the supercritical state possesses a high, *liquid-like* density. To a first approximation, the solvent power of a fluid is related to its density, and the high, liquid-like density achievable in supercritical fluids allows for substantial solubilities. Figure 2 shows the

Table 1. Critical temperature and pressure for some common fluids.

Fluid	Critical temperature (°C)	Critical Pressure (psi)
Helium, He	-268	33
neon, Ne	-229	400
argon, Ar	-122	706
nitrogen, N ₂	-147	492
oxygen, O ₂	-119	731
carbon dioxide, CO ₂	31	1072
sulfur hexafluoride, SF ₆	46	545
ammonia, NH ₃	133	1654
water, H ₂ O	374	3209

Table 2. Comparison of physico-chemical properties of a typical organic fluid in the liquid, gas, and supercritical fluid state.

	Diffusivity (cm ² /s)	Viscosity (mPa·s)	Density (kg/m ³)
Liquid	10 ⁻⁵	1	1000
Supercritical Fluid	10 ⁻³	10 ⁻²	700
Gas	10 ⁻¹	10 ⁻²	1

density-pressure-temperature surface for pure CO₂. The critical point for pure CO₂ (31°C and 1072 psi) is shown in the figure by the large, solid circle. It can be seen that relatively small changes in temperature or pressure, near the critical point, result in large changes in density. It is this tunability of density, and therefore tunability of solvent power, which is the most attractive attribute of supercritical fluids.

Supercritical fluids are currently used as solvents in many commercial applications, including the extraction of caffeine from coffee, fats from foods, and essential oils and spices from plants. CO₂-based supercritical fluids are particularly attractive because CO₂ is non-toxic, non-flammable, and inexpensive. Its critical conditions are easily achievable with existing process equipment. Also, there is already an extensive transportation infrastructure, as the food service industry relies on the use of solid and high-pressure gaseous CO₂ for food storage and carbonated drinks, respectively. This unique combination of physical, chemical and economic properties of CO₂-based supercritical fluids prompted Hewlett-Packard and Los Alamos to evaluate their use as a replacement for process solvents currently used in the semiconductor manufacturing process of photoresist stripping.

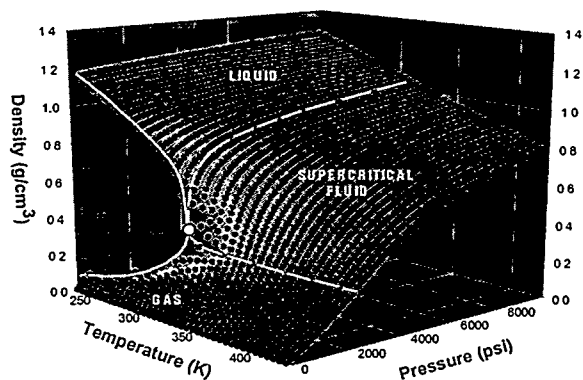


Figure 2. Pressure-temperature-density surface for pure CO₂. The supercritical fluid region is defined by the critical point (31°C and 1072 psi), shown by the large solid circle.

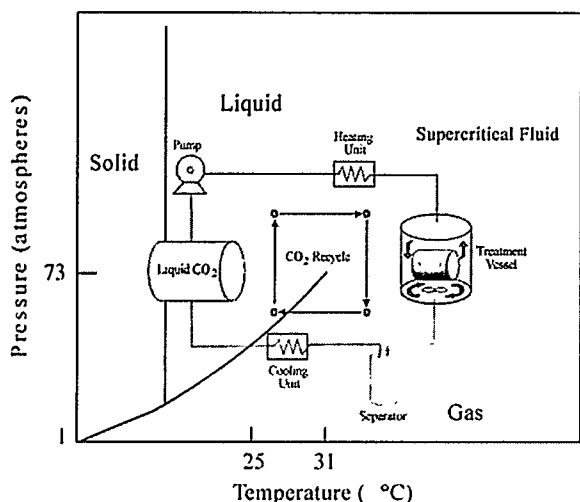


Figure 3. Pressure-temperature phase diagram of pure CO_2 with a superimposed flow diagram for a closed-loop supercritical fluid treatment system.

An inspection of Table 1 shows that the supercritical fluid state requires that fluids be pressurized. In order to maximize the efficiency and economy of a pressurized-solvent, it is desirable to use a recirculating, closed-loop system. Figure 3 shows a schematic pressure-temperature diagram for pure CO_2 , overlaid with a flow diagram of such a closed-loop treatment system. A typical treatment cycle begins with a liquid- CO_2 storage reservoir. The liquid pressure is raised to above the critical pressure during a pumping operation, which sends the pressurized liquid to a heating unit. The heating unit warms the pressurized CO_2 to above the critical temperature, so that a supercritical fluid is formed. The supercritical fluid enters the treatment vessel and is brought into contact with the substrate. During this time the materials to be removed are solubilized and entrained in the CO_2 . A constant flow of CO_2 is maintained through the treatment vessel, so that clean CO_2 is continuously made available. On exiting the treatment vessel, the supercritical CO_2 , containing the dissolved material, is sent to a separation vessel. In the separator, the CO_2 is allowed to expand into the gas phase (see Figure 2), reducing the density to a gas-like value. Once the CO_2 density has been reduced, the solubility of the dissolved materials is also reduced and these precipitate to the bottom of the separator. The clean CO_2 gas exits the top of the separator, where it is condensed into a liquid by a cooling unit before re-entering the storage vessel.

In addition to continuously cleaning and recirculating the solvent, a closed-loop system prevents any uncontrolled waste streams from exiting

the system. All of the extracted materials are retained in the separation vessel for subsequent analysis, recycle, treatment and/or disposal. Further, the extracted materials are concentrated in the separator, greatly reducing the volume of waste.

2. EXPERIMENTAL

2.1. The SCORR Process

The stripping of hard-baked photoresists generates more waste than any other single step in the IC manufacturing process. Unfortunately, alternative processing strategies, which can significantly reduce waste generation and personnel exposure, have not been forthcoming. This is due, in part, to the severe requirements placed on the stripping process, in terms of residual contamination and materials compatibility.

Pure, liquid propylene carbonate, abbreviated PCO_3 and shown schematically in Figure 4, has been found to be an effective, low-toxicity replacement for methylene chloride and methyl chloroform in the debonding of a negative, PMMA-based photoresist (RISTON T168).[1] This finding, and the fact that PCO_3 is not expected to adversely affect thin-film metallizations, prompted us to investigate the use of PCO_3 , as a supercritical CO_2 co-solvent, for its ability to remove hard-baked photoresists currently used by the Hewlett-Packard Company.

PCO_3 is an environmentally friendly solvent, having no Personal Exposure Limit (PEL). It is non-flammable, non-toxic, biodegradable, and has a low vapor pressure.[2,3] It is readily available in high purity and in large quantities. Finally, PCO_3 is completely miscible with high-pressure CO_2 , as shown in Figure 5.

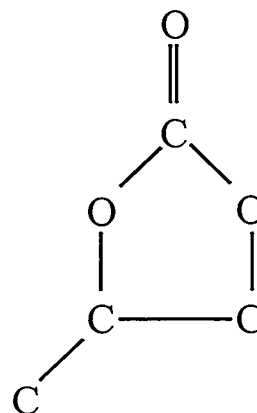


Figure 4. Structure of propylene carbonate (PCO_3). The hydrogen atoms are omitted for clarity.

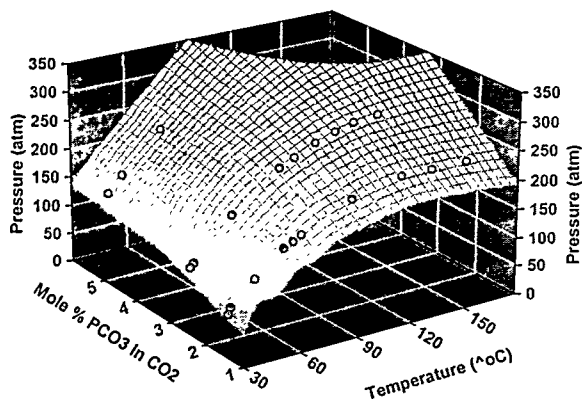


Figure 5. Vapor-liquid critical surface extrapolated from data given in [4]. The $\text{PCO}_3\text{-CO}_2$ mixture is single-phase at all conditions above the surface.

We have performed experiments on metallized Si wafers coated with hard-baked, positive-tone photoresists, and unmetallized Si wafers with positive-tone photoresists after ion-implantation. Column 1 in Table 3 lists the various types of metallization systems evaluated in this work.

For the initial experiments, a single-pass treatment system was constructed, shown schematically in Figure 6. The SCORR solvent is supplied by a pre-mixed, compressed gas cylinder. This cylinder feeds a high-pressure syringe pump, which supplies high-pressure, ambient-temperature SCORR solvent to a ballast tank and treatment vessel. The Si wafer sample is centrally mounted inside the treatment vessel.

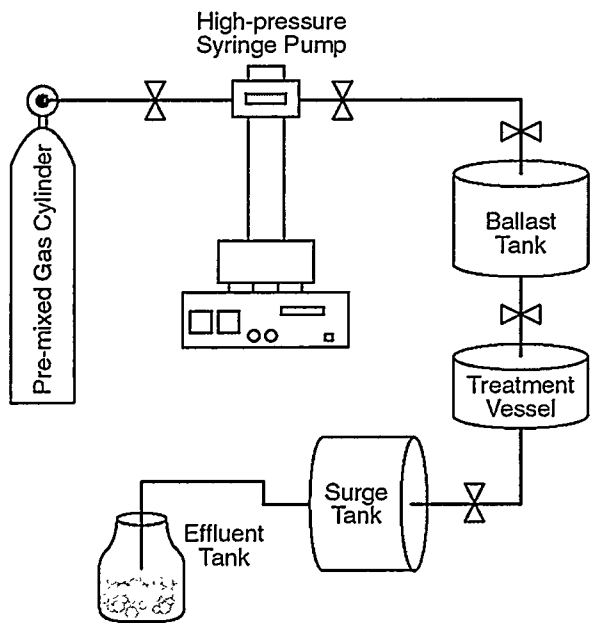


Figure 6. Schematic drawing of the initial SCORR treatment system.

Once the ballast tank and treatment vessel are brought to their initial conditions of temperature and pressure, a valve leading from the ballast tank to the treatment vessel is opened, allowing the solvent mixture to flow into the vessel and onto the sample surface. This flow, directed by a nozzle, continues for several seconds, until the pressures in the tank and vessel equilibrate. Subsequently, a needle valve leading from the treatment vessel to a surge tank is opened until the treatment vessel pressure drops back to its initial value. The surge tank is maintained at atmospheric pressure and empties into an effluent tank to retain the solvent and the spent photoresist for subsequent analysis.

While the treatment vessel de-pressurizes, the ballast tank is re-pressurized. This pressurization/de-pressurization cycle is repeated three times, applying the SCORR solvent in three separate pulses. Following completion of the third treatment cycle, the PCO_3 source is valved-off, and a pure CO_2 "rinse and dry" is applied to the treated sample, to remove all residual solvent. The sample is then removed from the treatment vessel and analyzed for residual photoresist.

2.2. Nuclear Reaction Analysis (NRA)

Nuclear Reaction Analysis (NRA) was used to evaluate the degree of removal of photoresist from the treated Si wafers. NRA is a rapid (< 15 min.) analytical method, which probes a relatively large surface area ($\approx 10 \text{ mm}^2$). The method provides a high degree of sensitivity to carbon (the main component of photoresists), and is non-destructive both to the substrate and to the metallizations. As a result, we are able to obtain high-precision, quantitative data on residual carbon concentration on the wafer surface,

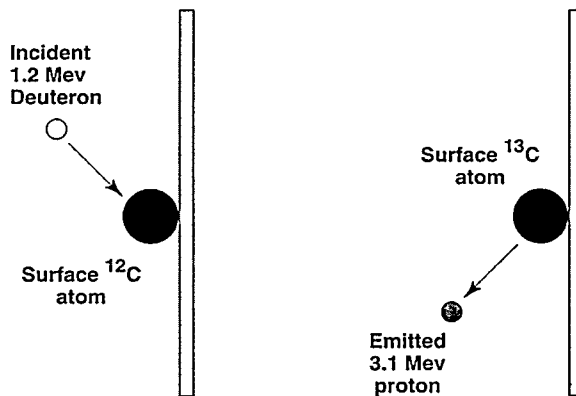
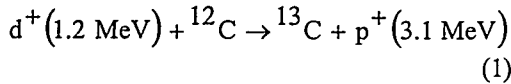


Figure 7. Schematic illustration of the nuclear reaction occurring on the surface of the silicon wafer.

while preserving the samples for subsequent electron microscopy analysis.

In an NRA experiment, Figure 7, a beam of 1.2 MeV deuterons is directed onto the wafer surface. Carbon nuclei have an absorption resonance for deuterons at this energy, so that the deuterons are "absorbed", temporarily raising the carbon nuclei to an excited-state. The carbon atoms relax by emitting a high-energy proton, which is detected. The overall nuclear reaction can be written :



where d^+ is the incident deuteron and p^+ is the emitted proton. Because the emitted protons have much higher energies than the incident deuterons, the signal to noise ratio is inherently high, making the technique very sensitive to low carbon concentrations.

The second column in Table 3 gives the experimental results for the samples treated with the SCORR system. The residual carbon on the wafer surfaces, after the treatment, amounts to 1-10 atomic layers. We note that the samples were treated in an open laboratory, and not in a clean-room environment. As a result, the NRA results will include carbon in the form of CO_2 left absorbed on

Table 3. NRA results for Si wafer samples treated with the SCORR process.

Sample description	Concentration of surface carbon atoms
Al on Si with positive resist	10 monolayers
In-Sn oxide on Si with positive resist	6 monolayers
Ti/W on Si with positive resist	2 monolayers
Pt/Ti on Si with positive resist	< 1 monolayer
Si with positive resist, implanted with $5 \times 10^{14} \text{ cm}^{-2} p^+$	3 monolayers
Si with positive resist, implanted with $6.2 \times 10^{15} \text{ cm}^{-2} \text{As}^+$	6 monolayers

the wafer surface during the final "rinse and dry" step of the treatment, as well as atmospheric CO_2 adsorbed during sample transfer between the laboratory and the NRA facility.

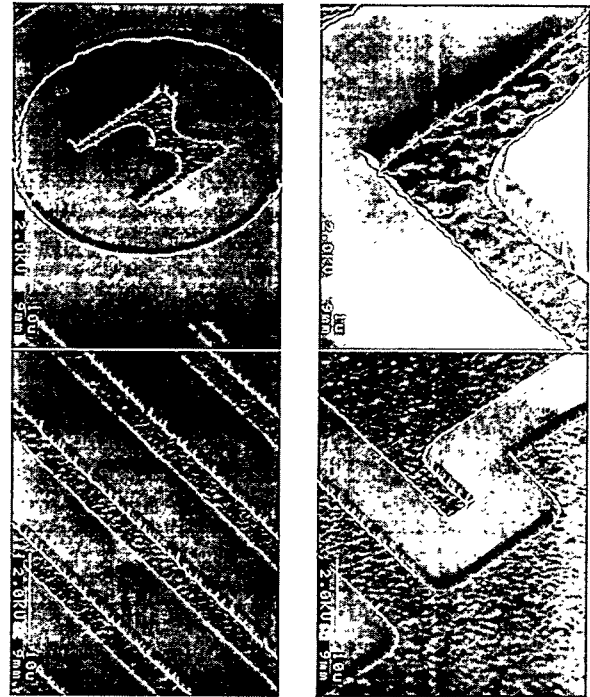


Figure 8. Composite of four electron micrographs of the SCORR-treated Si wafer with aluminum surface metallizations.

2.2. Scanning Electron Microscopy (SEM)

Verification of photoresist removal represents the first step of our research goal. The required second step is to demonstrate that the SCORR treatment is not destructive to pre-fabricated thin-film metallizations. We therefore performed Scanning Electron Microscopy (SEM) analyses on the metallized Si wafers which were SCORR-treated, to determine if the metallizations are in any way undercut or etched. All microscopy was done on the same samples which were analyzed by NRA. Figure 8 shows a composite of four electron micrographs of the SCORR-treated Al/Si sample. No etching, pitting, or undercutting are evident. A similar absence of deleterious reactions was seen for the remaining metallized samples, indicating that the supercritical fluid composition and treatment parameters appear to be compatible with existing IC processing procedures.

3. DISCUSSION

During the course of the SCORR experimental program, we have found that only by combining the PCO_3/CO_2 mixture in a flowing system could complete photoresist removal be achieved. The mechanism by which the SCORR treatment removes the photoresist has not yet been determined. However, it is well known that

polymeric materials can be made to swell by diffusion of CO₂ molecules and, in many instances, a significant reduction in the glass transition temperature can be produced. It is likely that such swelling occurs, effectively softening the resist. At the same time, the reactive ester group of the PCO₂ (see Figure 4) oxidizes the polymer, reducing its average molecular weight. This molecular weight reduction promotes solubility in the supercritical fluid, facilitating its removal.

It is a logical idea that a process which can remove photoresist films may also be suitable for the initial surface cleaning of wafers. In the supercritical state, CO₂ behaves as a non-polar organic solvent, similar to hexane in its solvating properties. Consequently, pure supercritical CO₂ is excellent for solubilizing other organics, such as greases, oils, lubricants and fingerprints. In addition, the negligible surface tension and low viscosity of supercritical CO₂ (see Table 2) promote particulate removal by significantly reducing the thickness of the surface boundary flow layer. Table 4 illustrates the desirable physical and chemical properties of dense-phase (supercritical and liquid) CO₂ compared to some liquids commonly used as surface cleaning solvents. There has been considerable work done in developing supercritical fluids, and supercritical CO₂ in particular, for the precision-cleaning of inorganic surfaces,[5,6,7] including metals,[8,9,10,11] optical elements,[12] Si wafers,[13] and micro-electromechanical devices fabricated on Si.[14,15,16]

The advantage of surface cleaning using

supercritical CO₂ is that the process would leave no organic residues. As a result, subsequent rinsing and drying steps would not be required. In addition to organic residues, however, semiconductor wafer cleaning may also involve the removal of inorganic particulates and trace metals. Pure CO₂ would require the use of specific modifiers, or co-solvents, for this purpose,[17] and there are a number of surfactants [18] and metal chelating agents [19] which can be used. Even if such modifiers were necessary, a final "rinse and dry" using pure CO₂ should eliminate their residues. There is a commercial system available for micro-electromechanical device fabrication that uses pure CO₂,[20] and large capacity systems for machine part cleaning are being installed in Europe.

4. CONCLUSIONS

Preliminary results indicate that CO₂-based supercritical fluids are attractive alternative solvents for photoresist stripping. Such fluids, as part of an appropriately designed, closed-loop treatment system, have the potential to significantly reduce waste generation and water use. The reduction in water use is particularly significant, as the amount spent by the semiconductor industry on ultrapure rinse water in 1996 equaled the amount spent on all other chemicals for wet cleaning.[21] The Semiconductor Industry Association has set itself the goal of reducing the amount of ultrapure water used in 0.25 μm fab facilities by 50%.

Table 4. Physico-chemical properties of dense-phase (liquid and supercritical) CO₂ and some fluids commonly used for surface cleaning. (Typical values at ambient conditions unless otherwise stated.)

Solvent	Viscosity (mPa·s)	Surface Tension (mN/m)	Relative Dielectric Constant	Dipole moment (Debye)	Density (kg/m ³)
Liquid CO ₂	0.02 (20°C, 59 atm)	1.5 (20°C, 58 atm)	1.5 (20°C, 59 atm)	0	792 (20°C, 60 atm)
Supercritical CO ₂	0.03 (35°C, 75 atm)	≡ 0 (T ≥ T _c and P ≥ P _c)	1.1 (35°C, 73 atm)	0	258 (35°C, 73 atm)
1,1,1-trichloroethane (methyl chloroform)	0.79	25.2	7.5	1.8	1339
1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)	0.66	17.8	2.4	0.9	1564
Methanol	0.54	22.1	32.7	1.7	791
Isopropyl alcohol	2.04	20.9	20.2	1.6	786
Acetone	0.31	23.5	21.0	2.9	790
hexane	0.30	17.9	1.9	0	655
Water	1.00	72.0	78.5	1.8	1000

The actual treatment schedule for the samples we have examined thus far was arbitrarily chosen, and only limited experimentation has been devoted to process optimization. We have determined, however, that increasing the treatment temperature significantly increases the amount of photoresist softening, for a given treatment time, and would therefore be expected to significantly reduce the required, overall treatment time. It must be established by electrical testing that there is no adverse effect of the SCORR treatment on pre-fabricated surface and sub-surface structures. Finally, additional work needs to be done on characterizing, using completed, production-size wafers, contamination due to widely dispersed particulates and trace concentrations of transition metals. We are currently performing experiments on 6" production wafers to evaluate these issues. The results will be discussed.

Although our research has focused on the photoresist-stripping step of the fabrication process, there are other fabrication steps which may benefit from supercritical fluid processing. For example, it has been established that supercritical fluids can be used to deposit metallic and insulating thin films, and so could be used for generate interconnect lines, barrier layers and low-dielectric constant films. It is also possible to use supercritical fluids, containing dissolved, reactive components, to etch metal and oxide films. Finally, supercritical water oxidation is a proven method for the complete oxidation of organic compounds, and so could be used for the treatment of wastewater.

Acknowledgments:

We acknowledge the assistance of Drs. Carl Maggiore and Joe Tesmer of the Los Alamos Ion Beam Materials Laboratory for their assistance with the NRA analyses, Dr. Robert Dickerson of the Los Alamos Electron Microscopy Facility for assistance with electron microscopy analyses, and Ms. Maida Trujillo of CST-12 for the graphic illustrations.

5. REFERENCES

1. K.I. Papatomas and A.C. Bhatt, "Debonding of Photoresists by Organic-Solvents" *J. App. Poly. Sci.* 59 (1996) pp.2029-2037.
2. H. Klein and T. Marquis, "Cyclic Organic Carbonates Serve as Solvents and Reactive Diluents", *Coatings World (May/June, 1997)*.
3. K.H. Beyer, Jr., W.F. Bergfeld, W.D. Berndt, W.H. Carlton, D.K. Hoffman, A.L. Schroeter and R.C. Shank, "Final Report on the Safety Assessment

- of Propylene Carbonate", *J. Amer. Coll. Toxicology* 6 (1987) pp. 23-51.
4. S.H. Page, D.E. Raynie, S.R. Goates, M.E. Lee, D.J. Dixon and K.P. Johnston, "Predictability and Effect of Phase Behavior of CO₂/Propylene Carbonate in Supercritical Fluid Chromatography", *J. Microcol. Sep.* 3 (1991) pop. 355-369.
5. D.P. Jackson, "Cleaning Process Using Phase Shifting of Dense Phase Gases", US Patent 5,013,366 (1991).
6. D.P. Jackson, "Dense Phase Gas Photochemical Process for Substrate Treatment", US Patent 5,068,040 (1991).
7. K.L. Hoy and K.A. Nielsen, "Methods for Cleaning Apparatus Using Compressed Fluids", US Patent No. 5,306,350 (1994).
8. L.J. Silva, "Supercritical Fluid for Cleaning Metal Parts", *Hazardous Waste Consultant*, 13 (1995) pp. A25-A26.
9. R. Purtell, L. Rothman, B. Eldridge and C. Chess, "Precision Parts Cleaning Using Supercritical Fluids", *J. Vac. Sci. & Tech.* A11 (1993) pp. 1696-1701.
10. W.K. Tolley, P.B. Altringer and D.C. Seidel, "Stripping Organics from Metal and Mineral Surfaces Using Supercritical Fluids", *Sep. Sci & Tech.* 22 (1987) pp. 1087-1101.
11. N. Dahmen, J. Schon, H. Schmieder, and K. Ebert, "Supercritical-Fluid Extraction of Grinding and Metal-Cutting Waste Contaminated with Oils", Supercritical Fluids - Extraction and Pollution Prevention, ACS Symposium Series, vol. 670 (ACS, Washington, D.C., 1997) pp. 270-279.
12. J. McHarty, T.B. Stanford, L.R. Benjamin, T.E. Whiting and S.C. Chao, "Progress in Supercritical Cleaning", *SAMPE J.* 29 (1993) pp. 20-27.
13. E. Bok, D. Kelch and K.S. Schumacher, "Supercritical Fluids for Single Wafer Cleaning", *Solid State Technology* 35 (1992) pp. 117-120.
14. M.A. Douglas and R.M. Wallace, "Method for Unsticking Components of Micro-Mechanical Devices", US Patent No. 5,482,564 (1996).
15. E.M. Russick, C.L.J. Adkins and C.W. Dyck, "Supercritical Carbon Dioxide Extraction of Solvent from Micromachined Structures", Supercritical Fluids - Extraction and Pollution Prevention, ACS Symposium Series, vol. 670 (ACS, Washington, D.C., 1997) pp. 255-269.
16. J. Bühler, F.-P. Steiner, R. Hauert and H. Baltes, "Linear Array of Complimentary Metal Oxide Semiconductor Double-Pass Metal Micromirrors", *Opt. Eng.* 36 (1997) pp. 1391-1398.

17. P.M. Gallagher and V.J. Krukonis, "Precision Parts Cleaning with Supercritical Carbon Dioxide", International CFC and Halon Alternatives Conference (Baltimore, MD, Dec. 3-5, 1991) pp. 262-271.

18. K. Jackson and J.L. Fulton, "Surfactants and Microemulsions in Supercritical Fluids" in Supercritical Fluid Cleaning, edited by J. McHardy and S.P. Sawan (Noyes Publications, Westwood, NJ, 1998) pp. 87-120.

19. C.M. Wai and S. Wang, "Supercritical Fluid Extraction: Metals as Complexes", *J. Chromatography*, A785 (1997) pp.369-383.

20. GT Equipment Technologies, Inc., 472 Amherst St., Nashua, NH 03063.

21. M.C. Lancaster, "Ultrapure Water: The Real Cost", *Solid State Technology* (Sept. 1996) pp. 70-75.