



Efficient stripping of photoresist on metallized wafers by a pause flow of supercritical fluid

Mu-Rong Chao^a, Jian-Lian Chen^{b,*}

^a Department of Occupational Safety and Health, Chung Shan Medical University, No. 110, Sec. 1, Chien-Kuo N Road, Taichung 402, Taiwan

^b School of Pharmacy, China Medical University, No. 91 Hsueh-Shih Road, Taichung 40402, Taiwan

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ABSTRACT

Utilization of supercritical fluids (SCFs) is studied here on the premises of a saving of hazardous organic solvents and of the specification for stripping the photoresist (PR) on metallization layers, which is one of the integrated circuit processing modules. By using factorial experimental designs with five factors and four level ranges, this research focuses on determining an optimized recipe with high stripping efficiency and to determine the stripping mechanism. In the case of PR on an aluminum layer, the initial use of the pulse flow mode could increase the extraction ratio remarkably when compared to the conventional continuous flow mode. Based on the limitation of a total volume of 30 mL purging SCF-CO₂ for economical considerations, the optimum conditions can be summarized as follows: 120 °C, oven temperature; 350 atm, CO₂ pressure; 0.2 mL of ethylacetate spiking to SCF-CO₂; 2.0 min, static equilibrium time; and five cycles of dynamic flow pausing. A recovery of 94.6% ($n = 3$, RSD = 6.5%) was obtained, while the diffusion of stripped PR from substrate matrix prevailed over the dissolution of binding PR into the SCF medium. In the case of copper, the optimum parameters in a pause flow mode were 140 °C, oven temperature; 500 atm, CO₂ pressure; 0.75 mL, ethylacetate spiking volume; 5.0 min, static time; and six cycles of flow pausing. These extreme parameters still did not produce an SCF environment suitable for diffusion or dissolution mass transfer, and thus a recovery of 76.2% ($n = 3$, RSD = 7.5%) was only obtained. Removing PR coated on a Cu layer was harder than that on an Al layer.

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1. Introduction

Microelectronics manufacturing is the largest industry in the world and has continued to keep pace with Moore's law of exponential progress for decades [1,2]. In a typical chip-fabrication plant, production of a 2 g microchip can consume 32 kg of water, 700 g of ultra-pure gases, 1.6 kg of fossil fuels and 72 g of chemicals [3]. Besides the considerations of the environment, safety and health, any alternative technologies are not just "greener" but provide valid technical advantages that may allow innovative component designs. Supercritical fluid (SCF) technology is not surprisingly the prime candidate for the identification of global challenges that will be met in 2015 and was outlined in the 2003 International Technology Roadmap for Semiconductors [4].

Some applications of SCF in integrated circuit (IC) manufacturing operations, including the processing of photoresists (PR), wafer cleaning and etching chemistries, the deposition of metals and dielectric constant films, and chemical mechanical planariza-

tion, have been reviewed [5–8]. PR stripping and cleaning is the first application of SCF in IC processing. A group in Los Alamos National Laboratory developed a series of SCF cleaning processes and collaborated with equipment makers to produce a commercial device [9–13]. They showed that SCF removal of PR minimizes the use of hazardous solvents and eliminates rinsing and drying steps. Moreover, SCF cleaning allows production of features of less than 100 nm due to the low surface tension and gas-like viscosity of supercritical CO₂.

Supercritical CO₂ is the solvent of choice because it is non-flammable, environmentally benign and exhibits convenient critical properties ($T_c = 304.3$ K, $P_c = 7.38$ MPa). The high compressibility of the CO₂-SCF medium allows the solubility and diffusivity to be widely varied with the pressure control. In addition, the temperature factor also affects the SCF density and the detachment of adhesive PR from wafer substrate. Furthermore, the addition of suitable cosolvents to the CO₂-SCF adjusts the polarity so that it is compatible to that of PR and thus facilitates the dissolving PR into SCF medium. All described parameters need to be coordinately optimized so that PR molecules can freely escape from the binding matrices and then blend smoothly with the modified SCF. To meet these needs, the factorial design [14,15] and multilinear regression [16,17] are formal optimization methods, which are certainly supe-

* Corresponding author. Tel.: +886 4 22053366; fax: +886 4 22031075.

E-mail addresses: mrchao@csmu.edu.tw (M.-R. Chao), cjl@mail.cmu.edu.tw (J.-L. Chen).

rior to the general univariate optimization and are the strategies of experimentation [18].

In a previous study, we used factorial experimental designs to optimize the stripping of commercial PR on silicon oxide and aluminum layers, and to determine the basic effects of processing variables on the ratio of recovery [19]. Progression of the lab-scale representation of SCF removal to commercial apparatus requires a full understanding of fluid flow and mass transport within an extractor. In this study, a pause flow mode was used to increase the PR removal efficiency on metallized substrates (which was barely satisfactory in a continuous flow reactor). Through the factorial optimization, we were able to improve mass diffusion.

2. Experimental

2.1. Reagents and chemicals

Ethylacetate (EA), purchased from Merck (Darmstadt, Germany), and CO₂ supercritical fluid, from Scott Specialty Gases (Plumsteadville, PA, USA), were the solvents used.

2.2. Apparatus

The ISCO SFX 220 Extraction System (Lincoln, Nebraska, USA) consists of an SFX 220 Extractor, a 260 D syringe pump, and an SFX 200 Controller, which controls all pumping and extraction operations through six motor actuated valves. The operation limits for the system are 510 atm and 150 °C. Test tube clips are attached to the side of the extractor to allow the extracted compounds to be conveniently collected in a collection tube. A line restrictor carried extracted compounds from the extractor to the collection tube. To prevent ice from clogging while a large volume expansion occurs in the atmosphere, a capillary restrictor was coaxially heated by a temperature controller to maintain the SCF dynamic flow at 1 mL min⁻¹. This controller heated the entire restrictor by passing an electric current through it for even heating.

A Jasco (Tokyo, Japan) spectrophotometer, V530, was used for UV absorption measurement of the extractives.

2.3. Wafer sample preparation

Samples were prepared by coating photoresist on metallized silicon wafers (200 mm diameter, 15–25 Ω cm, (1 0 0) face, Taisil Electronic Material Corp. (Hsinchu, Taiwan)). A silicon oxide layer, 2000 Å ± 4% thickness, was padded on the silicon substrate by plasma enhanced chemical vapor deposition (PECVD). An Al–Cu alloy (99.5:0.5) layer, 4000 Å ± 5% thickness, was sputtered onto the padded oxide layer by physical vapor deposition (PVD). The copper layer, 5000 Å ± 3%, was electrodeposited on 1500 Å Cu seed layer/300 Å TaN barrier layer/2000 Å pad oxide layer, and the Cu seed layer and TaN layer were deposited by PVD without breaking vacuum. Photoresist YSB 663, polymethylstyrene derivatives from Nippon Zeon Co., Ltd. (Tokyo, Japan) with a 6000 Å ± 2.3% thickness

Table 1
Design matrix for L16 (4⁵) factorial experiments.

Run	A ^a	B	C	D	E
1	1 ^b	1	1	1	1
2	1	2	2	2	2
3	1	3	3	3	3
4	1	4	4	4	4
5	2	1	2	3	4
6	2	2	1	4	3
7	2	3	4	1	2
8	2	4	3	2	1
9	3	1	3	4	2
10	3	2	4	3	1
11	3	3	1	2	4
12	3	4	2	1	3
13	4	1	4	2	3
14	4	2	3	1	4
15	4	3	2	4	1
16	4	4	1	3	2

^a Italic letters refer to the variable of SFE experimental condition defined in Tables 2–5.

^b Italic numbers refer to the level in a variable.

was spin-coated on the prepared metallized wafers, soft-baked at 90 °C for 30 s, and then hard-baked at 120 °C for 90 s. These wafer samples were cut into strips (4 cm × 1 cm) to fit to the SCF extraction vessel.

2.4. SCF extraction (SFE)

A four-level factorial design, L16, was employed to assess the significance of five variables (oven temperature, SCF–CO₂ pressure, static equilibrium time, dynamic elution time, and the volume of organic modifier added directly to solid samples) for optimizing the SFE conditions. The L16 orthogonal array table in Table 1 defined 16 sets of experimental parameters, which correspond to the factors arranged in Tables 2–5. For example, if the factors in Table 2 are applied to L16, experiment no. 9 will be assigned to the extractor parameters of 110 °C (oven temperature), 500 atm (CO₂, pressure), 5 min (static time), 10 min (dynamic time), and 0.50 mL (EA spiking volume).

There are two SCF flow modes for purging PR from wafer substrates in this study. In a continuous flow mode, samples undergo only one dynamic purging procedure after a period of static equilibrium. This mode is most commonly used, and was also operated under the controlled factors presented in Table 2. In a pause flow mode, however, a cycle of alternate static equilibrium and dynamic purges proceeds for several times. The number of cycles depends on which level of dynamic time is required in Tables 3–5. For example, in Table 3, the level 4 dynamic time is 5.0 min, that is, six purging cycles were executed for a total dynamic time of 30 min (SCF volume = 30 mL at a purge rate of 1 mL min⁻¹). Thirty minutes was fixed throughout this study for pause flow mode.

Just before an extraction started, cosolvent was spiked on the glass wool, which was inserted at the backside of a strip wafer

Table 2
Factors designed for the L16 factorial experiments and their contributions to stripping PR on Al layer in a complete flow mode.

Level ^a	A; Oven temp. (°C)	B; CO ₂ pressure (atm)	C; Static time (min)	D; Dynamic time (min)	E; Spiked EA (mL)
1	50 (16.4) ^b	500 (22.5)	15 (21.8)	30 (28.2)	0.75 (17.0)
2	80 (22.4)	400 (29.3)	10 (29.6)	20 (22.9)	0.50 (22.1)
3	110 (29.8)	300 (18.1)	5.0 (19.3)	15 (17.8)	0.25 (30.3)
4	140 (16.1)	200 (14.8)	0 (14.0)	10 (15.8)	0 (15.3)
Variance ^c	165.9	157.2	168.5	123.5	181.4

^a Corresponding to the italic numbers in Table 1.

^b Average response recovery of a factor.

^c Variance due to a variable.

Table 3

Factors designed for the L16 factorial experiments and their contributions to stripping PR on Al layer in a pause flow mode.

Level ^a	A; Oven temp. (°C)	B; CO ₂ pressure (atm)	C; Static time (min)	D; Dynamic time (min)	E; Spiked EA (mL)
1	50 (21.1) ^b	500 (22.9)	10 (22.0)	30 (24.0)	0.75 (21.2)
2	80 (31.7)	400 (37.6)	7.0 (26.9)	15 (28.3)	0.50 (32.7)
3	110 (41.9)	300 (43.0)	4.0 (32.7)	10 (35.6)	0.25 (40.1)
4	140 (36.8)	200 (28.0)	1.0 (49.9)	5.0 (43.6)	0 (37.5)
Variance ^c	315.9	330.8	591.8	296.2	279.9

^a Corresponding to the italic numbers in Table 1.^b Average response recovery of a factor.^c Variance due to a variable.**Table 4**

Improved factors designed for the L16 factorial experiments and their contributions to stripping PR on Al layer in a pause flow mode.

Level ^a	A; Oven temp. (°C)	B; CO ₂ pressure (atm)	C; Static time (min)	D; Dynamic time (min)	E; Spiked EA (mL)
1	85 (57.2) ^b	400 (55.5)	3.0 (58.8)	10 (52.1)	0.4 (57.1)
2	100 (59.0)	350 (67.9)	2.0 (67.8)	6.0 (64.3)	0.3 (59.9)
3	120 (64.9)	300 (54.3)	1.0 (54.9)	5.0 (63.2)	0.2 (63.8)
4	135 (49.8)	250 (53.2)	0.5 (49.4)	3.0 (51.3)	0.1 (50.1)
Variance ^c	154.9	187.6	239.9	194.8	133.6

^a Corresponding to the italic numbers in Table 1.^b Average response recovery of a factor.^c Variance due to a variable.

sample, to eliminate direct contact with the sample and allow for uniform spreading. A collection vial containing 6 mL EA was used to trap the eluted extracts. As a dynamic purging began, some EA vented from the vial due to high-pressure expansion. Some EA was also used to rinse PR residues out of the vial wall and make up the 6 mL level for the next evaluation of extraction efficiency after completing an extraction program.

2.5. Quantification of extraction efficiency

The extraction recovery was calculated by the molar ratio of the PR extracted through SFE to the one through EA solvent extraction, whose efficiency is supposed to be 100%. For solvent extraction, a strip of wafer sample whose size was the same as that used in SFE, was immersed in 20 mL of EA and stirred for 3 min. The UV absorbance of the extraction solutions was measured by a spectrophotometer set to 278 nm. That is the extraction recovery could be calculated by the following equation:

$$\text{Recovery (\%)} = \left(\frac{\text{Absorbance of SFE extracts}}{\text{Absorbance of EA extracts}} \right) \times \left(\frac{6 \text{ mL}}{20 \text{ mL}} \right).$$

3. Results and discussion

3.1. Stripping PR on aluminum surface

In a previous study on the SCF stripping of PR on aluminum substrates, there was only an approximate recovery of 70% obtained despite collection for 45 min [19]. This low recovery may result

from improper SCF conditions, which were formerly designed to strip another type of PR on silicon oxide substrate. Consequently, we expect a new L16 factorial optimization to improve on the extraction efficiency found in our previous work. Moreover, an improvement in the purge program of the SCF flow was used to raise the efficiency of PR extraction.

3.1.1. Continuous flow mode

In a continuous flow mode, SCF continuously purges PR from the extraction vessel to the collection vial for an entire dynamic time after a period of static equilibrium time. A single extraction loop is performed, and there are no additional dynamic or static stages. The continuous-flow mode is used in most SFE studies.

The experimental factors generated from an L16 factorial design for stripping PR from an aluminum substrate in continuous flow mode are listed in Table 2. The contributions of each factor to the total recovery are shown in Table 2, which also indicates which level produced the highest response for each variable. Based on these results, we concluded that the optimum parameters (110 °C, oven temperature; 400 atm, CO₂ pressure; 10 min, static time; 30 min, dynamic time; 0.25 mL, EA spiking volume) are different from those used in the former study (125 °C; 480 atm; 2.5 min, static time; 35 min, dynamic time; 1.25 mL) [19]. The predicted recovery calculated by the sum of each factor's contributions was only 62.5%, which seems far from expectation. Even though the dynamic time increased to 45 min, the actual experimental recovery only reached 71% ($n=4$, RSD = 11.8%). In addition, further modification of the level ranges used in Table 2 would be invalid, as the variances between levels for each of the variables were small (<200). Thus, we assumed

Table 5

Factors designed for the L16 factorial experiments and their contributions to stripping PR on Cu layer in a pause flow mode.

Level ^a	A; Oven temp. (°C)	B; CO ₂ pressure (atm)	C; Static time (min)	D; Dynamic time (min)	E; Spiked EA (mL)
1	80 (35.7) ^b	500 (48.8)	10 (35.5)	15 (38.0)	0.75 (48.5)
2	100 (39.8)	450 (45.1)	5.0 (48.0)	10 (43.5)	0.50 (40.5)
3	120 (42.9)	400 (36.8)	2.0 (42.6)	5.0 (47.8)	0.25 (39.2)
4	140 (46.3)	350 (34.0)	1.0 (38.6)	2.0 (35.4)	0 (36.5)
Variance ^c	81.5	192.2	116.6	123.6	106.5

^a Corresponding to the italic numbers in Table 1.^b Average response recovery of a factor.^c Variance due to a variable.

that whatever determined the stripping efficiency no longer originated in the composition of SCF but in the ways of SCF flow.

3.1.2. Pause flow mode

In a pause flow mode, the static equilibrium and dynamic purging steps are repeated alternately for several cycles. In order to compare the recovery between the pause and continuous flow mode, the total volume of SCF running for a whole program was restricted to 30 mL, which is reasonable for economic concerns and comparable to the previous data. The detailed experimental procedure is described in Section 2.4 and the factors generated by the L16 factorial design are listed in Table 3.

The contribution of each factor and the best level for a corresponding variable are listed in Table 3. The optimum parameters can be summarized as the following: 110 °C, oven temperature; 300 atm, CO₂ pressure; 1.0 min, static time; 5.0 min, dynamic time; 0.25 mL, EA spiking volume. The expected recovery was calculated to be 87.0%, which is obviously greater than those obtained in a continuous flow mode (62.5%). In fact, the averaged percentage of 84.2 ($n=3$, RSD=8.3%) was obtained by the optimum parameters. Accordingly, the primary results were satisfactory and successfully validated our assumption that the way of SCF flow purge was the most important factor affecting PR stripping efficiency in an extractor with confined capacity. The minor difference in the composition of SCF (which was much more strongly influenced by the oven temperature, CO₂ pressure and EA spiking volume) between the two operation modes supports the assumption further. The optimum CO₂ pressure decreased from 400 atm to 300 atm when moving from continuous flow mode to pause flow mode, but the optimal oven temperature (110 °C) and EA spiking volume (0.25 mL) were unchanged for these two modes. According to the equation [20]:

$$\delta = 0.47P_c^{1/2} \frac{\rho}{\rho_c}$$

where δ is the solubility parameter, P_c is the critical pressure, and ρ_c is the density at P_c , ρ is proportional to δ . As a result, the lower pressure lessens the solvating power. However, the lower pressure also reduces the SCF viscosity and thus enhances the SCF diffusivity. In our extraction system, the extent of diffusivity was more important than solubility. That is, the regular renewal of SCF, which was generated by purging and refilling the extractor with fresh SCF several times, increased diffusional transport from the wafer matrix to the bulk SCF for the stripped PR.

The above findings indicated that use of a pause flow mode is one way to boost extraction efficiency. Nevertheless, for a further improvement in the recovery, there is still a need to fine tune the levels of each variable, as the values of variance in Table 3 were much larger than 200. A significant improvement was especially expected from adjustment of the levels of the static and dynamic time, as their optimal values were quite distinct in pause flow mode. The improved L16 factorial design is shown in Table 4. Its corresponding results suggested the optimum parameters as the following: 120 °C, oven temperature; 350 atm, CO₂ pressure; 2.0 min, static time; 6.0 min, dynamic time; 0.2 mL, EA spiking volume. The expected recovery was calculated to be 97.8%, and a statistically identical 94.6% recovery ($n=3$, RSD=6.5%) was found experimentally. The possibility of improving PR recovery was foreseen because the variances of the static and dynamic times were still high (239.9 and 194.8%). These two high values also imply their susceptibility to the overall recovery in the pause flow mode.

3.2. Stripping PR on copper surface

First of all, the improved SCF conditions for stripping PR from an Al surface were tried for PR on a Cu surface, yielding an average recovery of 32% ($n=3$, RSD=10.5%). A new L16 factorial design,

which is shown in Table 5, was generated to find the most suitable factors for stripping PR from a Cu surface in a pause flow mode.

Table 5 also shows the recovery contribution of each factor in the new factorial design, with optimum parameters as follows: 140 °C, oven temperature; 500 atm, CO₂ pressure; 5.0 min, static time; 5.0 min, dynamic time; 0.75 mL, EA spiking volume. It was apparent that the optimal SCF composition for PR removal from Cu was quite different from that for PR removal from Al. The higher temperature (140 °C vs. 120 °C) overcame the adhesion energy between PR and Cu layer and increased fluid diffusion. By contrast, the higher pressure (500 atm vs. 350 atm) and larger volume of EA spiking (0.75 mL vs. 0.2 mL) emphasized the importance of SCF solvating power. Even these parameters were driven to the extreme regions, the static equilibrium time was still long and unfavorable (5.0 min vs. 2.0 min). Because the longer equilibrium time implied limited solubility and/or limited diffusivity in the SCF medium, the calculated and experimental recoveries, 74.7 and 76.2% ($n=3$, RSD=7.5%), respectively, were also limited. A further attempt to modify the factorial design was invalid, as the values of variance between levels were all below 200. Use of more extreme factor values or changing the EA modifier may raise the PR stripping rate.

4. Conclusion

In the case of stripping PR from an Al layer, pause flow mode (which consisted of several alternating cycles of static equilibrium followed by dynamic purges) yielded far better extraction efficiency than continuous flow mode. The duration of equilibrium (static time) and number of extraction cycles (dynamic time) collectively determined the degree of extraction in pause flow mode. For those extractions with high recoveries, diffusivity of the optimized SCFs participated in the most parts of stripping mechanism rather than dissolubility of SCFs. Any other factorial factors, including oven temperature, pressure and EA volume, affected the viscosity of SCF medium, and thus the diffusivity. Although an imperfect recovery of 94.6% was obtained, refining the factorial design may yield a near-ideal extraction efficiency.

In the case of PR on a Cu layer, the experimental hardware was insufficient to allow either diffusion or dissolution control to dominate the stripping mechanism in a pause flow mode. Equipment upgrades and other modifiers are expected to break the strong PR-Cu bond and improve upon the present recovery limit of 76.2%.

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