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Glove Permeation by Propylene Glycol Monomethyl Ether Acetate — A Photoresist Solvent Used in Semiconductor Device Processing

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Propylene glycol monomethyl ether acetate (PGMEA) has been introduced as a replacement solvent for ethylene glycol ethers and ether acetates in photoresist formulations used in semiconductor processing. While PGMEA does not exhibit the adverse reproductive health effects found with the structurally related ethylene glycol derivatives, it is nevertheless readily absorbed through the skin, and proper protection against dermal absorption is warranted. This article describes the results of permeation tests of several glove materials commonly used in semiconductor fabrication facilities when challenged with PGMEA. Glove permeation was tested at 25 and 37°C according to the ASTM F739-85 permeation test method. The effects of repeated exposure and post-exposure thermal decontamination on permeation resistance were also examined for one type of glove. Of the gloves tested, those composed of butyl rubber afforded the highest level of protection against PGMEA, with no breakthrough being observed after more than 4 hours of continuous exposure at 25°C. Nitrile rubber gloves also provided good protection against initial exposure (breakthrough time > 2.4 hr at 25°C). Gloves composed of natural rubber or blends of natural rubber with other polymers provided significantly less protection (breakthrough times < 22 min at 25°C). Increasing the exposure temperature from 25 to 37°C did not affect the performance of the butyl rubber gloves. For the other gloves, however, decreases in breakthrough times by factors of 2 to 3 and increases in steady-state permeation rates by factors of 2 to 4 were observed. Repeated exposure of nitrile rubber samples indicated that the PGMEA is retained in, and diffuses through, the gloves long after exposure has ceased. Following an initial exposure for about one-half the nominal breakthrough time and air drying overnight at room temperature, vapors of PGMEA at concentrations greater than the breakthrough-time detection limit were detected permeating through glove samples. Much higher steady-state permeation rates were measured for the previously exposed samples as well. Heating previously exposed nitrile rubber samples at 70 or 100°C for 24 hours prior to retesting failed to restore the initial permeation resistance.

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Introduction

The production of semiconductor devices involves a series of processing steps performed on a wafer of silicon or some other semiconducting material.(1) One of the key steps is the microlithographic patterning of circuitry on the surface of the wafer. For most commercial devices this entails solvent-casting a thin film of a light-sensitive photoresist onto the wafer, followed by ultraviolet exposure through a mask having transparent and opaque regions that define the circuit pattern. A developer solution is used to remove the exposed (or unexposed) photoresist, leaving a positive (or negative) image of the pattern. Additional processing (e.g., etching, dopant deposition/diffusion, and metallization) is then performed on the exposed surface to create the desired circuit elements. In most cases, these steps must be repeated many times using a series of different mask patterns to produce the final product.

Ethylene glycol ethers and their ester derivatives, often referred to by their Union Carbide trade name Cellosolves®, historically have been the most common solvents used in positive photoresist formulations. Evidence of adverse reproductive effects in male and female animals exposed to these solvents(2,3) has prompted photoresist manufacturers to replace them with less toxic solvents.(4,5) Among the solvents that have emerged as useful replacements are derivatives of the propylene glycol ethers, such as propylene glycol monomethyl ether acetate (PGMEA).

Commercially available forms of PGMEA consist primarily of the so-called alpha isomer, 1-methoxy-2-propyl acetate. Limited animal toxicity testing of the analogous compound, 1-methoxy-2-propanol, indicates no significant adverse reproductive health effects.(4) The difference in toxicity between the ethylene glycol derivatives and the propylene glycol derivatives is attributed to differences in their metabolic pathways.(2,4) The former compounds are metabolized to the corresponding alkoxyacetic acids, which are thought to be the proximate teratogens. For the alpha isomers of the latter compounds this metabolic route is blocked and the initial product of metabolism, propylene glycol, enters the Krebs cycle and is converted to carbon dioxide.

One feature that PGMEA shares with its ethylene glycol predecessors is the ability to readily penetrate the intact skin. Thus, while PGMEA apparently exhibits a low level of toxicity, good industrial hygiene practice dictates the use of proper procedures to avoid dermal contact.

With respect to the potential for contact with processing solvents, available statistics indicate that dermatoses and other skin disorders attributable to chemical exposures have historically accounted for a significant fraction of all injuries and illnesses in the semiconductor industry.(6,7) While the use of automated equipment tends to minimize direct contact with processing chemicals in most large-scale manufacturing facilities, routine maintenance and solvent transfer operations can give rise to periodic exposures. Additionally, in research laboratories it is more common to find processing being performed manually.

Responding to the need for chemical protection in the specialized environments of semiconductor production facilities (i.e., clean rooms), several glove manufacturers have developed low particulate chemically protective gloves. A recent survey of glove use practices in this industry indicated that these types of gloves are used extensively.(8) The survey also revealed that the most common types of gloves used for protection against organic solvents were composed of nitrile rubber, although various other glove materials were also used. Most of the respondents indicated that gloves were used for up to several days before being replaced.

In a previous report from this laboratory, the permeation behavior of several glove materials challenged with semiconductor processing mixtures containing ethylene glycol–ether derivatives was described.(9) Among the results from that study, it was found that increasing the temperature from 25 to 37°C caused marked decreases in permeation resistance of most of the gloves. In addition, previously exposed gloves were found to retain the glycol–ether derivatives for up to several days following exposure, serving as a potential source of exposure to these solvents upon subsequent reuse. In light of these results and the limited amount of published information on the permeation behavior of PGMEA, we investigated the permeation of PGMEA through various gloves commonly used in semiconductor fabrication facilities.

Experimental

Table I lists the model, polymer component(s), and range of measured thicknesses of each type of glove tested. All gloves were used as received. PGMEA (99%, Aldrich, Milwaukee, WI) consisted of a mixture of alpha and beta isomers that could not be resolved under the gas chromatographic conditions used in this study.

Permeation tests were performed according to the American Society for Testing Materials (ASTM) F739-85 method(10), using a 5.1-cm (2-inch) diameter test cell (Pesce Lab Sales, Inc., Kennett Square, PA) in an open-loop configuration with nitrogen as the collection medium. The nitrogen flow rate was maintained at 0.5 L/min for all experiments. Components downstream from the test cell were constructed of Teflon® or stainless steel. A thermostatted water bath controlled the cell temperature to ±0.5°C. The test cell and the flask in which the PGMEA was stored were thermally equilibrated in the water bath prior to testing. The PGMEA was then transferred to the challenge side of the cell by cannula under a positive pressure of air. The temperature of the nitrogen gas stream was also regulated to ±1.5°C by heating a coiled section of the upstream tubing with a heating mantle prior to passing it through the test cell. The nitrogen temperature was monitored with a thermocouple inserted in-line just upstream from the cell.

Test samples cut from the gauntlet region of each glove were clamped into the cell and tightened to a constant pressure of 30 inch-lb with a torque wrench. Sample thicknesses were determined by averaging micrometer measurements at five locations across the sample.

The gas downstream from the test cell was sampled periodically with a 5-mL gas-tight syringe and analyzed with a gas chromatograph (Model 3700, Varian Associates, Palo Alto, CA) equipped with a packed column (2-ft stainless steel, ⅛-inch o.d., packed with 1 percent SP-1000 on 60/80 mesh Carbopack B, Supelco, Inc., Bellefonte, PA) and a flame-ionization detector. The output from the detector was monitored with a standard strip chart recorder and peak heights were used to quantify signal intensities. Instrument calibrations were performed daily using test atmospheres of PGMEA diluted in air in 100-L Tedlar® bags (SKC, Inc., Eighty-four, PA).

Steady-state permeation rates (SSPR) were calculated by averaging five measurements obtained after the downstream solvent concentration showed no further increase with time. The breakthrough time (BT) was defined as the time required for the downstream vapor

TABLE I. Permeation Test Results[A]

Glove Material	Polymer Components	L (cm)	BT(25°C) (min)	BT(37°C) (min)	BT Ratio (37°C:25°C)	SSPR(25°C) (μg/cm^2-min)	SSPR(37°C) (μg/cm^2-min)	SSPR Ratio (37°C/25°C)
North (B-161)	Butyl rubber	0.040	>250	>300	—	—	—	—
Edmont Nytek (49-155)	Nitrile rubber	0.041- 0.045	147(1) [87]	83(2) [40]	0.56 [0.46]	55(3) [2.3]	221(22) [9.9]	4.0 [4.3]
Pioneer Trionic	Natural/nitrile/neoprene blend	0.047- 0.048	16(2) [7]	6(0) [3]	0.38 [0.43]	61(0) [2.9]	221(11) [10.4]	3.6 [3.6]
Playtex (827)	Natural/neoprene blend	0.039- 0.042	19(3) [11]	6(0) [4]	0.32 [0.36]	81(7) [3.4]	256(4) [10.0]	3.2 [2.9]
Edmont Puretek (30-139)	Natural rubber	0.051- 0.053	22(1) [8]	10(0) [4]	0.45 [0.50]	58(0) [3.1]	151(1) [7.7]	2.6 [2.5]

[A] L = sample thickness. Numbers in parentheses are standard deviations. Numbers in brackets represent thickness-normalized BT (BT/L^2; units are 10^3 min/cm^2) and SSPR (SSPR × L, μg/cm-min) values at 25°C.

concentration to reach 4 mg/m^3, which corresponds to a permeation rate of 0.1 μg/cm^2-min for this test system. This concentration corresponds to an injected mass of 19 ng (for a 5-mL injection), which was well above the limit of detection for PGMEA (0.38 ng). Where necessary, BT values were interpolated from measurements collected just before and after the critical concentration was measured in the collected samples. For exposures at 37°C, the appropriate temperature correction was applied to the air concentrations of the injected samples. All permeation tests were performed in duplicate unless otherwise indicated.

For repeated exposures, samples were exposed initially, removed from the test cell and patted dry with a paper towel to remove visible liquid, and then allowed to air dry in an exhaust hood having a face velocity of 90-100 ft/min. Thermal decontamination was performed by placing exposed gloves in a vented oven at 70 or 100°C.

Results and Discussion

Table I shows the BT and SSPR values measured for each glove at 25°C. The butyl rubber gloves (North B-161) provided the highest level of resistance to PGMEA, with no breakthrough being observed over the 4-hour test period. Nitrile rubber gloves (Edmont Nytek®) also provided excellent permeation resistance (BT = 147 min). Gloves composed of natural rubber (Edmont Puretek®) and blends of natural rubber with other polymers (Pioneer Trionic® and Playtex 827) provided much shorter BTs. SSPR values were similar, and rather low, for all of the gloves exhibiting breakthrough, with values ranging from 55 to 81 μg/cm^2-min (see Table I).

At the conclusion of each permeation test qualitative assessments were made of the degree of deformation of the glove sample. With the exception of the North gloves, all of the gloves showed some degree of swelling, which persisted after removal from the cell. Interestingly, while the Nytek gloves showed the highest permeation resistance of these gloves, they also showed the most deformation after exposure. For experiments involving repeated exposure of the Nytek gloves, recovery of the shapes of the samples was evident after standing in a fume hood overnight.

Thickness-normalized BT and SSPR values are also presented in Table I (in brackets below the corresponding measured values). Thickness-normalized BT values were calculated by dividing the measured BT values by the square of the sample thickness, L.(11) Thickness-normalized SSPR values were calculated by multiplying the observed SSPR values by L. This latter quantity (SSPR × L) is equivalent to the permeability coefficient, P.(12)

Comparison of the thickness-normalized BTs and SSPRs for the different gloves emphasizes that the Nytek gloves have inherently greater permeation resistance than the other materials for which breakthrough was detected. It is interesting to note that the Playtex gloves, which contain roughly equal proportions of neoprene and natural rubber (according to the manufacturer), provide about the same permeation resistance as the natural rubber Puretek gloves. On the basis of solubility considerations, one might have expected the more polar neoprene to improve the permeation resistance of this material. A similar argument might be made for the Trionic gloves. However, according to the manufacturer of this glove, the product consists primarily of natural rubber with only traces of the other polymers added. Thus, the similarity between permeation behavior of the Trionic and Puretek gloves is not surprising.

Table I also presents the BT and SSPR values obtained at 37°C along with the ratios of these values to those obtained at 25°C. These temperatures were chosen because most operations involving potential exposure to positive photoresists are performed at room temperature. Thus, the glove temperature is likely to be between room temperature and body temperature.

With the exception of the North gloves, significant decreases in permeation resistance were observed at the higher temperature. BT values (and thickness-normalized BT values) decreased by factors of 2 to 3, with slightly larger effects observed for the Trionic and Playtex gloves than for the Nytek and Puretek gloves. SSPR values increased by factors of 2.6–4.0 (2.5–4.3 for thickness-normalized values). The influence of temperature on the SSPRs was greater than that on BT values in all cases. The largest effect on the SSPR was seen for the Nytek gloves. This result, together with the low SSPR observed at the lower temperature, is consistent with a higher activation energy for permeation.(13)

The rather large influence of temperature on the BT and SSPR values seen in this study is characteristic of the Arrhenius (i.e., positive exponential) temperature dependence of the permeation process. In this case, a change in absolute temperature of only 4 percent caused changes in these permeation parameters of 100–300 percent. The magnitudes of the temperature effects observed here are similar to those reported for other solvent/glove combinations.(13,14) These results point out the limitations of using data collected at 25°C as the basis for selecting gloves for use in hot work environments. Even for normal temperature working conditions, heat from the body may be sufficient to reduce the permeation resistance significantly. This also emphasizes the need for careful temperature control during permeation testing.

Following these initial tests, a series of experiments was performed to determine the effects of repeated exposure of the Nytek and North gloves to PGMEA. Given the relatively low resistance of the other gloves, they were not included in this second series.

The North samples continued to show excellent permeation resistance, with no evidence of solvent breakthrough following a 4-hour exposure, air-drying overnight at room temperature, and a 4-hour reexposure. Table II presents the BT and SSPR values for Nytek glove samples for an initial exposure of 1.3 hour (i.e., slightly greater than one half the nominal BT value) followed by one of several postexposure treatments and then reexposure. The mean BT and SSPR values for previously unexposed samples are also presented in Table II for comparison.

The first postexposure treatment examined was air-drying in a fume hood at room temperature (~23°C). Replacement of the test samples in the cell following this treatment resulted in concentrations of PGMEA in the collected samples, prior to filling the challenge side of the test cell with solvent, that were already above the BT detection limit. This result demonstrates that the PGMEA continues to diffuse through the glove during periods of nonuse. The SSPR values upon reexposure were about three times higher than those observed for the previously unexposed gloves, suggesting some alteration of the glove structure caused by the PGMEA (e.g., extraction of nonpolymer glove components, or nonreversible swelling of the polymer network).

TABLE II. Permeation Resistance of Preexposed Nytek Gloves

Exposure and Treatment Conditions^A	BT (min)	SSPR ($\mu g/cm^2$-min)
No preexposure	147(1)	55(3)
Preexposed (1.3 hr), dried 23°C (24 hr)	0(–)	165(3)
Preexposed (1.3 hr), dried 70°C (24 hr)	74(9)	182(11)
Preexposed (1.3 hr), dried 100°C (24 hr)^B	106(–)	200(–)

^A All exposures were performed at 25°C. Numbers in parentheses are standard deviations.
^B Only one trial was performed under these conditions.

It has been shown for solvents which penetrate the outer surface of a glove that thermal treatment is more effective for removal of residual solvents than surface cleaning with solvents or detergents.(15,16) Our initial attempts at thermal decontamination involved heating the previously exposed samples at 70°C. In this case, there was some recovery in the BT values, but SSPR values were still elevated relative to those obtained for previously unexposed gloves (see Table II). Raising the postexposure treatment temperature to 100°C resulted in BT values that were still only 75 percent of the expected value. SSPR values were even higher than those observed following the 70°C treatment. Apparently, elevated temperatures promote whatever degradation process is occurring in the glove.

The results obtained here can be compared to those obtained for the same glove materials in our previous study involving ethylene glycol monoethyl ether acetate (EGMEA), which is an isomer of PGMEA.(9) The relative order of permeation resistance exhibited by the gloves was the same for both solvents. That is, the North B-161 butyl rubber gloves provided the highest resistance, followed by the Edmont Nytek nitrile gloves and then the gloves containing natural rubber. For a given glove type, EGMEA consistently had shorter BT values and higher SSPR values than PGMEA. This is consistent with the smaller diffusion coefficient expected for the PGMEA as a result of the greater degree of branching in this isomer.(12) The permeation of EGMEA at 37°C was not examined for the neat solvent, but rather as a mixture (82 percent) with xylene (9 percent) and *n*-butyl acetate (9 percent) in a photoresist thinner formulation. Still, the decreases in BT values for the EGMEA at the higher temperature were similar to those observed for PGMEA. Increases in the SSPR values with temperature were somewhat smaller for the EGMEA than for the PGMEA.

As with the PGMEA, EGMEA was found to persist in the Nytek gloves following initial exposure and air-drying. For the EGMEA (in the mixture), however, it was possible to decontaminate the gloves by treatment at 70°C. Upon reexposure to EGMEA, the BT and SSPR values of the thermally decontaminated gloves were virtually the same as those of the previously unexposed gloves. Considering that both the vapor pressure and evaporation rate of PGMEA are higher than those of EGMEA(4), the difference in the thermal decontamination results is curious. Apparently the larger diffusion coefficient of the EGMEA coupled, perhaps, with the presence of the other solvents (which did enhance the permeation of the EGMEA) predominated in effecting evaporation of the EGMEA from the samples. The elevated SSPRs observed for PGMEA in the thermally treated previously exposed gloves, however, remains unexplained.

Finally, there remains the question of whether the results obtained for neat PGMEA are representative of those expected for photoresist formulations containing PGMEA. The nonsolvent components of the positive photoresists are present in concentrations ranging from about 15 to 35 percent by weight and consist of polymeric resins and photoactive compounds designed to promote dissolution upon exposure to ultraviolet radiation.(17) These components are high molecular weight and/or ionic compounds that would not be expected to permeate the gloves studied here.

Referring again to the results for EGMEA, one sees that the presence of the resins and photoactive components served to reduce the EGMEA permeation slightly. Given the similarities in the permeation of EGMEA and PGMEA (for initial exposure, at any rate), a similar reduction in the permeation of PGMEA in photoresist formulations, relative to the neat solvent, seems likely.

Summary and Conclusions

Of the gloves tested in this study, the North Model B-161 butyl rubber gloves provided the best protection against PGMEA. The BT was greater than 4 hours, and there was no evidence of persistent permeation or significant temperature effects from 25 to 37°C. While these gloves are not suitable for clean-room environments because of the need to apply powder to the gloves during packaging (to reduce the inherent tack of the butyl rubber), these gloves would be useful for emergency spill response or for handling microlithographic solvent formulations in service areas outside of the clean room.

The Edmont Nytek nitrile rubber gloves also provided good resistance to PGMEA under continuous exposure conditions. While the permeation resistance of the Nytek gloves decreased significantly from 25 to 37°C, the BT observed at the higher temperature (83 min) is long enough to provide adequate protection for many work situations. The Edmont Puretek (natural rubber), Pioneer Trionic (natural/nitrile/neoprene rubber blend), and Playtex Model 827 (natural/neoprene rubber blend) gloves showed significantly lower levels of protection against all of the solvent mixtures than the nitrile and butyl rubber gloves. BT values were all less than 22 minutes at 25°C and were less than 10 minutes at 37°C. These gloves may provide protection against incidental contact but should not be used for any length of time following exposure.

Of some concern are the persistent permeation of the PGMEA through the Edmont Nytek gloves after a relatively short initial exposure and evidence (i.e., significantly higher SSPR values) of a change in the structure of the glove as a result of the initial exposure. Heating previously exposed Nytek gloves at 70 or 100°C overnight led to only partial recovery of BT values (75 percent of that expected) and still higher SSPR values. Thus, thermally reconditioned Nytek gloves should be used for only limited periods following an initial exposure. Repeated thermal reconditioning cannot be recommended at this point.

With respect to the relative permeation resistance of the gloves investigated, the results observed for PGMEA are similar to those observed for its isomer, EGMEA, in a previous study.(9) The effects of temperature on permeation were also similar for the two solvents. However, thermal decontamination of previously exposed gloves, which was successful for EGMEA, was not successful for PGMEA even though PGMEA is more volatile. The persistence of PGMEA in the gloves may be attributable to its smaller diffusion coefficient relative to EGMEA. The marked increase in SSPR values for gloves previously exposed to PGMEA, which was not seen in the case of EGMEA, warrants further investigation.

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