

## Outgas Analysis for Wafer Industries

### Analysis of Trace Organic Contaminants on Silicon Wafer Surface

#### 1. Analytical Procedure

Figure 14 illustrates schematic diagram of the VOC collector for silicon wafer to be used for later studies.

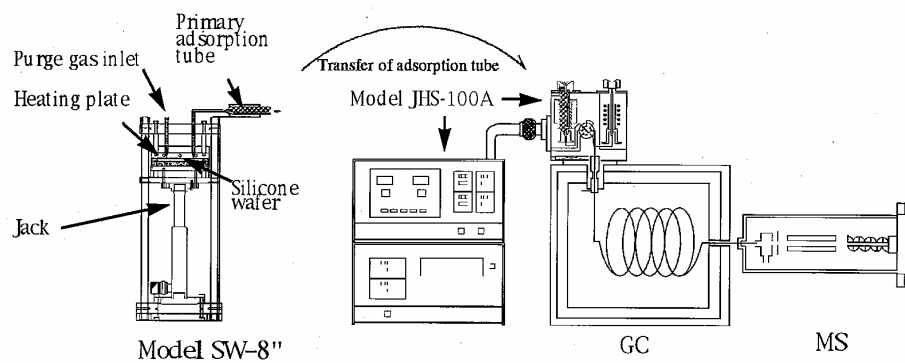


Fig. 14 schematic diagram of the Voc collector for silicon wafer

Model SW-8" is used to collect volatile organic compounds (VOC) generated from silicon wafer. After the VOC collection, the primary adsorption tube is moved to the Purge & trap (P&T) equipped with GC/MS. Characterization of VOC can be then performed. A silicon wafer of less than 8" dia. can be heated up to 500 .

A wafer was set on to the sample stage in which the sample stage and the heating plate were kept at room temperature. After raising the sample stage to the heating plate, the wafer is heated to generate VOC from one side, which is collected into the primary adsorption tube.

The primary adsorption tube is then transferred to the Purge & trap (P&T) which is equipped with GC/MS .

#### 2. Experimental

##### A) Sample

A 6" wafer was used for to prepare the calibration.

Also a new 8" wafer was used to measure organic contaminants at the manufacturing site for the wafer.

##### B) Condition of SW-8

The wafer was heated at 250 or 300 for 30 min by the sample stage and heating plate coated with gold. Helium was used as purge gas.

The primary adsorption tube : 2.5 g of Tenax was packed into the tube.

The transfer tube : Inert tube was used as inner tube, the temperature of the tube was kept at 250 .

C) Condition of JHS-100

Temperature of the primary adsorption tube : 300 for 15 min.

Oven temperature : 200 .

Adsorbent for secondary adsorption tube : quartz wool was used as adsorbent cooled at -60 during secondary trapping. After the secondary trapping, the adsorption tube was heated at 255 for 30 s by Curie point heating.

D) Condition of GC/MS (HP, model 6890/5973 )

Carrier gas : helium, 1.2 ml/min

Temperature of injection port : 250

Column : DB-5, 0.18 mm i.d. x 20 m, 40 (hold 3 min) to 250 , 10 /min.

Split ratio : 1/10

Scan speed : 1 scan/0.5 s.

### 3. Results and Discussion

#### 3. 1 Calibration for Organic Contaminants

20  $\mu\text{g}$  each of n-octadecane, n-eicosane, n-docosane and di-n-octyl phthalate (DOP) were dissolved into 200 ml of n-hexane for the standard solution. The solution was dropped by a micro-syringe onto room temperature of the wafer which was previously baked at 400 30 min.

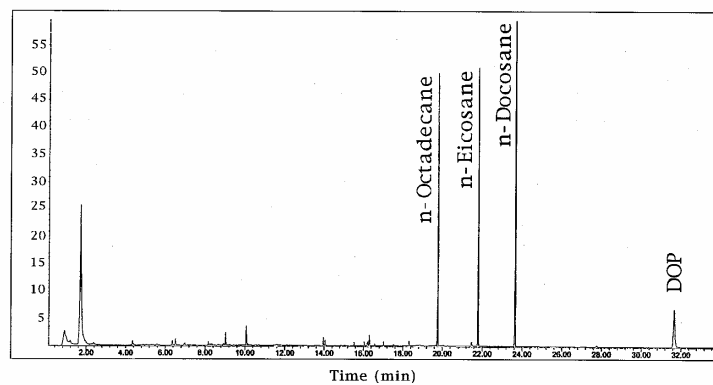


Fig. 15 A typical chromatogram obtained from standard solution which was dropped onto a wafer

Figure 15 shows a typical chromatogram. The peak areas (detector output) vs. amounts were summarized in Fig. 16 as the calibration curves.

n-Octadecane, n-eicosane and n-docosane peak show straight calibration. However, DOP shows curved calibration.

Moreover, the peak area of DOP is lower than that of n- paraffin.

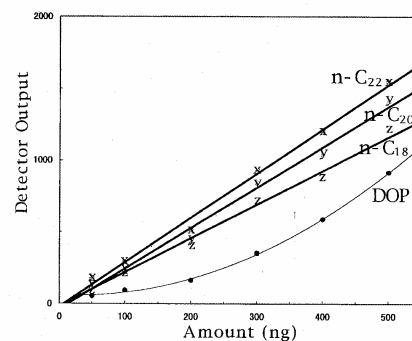


Fig. 16 Calibration curves for n-parafrin and DOP

From these facts, it is reasonable to postulate that DOP was decomposed during the measuringT process.

### 3.2 Quantitative Analysis of Alkyl Phthalates as Contaminants

Before the experimentation, a 6" wafer was heated in the oven of SW-8" at 400 for 10 hours in order to remove the contaminants. After that, the wafer was then left in the conference room (5 ) for 12 hours.

The wafer was set on the sample stage which was maintained at 20 . The stage was hydraulically raised to the heating plate (also 20 ). The stage and the plate were then closed tightly, that is the plate and processing surface for the wafer formed a wafer oven.

After that, organic contaminants on the processing surface for the wafer were thermally extracted by helium at 250 for 30 min, simultaneously vaporized organic contaminants were trapped into the primary adsorption tube.

Figure 17 shows a contaminants chromatogram obtained from a 6" wafer.

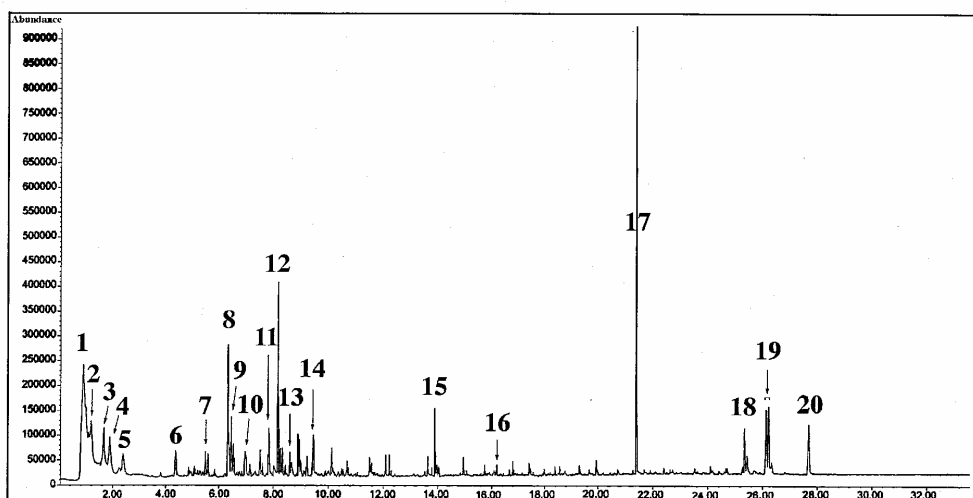


Figure 17 Contaminants chromatogram obtained from a 6" wafer

1 Carbon dioxide; 2: Acetone; 3: Acetic acid; 4: Chloroform; 5: Benzene; 6: Toluene; 7: Dimethyl pentanal; 8: 3-Methyl hexanal; 9: 5-Methyl hexanal; 10: Styrene; 11 : Heptenal; 12: 2-ethylhexanal; 13: Phenol; 14: 2-Ethyl hexanol; 15: Phthalic- acid anhydride; 16: BHT; 17: Dibutyl phthalate; 18: Di-2-ethylhexyl adipate; 19: Diheptyl phthalate; 20: Di-2-ethylhexyl phthalate.

The peak of dibutyl phthalate (DBP) was found to be the largest. The DBP originated from chair leather and wall material (PVC). Also, di-2-ethylhexyl adipate (DOA, chair leather), diheptyl adipate and di-2-ethylhexyl phthalate (Di-Et-hex P) were also detected.

Other compounds detected were 2-ethyl hexanol and Phthalic acid anhydride. They were generated by hydrolysis reaction with trace amount of moisture in the wafer from the Di-Et-hex P.

In addition, several kinds of aldehyde compounds were observed. It is postulated that the generated alcohols were oxidized by occluded oxygen in the wafer to aldehyde compounds.

Amount of the Di-E1-hex P on Fig. 17 was measured to be 320 ng by use the calibration of Fig. 16.

As a result the amount of the Di-Et-hex P could be measured with RSD of less than 4.6 due to the inert treatment of oven wall and inside wall of the transfer tube.