Adsorption Properties of an Organic Thin Film Prepared by an R.F. Sputtering with a Poly(Ethylene Terephthalate) (PET) Target for Volatile Organic Compounds (VOCs)

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Keywords : Volatile organic compounds (VOCs), Quartz crystal microbalance (QCM), R.f. sputtering, Poly(ethylene terephthalate) (PET)

Harmful volatile organic compounds (harmful VOCs), e.g., lacquer, thinner, solvent for paint, are used in a painting process in factories. Highly sensitive detection methods for these harmful VOCs are needed in working environment handling the VOCs. Simple and accurate detection methods for these VOCs are needed. The use of a quartz crystal microbalance (QCM) is one such method, and organic thin films prepared by plasma processes, i.e., sputtering and plasma enhanced vacuum evaporation, have complex molecular structures, and a potential to show characteristic adsorption properties for these harmful VOC vapors.

In this paper, we report on adsorption properties for these VOCs and water with two kinds of organic thin films deposited onto a quartz crystal by an r.f. sputtering with poly(tetrafluoroethylene) (PTFE) and poly(ethylene terephthalate) (PET) targets (sputtered PTFE thin film and sputtered PET film). Quartz crystal microbalance (QCM) method was used for the evaluation of the adsorption properties for these VOCs.

Fig. 1 indicates FT-IR spectra of pristine PET and sputtered PET thin film.

Peak number at the sputtered PET thin film dramatically decreased, and peak area in each peak becomes larger compared to the pristine PET. These results indicate that chemical structure of the sputtered PET thin film differs from the pristine PET.

We already reported that adsorption properties for low molecular weight VOCs such as aliphatic alcohols and acetone were analyzed with fluorocarbon thin films deposited onto a QCM by an r.f. sputtering and spin coating(1). The adsorption mass of the thin film prepared by the r.f. sputtering for the VOCs was much higher than that prepared by the spin coating. This high adsorption property would be owing to the chemical structures and low density of the thin film(1). It is considered that adsorption properties of the sputtered PET thin film differ from those of the pristine PET due to the difference of the chemical structure.

Fig. 2 indicates adsorption masses of the sputtered PTFE and PET thin films for toluene, acetaldehyde, ethanol, acetone and water. The frequency shift of the sputtered PTFE thin film due to the adsorption of the VOCs was larger than that of the sputtered PET thin film when acetaldehyde or toluene was used for the VOC. It suggests that the adsorption mass of these VOCs to the sputtered PTFE thin film was higher than that to the sputtered PET thin film. The adsorption amount of water of the sputtered PET thin film was higher than those of other VOCs, and response to the detection of water was excellent. Therefore, it is considered that the sputtered PET thin film can be used for reference sensor for water.

Fig. 1. FT-IR spectra of pristine PET and sputtered PET thin film.

Fig. 2. Adsorption masses of the sputtered PTFE and PET thin films for toluene, acetaldehyde, ethanol, acetone and water.

References

Effect of Bonding Pressure on the Strength of Cu/Cu Direct Bonding by Surface Activated Method

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Keywords: surface activated bonding method, room temperature bonding, Cu film, surface roughness, tensile strength, Ar ion beam

This paper describes Cu-Cu direct bonding by surface activated bonding (SAB) method at room temperature for the applications of 3D integration. In the SAB method, surface roughness has a strong influence on the bonding and metal bonding needs high bonding pressure in general. For successful bonding, it is important to reveal the influence of surface roughness and bonding pressure on the bonding properties. Therefore, we investigated the relationship between surface roughness and bonding strength with bonding pressure as the parameter.

We used specimens deposited thin Cu film for bonding experiments. The thickness of Cu film was 0.13 µm, 0.57 µm and 1.18 µm. The surface roughness of Cu film was measured by atomic force microscope. Fig. 1 shows the relationship between surface roughness and thickness of Cu film. The interpolated and extrapolated surface roughness of the respective thickness is shown in Table 1. Surface of these specimens were activated by Ar ion beam in high vacuum chamber (Fig. 2). After activating, immediately two specimens were mated in the same chamber and pressed. Two specimens have the same thickness of Cu film. Bonding pressure is 10 MPa, 50 MPa and 100 MPa. Table 2 shows the operation parameters for bonding experiments.

Bonding strength was measured using tensile test. Fig. 3 shows the relationship between bonding strength and surface roughness of Cu film with bonding pressure as the parameter. The bonded specimen, of which Cu film’s thickness was 0.13 µm, was successfully bonded by bonding pressure of 10 MPa. The bonding strength was about 15 MPa. However, both of specimens, of which Cu film’s thickness was 0.52 µm and 1.18 µm, didn’t achieve bonding. For bigger roughness than 1 nm Ra, the successful bonding needs bonding pressure of 50 MPa.

Table 1. Calculated surface roughness related to thickness of Cu film used in bonding experiment

<table>
<thead>
<tr>
<th>Thickness (µm)</th>
<th>Surface roughness (nm Ra)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.13</td>
<td>0.4</td>
</tr>
<tr>
<td>0.52</td>
<td>1.3</td>
</tr>
<tr>
<td>1.18</td>
<td>2.9</td>
</tr>
</tbody>
</table>

Table 2. Summary of bonding conditions

<table>
<thead>
<tr>
<th>Ion beam</th>
<th>Ar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Background vacuum pressure</td>
<td>about $8 \times 10^{-6}$ Pa</td>
</tr>
<tr>
<td>Beam energy</td>
<td>60 - 80 eV</td>
</tr>
<tr>
<td>Beam current</td>
<td>3.0 A</td>
</tr>
<tr>
<td>Irradiation time</td>
<td>15 min.</td>
</tr>
<tr>
<td>Load at bonding</td>
<td>10, 50, 100 MPa</td>
</tr>
</tbody>
</table>

Fig. 1. Relationship between surface roughness and thickness of Cu film

Fig. 2. Schematic illustration of bonding apparatus

Fig. 3. Relationship between bonding strength and surface roughness of Cu film. (■) are by bonding pressure of 10 MPa, (○) are by bonding pressure of 50 MPa, (▲) are by bonding pressure of 100 MPa
Production and Evaluation of MEMS Gas Sensor by All Dry Processes

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Keywords: MEMS, thin film, gas sensor, poly-Si, Pt electrode, Bosch process

The MEMS gas sensor was produced by all dry processes for the purpose of miniaturizing the sensor and reducing the consumed power. The heating properties and the gas response properties of this sensor were evaluated.

The MEMS gas sensor shown in the Fig. 1 was produced as the following three steps.
1. Forming a poly-Si heater by standard process
2. Forming a gas sensor and electrodes by lift off process
3. Forming a membrane by Bosch process

Fig. 2 shows the microscope image of MEMS gas sensor produced by the above method. As shown in the Fig. 2, the poly-Si thin film heater and the SnOx gas sensitive film were formed onto the membrane with the vertical silicon side wall.

The heating properties of the MEMS heaters were evaluated by measuring current-voltage properties using the value of temperature calibrated from the resistance-temperature properties of the poly-Si film. Fig. 3 shows the heating properties of the MEMS heater. As shown in the Fig. 3, this heater was heated up to 400°C at consumed power of 52.3 mW.

Fig. 4 shows the gas response properties of the MEMS gas sensor. As shown in the Fig. 4, 50 ppm ethanol gas was stably detected in smaller size and at lower-power consumption by the MEMS gas sensor than by the conventional bulk gas sensor.

Consequently, this MEMS gas sensor is useful for miniaturizing the sensor and reducing the consumed power.

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Fig. 1. Cross-section view of MEMS gas sensor.

Fig. 2. Microscope image of MEMS gas sensor.

Fig. 3. Heating properties of poly-Si film heater.

Fig. 4. Gas response properties of MEMS gas sensor.