Characterization of incoming PVA brush for 10nm below post CMP cleaning process

April 11, 2018

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Post CMP Cleaning

- Configurations of post CMP Cleaning Module

- **PVA Brushes!!**

  From Aion
Advantages of Brush Cleaning

- **High cleaning efficiency** due to its physical force by direct contact between brush and wafer surface.
- **Effective low cost of ownership (COO)**
- **Process flexibility with various solutions**
Defects from PVA Brush as a function of process time

- Defect Count Vs. Wafer Run No.

- Defect Level: Initial-stage > Last-stage >> Main Process

- An incoming brush shows higher defect level at initial-stage due to the presence of residual impurities inside the brush.

- Pre-treatment process (break-in process) for the removal of impurities from incoming brush is necessary before using.
**Break-in Process of Incoming Brush**

- **Effect of Brush Break-in Process**

![Break-in Process of Incoming Brush](image)

- Conventional break-in process applies DIW flow through method and scrubbing on dummy wafer to remove impurities from incoming brush.

- Break-in process significantly reduces impurities of new brush and increases efficiency of post CMP cleaning.

*Ref: Hong Jin Kim, Korea Cleaning UGM 2016*
Conventional Analysis Methods of Impurities

- Analysis of brush Impurities after DIW Flow Through Break-in

DIW Flow Through

- PVA Brush
- Residual Impurity

Impurity Capturing & Analysis

- Extremely Low Conc. Impurities
- Extracted Impurity

- Composition of impurities from incoming brush was not studied due to extremely low concentration of impurities in conventional break-in method.
Developed Analysis Methods of Impurities

- Analysis of brush Impurities after Ultrasonication process

- Developed characterization method using ultrasonication can extract impurities at higher concentration and make it possible to analyze the impurities.
Characterization Procedure and Equipment

1. ICP-MS
   - Impurity in brush manufacturing
   - Incoming brush
   - Impurity extraction with ultrasonic*
   *ultrasonic: 40 kHz, 600W

2. LPC (Liquid Particle Counter)
3. ICP-MS
4. LC-MS
5. FE-SEM
6. TOF-SIMS

Drying

Captured impurity in solution (DIW)

Captured impurity without solution

Si Wafer

### Characterization Procedure and Equipment

<table>
<thead>
<tr>
<th>Status</th>
<th>Equipment</th>
<th>Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet</td>
<td>Liquid Particle Counter$^2$</td>
<td>Number of particulate impurity</td>
</tr>
<tr>
<td></td>
<td>ICP-MS$^1, 3$</td>
<td>Type of Impurity (Inorganic)</td>
</tr>
<tr>
<td></td>
<td>LC-MS$^4$</td>
<td>Type of Impurity (organic)</td>
</tr>
<tr>
<td>Dry</td>
<td>FE-SEM$^5$</td>
<td>Shape, Size</td>
</tr>
<tr>
<td></td>
<td>TOF-SIMS$^6$</td>
<td>Type of Impurity (organic)</td>
</tr>
</tbody>
</table>

Wet: Measurement of impurities in solution  
Dry: Measurement of impurities after drying process
1. ICP-MS Analysis of Incoming Brush without Extraction

Analysis Procedure of Inorganic Impurity from Brush (Without Extraction)

- Impurity in brush manufacturing
- Inorganic impurities in brush w/o extraction process was analyzed by using ICP-MS analysis.
1. ICP-MS Analysis of Incoming Brush without Extraction

- **Analyzed Inorganic Impurities from Brush**

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (ug/g, ppm)</th>
<th>SD (Standard Deviation)</th>
<th>Relative SD (%)</th>
<th>Composition (%)</th>
<th>Total Amount (ug/g, ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>4278.596</td>
<td>157.878</td>
<td>3.690</td>
<td>88.650</td>
<td>4,826</td>
</tr>
<tr>
<td>Ti</td>
<td>523.721</td>
<td>25.080</td>
<td>4.789</td>
<td>10.851</td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>0.036</td>
<td>0.002</td>
<td>4.162</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>14.118</td>
<td>0.672</td>
<td>4.764</td>
<td>0.293</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>9.916</td>
<td>0.751</td>
<td>7.575</td>
<td>0.205</td>
<td></td>
</tr>
</tbody>
</table>

- ICP-MS analysis shows the presence of **Si** residues in an incoming brush.
- An incoming brush contains high level of **Si based impurity**.
Most of the particulate contaminants were extracted completely within 10 min.

- Particle size range: $X$ nm ~ 4 um

- Ultrasonication is very effective and fast process to capture the impurities from the brush.
3. ICP-MS Analysis of Extracted Solution

- Analysis Procedure of Inorganic Impurities from Extracted Solution

- Inorganic impurities with ultrasonically extraction process was analyzed by using ICP-MS analysis.
3. ICP-MS Analysis of Extracted Solution

ICP-MS results of Extracted Solution from Brush

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (ng/mL, ppb)</th>
<th>Relative SD (%)</th>
<th>Composition (%)</th>
<th>Total Amount (ng/mL, ppb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>35.355</td>
<td>2.8</td>
<td>74.11</td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>3.422</td>
<td>4.3</td>
<td>7.17</td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>2.403</td>
<td>2.3</td>
<td>3.36</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>4.924</td>
<td>1.8</td>
<td>10.32</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>1.602</td>
<td>2.4</td>
<td>5.04</td>
<td></td>
</tr>
</tbody>
</table>

ICP-MS analysis confirms the presence of Si impurities in ultrasonically extracted solution of incoming brush.
4. LC-MS Analysis of Extracted Solution

- Siloxane peaks were observed from extracted solution.
- This LC-MS result is well matched with ICP-MS results.
5. FE-SEM Measurement of Dried Sample

FE-SEM Images of Impurities after Drying Process

- Organic contaminants and particles were observed.
- Density: organic contaminants >> particles
5. FE-SEM Measurement of **Particles**

- **FE-SEM Images of Particulate Impurities**

  - **X 100k**
    - 258 nm
    - 500 nm
  - **X 50k**
    - 589 nm
    - 1 μm
  - **X 50k**
    - 1.06 um
    - 1 μm
  - **X 20k**
    - 3.7 um
    - 2 μm

- **Particle size range**: 200 nm ~ 4 μm
- **These FE-SEM results are well matched with LPC results.**
5. EDX Analysis of Contaminants

- **Uncontaminated Area**
  - EDS Quantitative Results
  - | Element | Wt% | At% |
  - | CK | 100.00 | 100.00 |

- **Contaminated Area**
  - EDS Quantitative Results
  - | Element | Wt% | At% |
  - | CK | 43.34 | 64.14 |
  - | SiK | 56.66 | 35.86 |

- **Contaminated particle**
  - EDS Quantitative Results
  - | Element | Wt% | At% |
  - | CK | 61.60 | 73.30 |
  - | OK | 18.62 | 16.64 |
  - | SiK | 19.78 | 10.07 |

- **Contaminated Area**
  - EDS Quantitative Results
  - | Element | Wt% | At% |
  - | CK | 61.38 | 74.60 |
  - | OK | 13.57 | 12.38 |
  - | SiK | 25.06 | 13.02 |

- Organic contaminants and particles show carbon peak.
6. TOF-SIMS Analysis of Dried Sample

- **Siloxane** peaks were observed from incoming brush after dried on Si wafer substrate.
- This TOF-SIMS result is well matched with ICP-MS and LC-MS results.

Siloxane: Organosilicon with Si-O-Si linkage

Si backbone
Carbon side chain
FE-SEM Images of **Ultrasonicated Brush**

- **Initial**
  - 500x magnification
  - 100 µm scale
  - 5 kx magnification
  - 10 µm scale

- **Ultrasonic treat.**
  - 500x magnification
  - 100 µm scale
  - 5 kx magnification
  - 10 µm scale

- **Ultrasonic treat.**: 6 hr in DIW

- **No noticeable damages** were observed from ultrasonically characterization process
Porosity Measurement of Ultrasonicated Brush

Porosity % = \( \frac{B - A}{(B - A) + \frac{A}{D_{\text{pva}}}} \)

- **A**: empty weight of the brush
- **B**: weight of the brush after soaking in water
- **\( D_{\text{pva}} \)**: density of the PVA (1.3 g/cm\(^3\))

- Porosity of PVA brush was not changed after ultrasonication for 6 hours.
PVA brush was dipped in 1wt.% TMAH sol. to observe the effect of cleaning chemicals (TMAH) on impurity generation from brush.
Effect of TMAH chemistry on PVA brush

- FE-SEM Images of Organic Residue from Brush Dipped in 1wt.% TMAH

- Brush dipped in 1wt.% TMAH solution shows organic residue contamination.
1. In general, organic residues were observed in metal (especially Cu) CMP process.

2. Cleaning chemical of Cu CMP include TMAH.

3. TMAH has high dissolution ability of siloxane.

4. Siloxane can be delivered from inside of brush to metal surface due to cleaning chemical.
Summary

1. **Ultrasonication** is very effective and fast method to characterize the PVA brush.

2. Incoming PVA brush contains 2 types of impurities (*soluble and insoluble*).

3. Soluble impurities such as **siloxane** may create organic residues.

4. Insoluble impurities such as **PVA debris** may create particle residues.

<table>
<thead>
<tr>
<th></th>
<th>Soluble Impurity</th>
<th>Insoluble Impurity</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Composition</strong></td>
<td>Siloxane</td>
<td>Weakly bonded PVA debris</td>
</tr>
<tr>
<td><strong>Shape</strong></td>
<td>Thin and circular organic residue</td>
<td>Particle</td>
</tr>
<tr>
<td><strong>Analysis Method</strong></td>
<td>ICP-MS, TOF-SIMS, LC-MS</td>
<td>LPC, FE-SEM, TOF-SIMS</td>
</tr>
</tbody>
</table>
Summary

5. **Soluble impurities may be a root cause of organic residues** after post CMP cleaning process.

(a) Organic Residue from Brush

(b) Organic Residue after post Cu CMP Cleaning

*Courtesy from GlobalFoundries*

6. **Siloxane can be delivered** from inside of brush to **metal surface due to etching ability of cleaning chemical**.
THANK YOU FOR YOUR ATTENTION