CMP Waste Treatment: Electrophoretic Cross-Flow Filtration

(Subtask A4-1)

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Outline

• Significance of CMP waste problem
• Principles of electrophoreotic cross-flow filtration
• Illustration of apparatus
• Electrophoreotic filtration of silica suspensions
• Electrophoreotic filtration of dissolved copper
• Power consumption of electrophoreotic filtration
• Summary of results and future work
Significance of CMP Waste Problem
Single fabrication plant produces 200 GPM of CMP effluent†

† Maag, Benoit, “Copper CMP Effluent Flow in a Semiconductor Facility”, ERC TeleSeminar, April 6, 2000

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Waste Characteristics versus Environmental Regulations

- Effluent contains approximately 500-5000 ppm TSS and 5-50 ppm Cu‡
- Environmental regulations require that effluent be reduced to <5 ppm TSS and 0.1-2 ppm Cu before it may be discharged to waste treatment system‡


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Principles of Electrophoretic Cross-Flow Filtration
Cross-Flow Filtration

- Cross-Flow Filtration reduces membrane fouling (filter cake build-up) by continuously sweeping the membrane surface.
- Such action extends the life of the membrane and helps sustain flow of permeate through the membrane.
Cross-Flow Filtration with Electric Field

- An electric field enhances cross-flow filtration process by electrophoretically driving particles away from filter surface.
- Thus, filter cake is suppressed and filter effectiveness maintained.

V_f - direction of flow of permeate water
V_p - direction of electrophoretic migration of particles
Filter Cake Prevention

- The upward slope of several of these curves illustrates the decrease in filtration rates resulting from filter cake build-up.

- The slope of the lines decreases with applied voltage.

- This is evidence of decreased rate of filter cake deposition due to electrophoretic transport of particles away from the filter surface.

Plot T/V versus V for clay suspension of 450 mg. Clay (200 mesh) and 450 mg. NaCl per liter

Reasons for Investigating the Technology

• An electric field suppresses filter cake build-up and enhances cross-flow filtration.

• An electric field biases the transport of copper and other solutes.
Electric Field Biases Transport of Cu

- $V_f$ - direction of flow of permeate water
- $V_p$ - direction of electrophoretic migration of particles

**Diagram**
- Filter Membrane
- $E$ - electric field
- $V_f$ - permeate flow
- $V_p$ - electrophoretic migration
- Cu$^{2+}$
- Retentate
- Permeate
Illustration of Apparatus
Single Cell

CMP aqueous waste suspension

Constituents:
- Solids (0.05-0.5 w/v %)
  e.g. Silica (80 nm)
- Copper
  Copper ions (5-50 ppm)
  Copper complexes
- Copper chelating agents
  e.g. Citric Acid
- Corrosion inhibitors
  e.g. BTA
- Conductivity
  300-1500 µS/cm

Permeate

V_f - direction of flow of permeate water
V_p - direction of electrophoretic migration of particles
Expanded Cell Assembly

A  End plate with Electrode
B  Dialyzing Membrane
C  Input Spacer for 1st Cell
D  Micro Filter (0.8 µm)
E  Output Spacer for 1st Cell
F  Dialyzing Membrane
G  Input Spacer for 2nd Cell
H  Micro Filter (0.8 µm)
I  Output Spacer for 2nd Cell
J  Dialyzing Membrane
K  Input Spacer for 3rd Cell
View of Endplate with Electrode

Outside View of Endplate
- Permeate Outlet
- Pressure Tap
- Retentate Outlet

Side View of Endplate
- 2.2 cm
- 2.9 cm

Inside View of Endplate
- Pt Electrode
- Permeate Outlet
- Retentate Outlet
- 0.64 cm
- 6 cm
- 10.5 cm
- 19.5 cm
- 33.5 cm
- 16.5 cm
- 24.5 cm
Front View

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Entire Device
Components of Device

• Electrodes - platinum

• Membranes - dialyzing cellophane

• Filter membranes - \( \sim 0.8 \, \mu m \) porosity
Electrophoretic Filtration of Silica Suspensions

- Oxide CMP Waste
- Model Silica Suspensions
- Model Silica Suspensions doped with Copper
Electrophoretic Filtration of Oxide CMP Waste

(Oxide CMP waste provided by: Microelectronics Lab, U of A)

Initial Waste Characteristics:
- Turbidity: 2300 NTU
- Concentration: ~4500 ppm
- pH: 7.6
- Conductivity: 110 µS/cm
- Particle size: ~80 nm

95% removal

Electric Field (volts/cm)
Electrophoretic Filtration of Silica Suspension
(Klebosol Colloidal Silica)

Initial Waste Characteristics:
- Concentration of SiO₂: 1000 ppm
- pH: 6.0
- Conductivity: 600 μS/cm
- Particle Size: 80 nm

20% removal
91% removal
Electrophoretic Filtration of Silica Suspension Doped with Copper

Initial Waste Characteristics:
- Concentration of Cu\(^{2+}\): 17 ppm
- Concentration of SiO\(_2\): 1000 ppm
- pH: 5.5
- Conductivity: 700 \(\mu\)S/cm

- >72% Cu removal
- >93% SiO\(_2\) removal
Electrophoretic Filtration (Electrodialysis) of Dissolved Copper
Electric Field Biases Transport of Cu

- $V_f$ - direction of flow of water
- $V_{p}$ - direction of electrophoretic migration of particles

Retentate

Permeate

$\text{Cu}^{2+}$
Electrophoretic Filtration (Electrodialysis) of Copper

Initial Waste Characteristics:
- Concentration of Cu$^{2+}$: 17 ppm
- pH: 5.5
- Conductivity: 650 $\mu$S/Cm

>73% removal
Power Consumption of Electrophoretic Filtration
Power Consumption of Electrophoretic Filtration of Silica

Initial Waste Characteristics:
- Concentration of SiO$_2$: 1000 ppm
- pH: 5.4
- Conductivity: 700 $\mu$S/cm
- Particle Size: 80 nm

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## Power Consumption:
Mechanical Filtration versus Electrophoretic Filtration

<table>
<thead>
<tr>
<th>Filter Type</th>
<th>Power Equation</th>
<th>Power Consumption</th>
<th>Power Consumption/Permeate Flow Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical Filtration (Ultrafiltration)</td>
<td>Q x DP</td>
<td>17 Watts</td>
<td>8 MJ/m³</td>
</tr>
<tr>
<td>(with 45% pump efficiency)</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Electrophoretic Filtration of Simulated Copper CMP Waste (conductivity = 700 µS/cm)</td>
<td>V x I</td>
<td>50 Watts</td>
<td>32 MJ/m³</td>
</tr>
<tr>
<td>(Electric Field of 20 volts/cm)</td>
<td></td>
<td>(Electric Field of 20 volts/cm)</td>
<td></td>
</tr>
<tr>
<td>Electrophoretic Filtration of Oxide CMP Waste (conductivity = 100 µS/cm)</td>
<td>V x I</td>
<td>2.5 Watts</td>
<td>4 MJ/m³</td>
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<tr>
<td>(Electric Field of 10 volts/cm)</td>
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<td>(Electric Field of 10 volts/cm)</td>
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Summary of Results

In the suspension studied thus far:

• Electrophoretic filtration readily removes more than 90% of silica particles from CMP suspension

• Electrophoretic filtration removes approximately 75% of copper from the permeate stream
Potential Applications

1. Use immediately before standard ultra-filtration and ion-exchange processes to pre-filter CMP waste.

2. Use immediately after mechanical filtration process to further filter concentrated effluent.
Electrophoretic Filtration of Concentrated CMP Suspension

![Graph showing electrofiltration of concentrated oxide CMP](image)

- Turbidity of Initial Concentrated CMP Suspension (NTU)
- Turbidity of Permeate/Initial CMP Suspension
3. Small footprint enables each CMP tool to have its own individual filtration process.
Future Work

• Add chelating agent to the model copper suspension
• Concentrated silica suspensions (e.g. CMP sludge)
• Experiment with actual copper CMP waste (need samples !!!)
• Experiment with other types of filters