

## USING THE COULTER PRINCIPLE TO QUANTIFY PARTICLES IN AN ELECTROLYTIC SOLUTION FOR COPPER ACID PLATING

### INTRODUCTION

Acid copper plating is one electrochemical process for manufacturing printed circuit boards. The action of the ionic current in an electrolytic copper plating tank is such that, during plating, any and all suspended particulates will be forced toward the cathode (the panel that you are plating) and deposited. If the particles are large enough (5 to 20 microns), their presence can disrupt the local electrical field enough to reduce the effectiveness of the levelers present in the electrolyte. The net result of this "electrophoretic" migration is rough plating with the contaminating particles trapped in the electrolytic layer right on top of a critical circuit element. In a production setup, the plating electrolyte is usually filtered continuously using one or more 1 micron filters specially made for "polishing" electro-chemicals.

### MATERIALS AND METHODS

#### Instrument Set up and Calibration

A 100  $\mu\text{m}$  aperture tube was used for the analysis. The linear dynamic range for any aperture is 2% to 60% of its diameter. A 100  $\mu\text{m}$  aperture tube will be capable of analyzing the particle concentration and size distribution from 2  $\mu\text{m}$  to 60  $\mu\text{m}$ . If a different size range is needed, another aperture tube could be used.

The instrument was calibrated according to the Multisizer 3 Operator's Manual. For determining concentration and size distribution of particles in the sample, the results were obtained in number/mL. The Size Interpolation feature in the software was used to quantify the number of particles at different size levels. The control mode for the instrument was Volumetric Mode selecting 500  $\mu\text{L}$  as the run volume.

#### Procedure

##### 1. Electrolyte

As the sample is a conductive solution, it was analyzed with no further dilution into another electrolyte. For the external electrolyte jar, 0.45 $\mu\text{m}$  filtered sample was used.

In the sample information dialog of the Multisizer 3 software the information below was entered:

Sample Volume: 150 mL

Electrolyte Volume: 0 mL

Analytical Volume: 500  $\mu\text{L}$

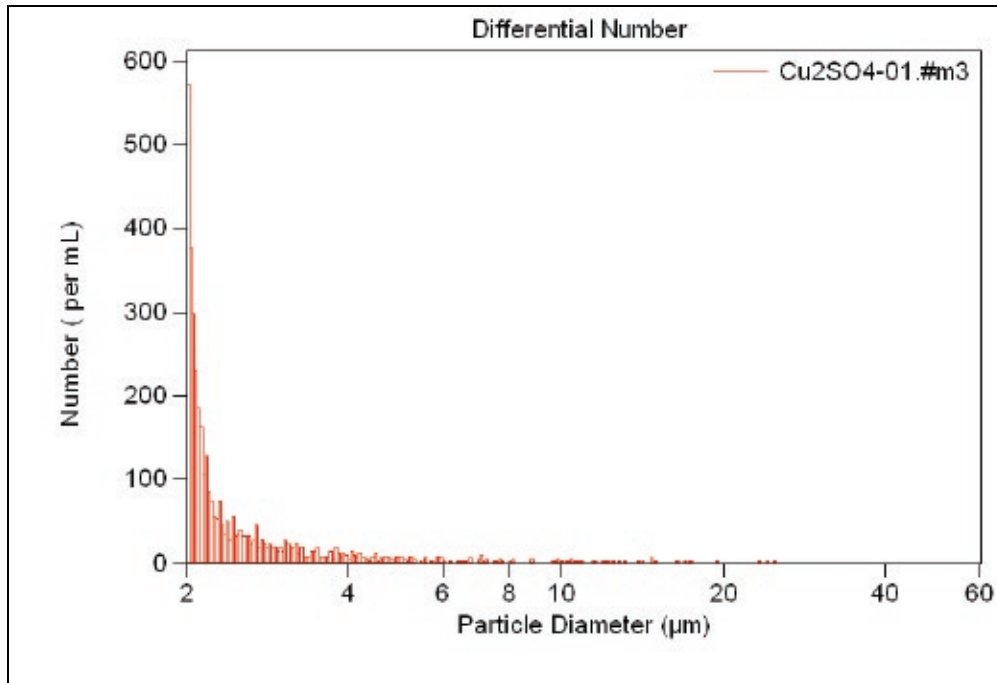
## 2. Sample Analysis.

### a) Running the sample

A round bottom beaker containing the sample preparation was analyzed using a 100  $\mu\text{m}$  aperture. The aperture tube was flushed before each analysis. After each run the aperture and electrode were rinsed before proceeding to the next sample.

## RESULTS

The results are expressed in particles/mL. The graph below shows the size distribution of one sample.



**Particle Concentration:** 3,758 particles/mL larger than 2 $\mu\text{m}$ . To show the repeatability of the method, the same sample was analyzed five consecutive times. Table 1 shows the results.

	<b>Particles</b>	<b>Mean</b>	<b>d10</b>	<b>d50</b>	<b>d90</b>
<b>Run</b>	<b>per mL</b>	<b><math>\mu\text{m}</math></b>	<b><math>\mu\text{m}</math></b>	<b><math>\mu\text{m}</math></b>	<b><math>\mu\text{m}</math></b>
1	3,758	2.73	2.01	2.15	3.80
2	3,294	2.73	2.02	2.16	3.71
3	3,654	2.69	2.02	2.14	3.50
4	3,744	2.77	2.01	2.13	3.70
4	3,714	2.72	2.02	2.14	3.57
<b>Aver.</b>	<b>3,633</b>	<b>2.73</b>	<b>2.02</b>	<b>2.14</b>	<b>3.66</b>
<b>C.V.</b>	<b>5.3%</b>	<b>1.0%</b>	<b>0.0%</b>	<b>0.6%</b>	<b>3.2%</b>

**Table 1** Repeatability of results

In addition to the total concentration of particles, by using the Interpolation Points feature in the software, it is possible to determine the concentration of particles above pre-set size levels.

Particles per mL larger than	Diameter ( $\mu\text{m}$ )
3,758	2
668	3
323	4
187	5
58.	10
1	25
0	50

## CONCLUSIONS

As the Coulter Principle is the highest resolution technology available for sizing and counting particles, it is an excellent tool for monitoring cleanness of the electrolytic solution in the copper plating process processes.

The procedure described here may also be used to evaluate filtration efficiency during the cooper plating.

