

A Primer on Slurries

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Introduction

Slurry is a suspension of particulate solids in a liquid. For semiconductor manufacturing, slurries are used in Chemical Mechanical Planarization (CMP) as abrasives for polishing silicon wafers.

Slurries may be named for the type of metal oxide particles (*i.e.* "silica" or "ceria") or the type of substrate removed (*i.e.* "copper"/"copper barrier" or "tungsten"). At times, other names (such as "colloidal silica") are used to distinguish a particular aspect of the slurry manufacturing process.

Slurry Composition

Most slurries are composed of metal oxide particles and a mix of additives. The particulate matter serves as the abrasive during CMP processing while the additives exist both to provide the chemical component of material removal and to maintain the quality of the slurry. In particular, there are two main types of additives: surfactants and pH buffers. Both of these attempt to prevent agglomeration and settling of the particles.

Colloquial Name	Primary Metal Oxide
Oxide	Silica
Colloidal, Precipitated or Fumed Silica	Silica
Copper or Copper Barrier	Silica
Tungsten	Silica
Ceria	Ceria
Alumina	Alumina (can be different crystal forms)
Iron	Mixed Iron Oxides
Zirconia	Zirconia

Particle Size Distributions

Slurry particle size distributions tend to be log-normal – in other words, the logarithm of particle size is Gaussian (or fits to a normal distribution). In strictly statistical terms, the mode is the most probable value and, therefore, the peak of a distribution. However, within the semiconductor industry, the mode is often referred to as the "mean mode." The true mean or average particle size lies to the right of the mode due to the extended tail on the right hand side of the distribution.

As indicated on the diagram below, the mode can have as many as 10^{15} particles per milliliter (or more depending on the slurry type), and the tail above 1 micron may contain as many as 10^4 - 10^6 particles per milliliter.



Figure 1: Typical Particle Size Distribution for Silica Slurries¹

Relation to CMP Processing

In general, it is the bulk particle distribution that results in substrate removal during CMP processing. Larger particles, however, can cause scratching on the wafer. The exact correlation between particle size and concentration with wafer scratching has not been rigorously studied, in part due to the lack of metrology tools available. Some reports indicate that particles greater than 1 μ m cause scratching while other reports suggest that the minimum size to cause scratching is 0.5 μ m. No reports have been found which address minimum concentration levels for any particle size to induce scratching.

To prevent large particles from contacting the pad/wafer, CMP tools contain point-of-use filters, with size levels from 0.2 μ m to 1 μ m. However, SlurryScope has shown using undiluted measurements that these filters are not 100% efficient and that large particles in the slurry tail still persist after the filter.

Important Considerations for SlurryScope

The lower size limit for SlurryScope is 1.0 μ m, but that does not mean that sub-micron particles do not effect measurements. These small particles do scatter light – instead of creating signal, though, the high concentration of particles below 1 μ m creates a uniform background for which SlurryScope software can compensate.

Undiluted measurements allow for accurate characterization of the LPC tail for two reasons. First, there are no errors introduced by dilution. Consider the following thought experiment: if there is a solution of 100 particles per mL and you diluted it 100-fold and then measured 1 mL, what is the probability that you would measure 1 particle (yielding the correct

¹ Vasilopoulos, G. et al. "Techniques for evaluating particles in CMP slurries." <u>Semiconductor Online</u>. 26 Dec 2000. http://images/6324AEB8-D8FF-11D4-A76E-00D0B7694F32/vasil1.jpg

result after back-calculation)? In fact, the answer is \sim 36.8%; surprisingly, this is also the probability of measuring **zero** particles in this scenario. Another key source of error is that dilution changes the chemistry of the slurry. Most notably, the concentration of the additives decreases and the pH shifts towards 7 (though this can be alleviated with buffered diluents, which are less common than they should be) – both of these effects can cause agglomeration and change the appearance of the LPC tail.

Many customers press for a lower size limit since existing dilution systems size particles as small as 0.5 μ m. While we continue to develop enhanced functionality for our product, we remind our customers that our system is still detecting $10^4 - 10^6$ particles per mL above 1 μ m after their filters, a problem that can be immediately addressed with our undiluted and continuous technique. Furthermore, there is no data that conclusively shows that particles smaller than 1 μ m cause scratching (studies that claimed such results extrapolated down to 0.5 μ m).