

SEMI C98-1219

GUIDE FOR CHEMICAL MECHANICAL PLANARIZATION (CMP) PARTICLE SIZE DISTRIBUTION (PSD) MEASUREMENT AND REPORTING USED IN SEMICONDUCTOR MANUFACTURING

This Standard was technically approved by the Liquid Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on August 19, 2019. Available at www.semiviews.org and www.semi.org in December 2019.

1 Purpose

1.1 The purpose of this Document is to provide a guide for the measurement of and reporting of particle size distributions (PSD) for chemical mechanical planarization (CMP) slurries. Most CMP slurries have a wide range of particle sizes from a few nanometers to several hundreds of nanometers. This Guide does not define which measurement technique should be used but focuses on the parameters to report on a Certificate of Analysis (CoA) and on the roadmap for the development of new semiconductor technologies. Reference to this Guide can be used in the corresponding CoA from a CMP supplier to its customers.

2 Scope

2.1 This Document provides guidance on how CMP PSD should be reported on a CoA.

2.2 The Related Information section provides extensive background information on several topics:

- What a full CoA for CMP slurries should contain (not just PSD).
- The fundamentals for measuring any PSD with specific references to what should be reported for CMP slurries.
- CMP sample preparation.
- Details for four CMP PSD measurement techniques together with the advantages and disadvantages of each technique.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Due to the proprietary nature of CMP slurries this Guide cannot recommend a specific sample preparation before a particular measurement technique. Some slurries are best measured for PSD with one metrology that is inappropriate for other slurries.

3.2 Typical CMP slurries use a wide variety of processing concentrations. Depending on the metrology selected, sample dilution with ultrapure water (UPW) may be required. Sample dilution may change the size distribution measurement.

3.3 Small particle size slurries are commonly quite dilute with low overall percentage of solids and therefore are not ideal for PSD-type measurements using light scattering techniques.

3.4 This Guide does not comment on the advantages or disadvantages of a specific methodology to measure width of a PSD distribution.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C96 — Test Method for Determining Density of Chemical Mechanical Polish (CMP) Slurries

SEMI F63 — Guide for Ultrapure Water used in Semiconductor Processing

4.2 Other Documents

ISO 9276-2:2014 — Representation of Results of Particle Size Analysis – Part 2: Calculation of Average Particle Sizes/Diameters and Moments from Particle Size Distributions

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *CMP* — chemical mechanical planarization

5.1.2 *CoA* — Certificate of Analysis

5.1.3 *CPC* — condensation particle counter

5.1.4 *DLS* — dynamic light scattering

5.1.5 *DMA* — differential mobility analysis

5.1.6 *EM* — electromagnetic

5.1.7 *FWHM* — full width half max

5.1.8 *LED* — light emitting diode

5.1.9 *MPS* — mean particle size

5.1.10 *PoU* — point of use

5.1.11 *PSD* — particle size distribution

5.1.12 *UPW* — ultrapure water

5.2 Definitions

5.2.1 *Dxx* — Diameter of “xx” percent of the total distribution of particles measured

6 Background to Data Reporting of CMP PSD

6.1 An overview of all the data that should be reported on a CoA is presented in Related Information § R1-1. Multiple metrologies to measure and report parameters of PSD exist on the market. The usefulness and practicality of these metrologies depend on the specific application and properties of the slurry being measured.

6.2 PSD metrologies are based on various measurement principles, such as dynamic light scattering (including back, forward and side scattering), static light scattering, nano-tracking, BET (based on Brunauer-Emmett-Teller adsorption theory), Nebulization followed by Dynamic Mobility Analysis, optical blocking plus centrifugation, Nuclear Magnetic Resonance, Single-angle X-ray Scattering, stroboscopic techniques, capillary fractionating, SEM, etc.

6.3 PSD metrologies can report a variety of PSD parameters. An overview of PSD statistics is presented in Related Information § R1-2. Commonly reported parameters are mean particle size (MPS) and peak width.

6.4 Calculations of these parameters can be done using different signals, such as intensity of the reflected light, particle volume, or particle number.

6.5 Calculations of the PSD parameters depend on metrology software and how the software uses abrasive refractive index, diluent viscosity, etc.

6.6 PSD parameters depend on test conditions, such as environmental temperature, measurement duration etc.

6.7 PSD parameters depend on analytical sample preparation, of which sample dilution may be critical and have a profound effect on the measured PSD.

7 The Challenges of Data Reporting for the PSD of CMP Slurries

7.1 A limited number of PSD parameters are currently reported on CoA.

7.1.1 The most common parameter reported on CoA is an MPS. However, MPS alone does not adequately describe PSD for slurries.

7.1.2 A CMP slurry is frequently made from several different abrasive materials. Each material can have its own PSD, resulting in a bimodal or trimodal PSD. The numbers of modes are typically not reported, rendering an MPS for the entire PSD virtually meaningless unless the slurry is monomodal.

7.1.3 For multimodal CMP slurries, parameters for each observed peak should be measured and reported.

7.1.4 Measurement metrologies, test conditions, and sample preparation are not currently reported.

8 Response to Semiconductor Technology Advances

8.1 As node sizes continue to decrease, new requirements for CMP PSD are becoming increasingly important.

8.2 As CMP slurries move towards smaller particle sizes more nano-size sensitive PSD metrologies are needed.

8.3 More PSD information is needed at smaller particle sizes (i.e., more particle size bins in the nanometer size range).

8.4 Metrologies capable of differentiating the nature of the measured particles are needed.

8.5 CMP PSD metrologies capable of measuring non-diluted slurries to replicate slurry conditions at Point of Use (POU) should be developed.

9 CMP PSD CoA Data Reporting

9.1 The slurry sample used for CoA data reporting should be representative of the final CMP Slurry product received at the end-user site.

9.2 The MPS and width of the peak should be reported.

9.3 The number of peaks in the PSD should be reported. If bimodal or trimodal the MPS and width for each peak should be reported.

9.4 The differential sizes at D5 and D10 should be reported for each peak in the distribution (see the Related Information § R1-2 for the meaning of D5 and D10).

9.5 If CMP slurry dilution was required to measure the PSD, the degree of dilution should be reported.

9.6 The metrology used to measure the PSD should be reported.

10 Related Documents

10.1 Kamiti, K., Popadowski, S., and Remsen, E. "Advances in the Characterization of Particle Size Distributions of Abrasive Particles used in CMP." Materials Research Society Symposium Proceedings, Vol. 991, 2007.

10.2 Babu, S. (Ed) "Advances in Chemical Mechanical Planarization (CMP)." Woodhead Publishing Series in Electronic and Optical Materials: Number 86, 2016. Chapter 12, pp. 299–325, "Chemical metrology methods for CMP quality." Pate, K and Safier, P.

10.3 Provder, T. "Challenges in particle size distribution measurement past, present and for the 21st century." Progress in Organic Coatings, 32 (1997): pp. 143–153.

10.4 Allen T. "Particle Size Analysis," 4th ed. Chapman and Hall, London, 1992.

RELATED INFORMATION 1

BACKGROUND INFORMATION

NOTICE: This Related Information is not an official part of SEMI C98 and was derived from the work of the global Liquid Chemicals Technical Committee. This Related Information was approved for publication by full letter ballot procedures on August 19, 2019.

R1-1 Certificate of Analysis Contents

R1-1.1 Ideally on a Certificate of Analysis (CoA) for CMP slurries this task force would like to see the following parameters reported:

- pH
- % solids content
- Density (SEMI C96-0618, Test Method for Determining Density of Chemical Mechanical Polish (CMP) Slurries)
- Viscosity
- Mean particle size
- Manufacturer and Model number of the analytical device used to generate data for particle size distribution inference.
- Manufacturer's material identification and traceability data

R1-1.2 Other parameters may be reported, depending on the specific vendor, vendor-customer agreement/contract include, but are not limited to the following:

- Conductivity (SEMI Guide in process)
- Trace metals
- Particle size distribution (see main body of this Document and Related Information § R1-2)
- Modality of particle size distribution
- Zeta potential

R1-2 Fundamentals of Reporting PSD Data for CMP Slurries

R1-2.1 ISO 9276-2:2014 provides relevant generic equations and coherent nomenclatures for the calculation of moments, mean particle sizes and standard deviations from a given particle size distribution. The equivalent diameter of a particle of any shape is taken as the size of that particle. Particle shape factors are not considered. The calculation of mean particle sizes and standard deviation from histogram data are presented in this ISO standard. When the PSD of a CMP slurry is measured the following information may be useful in addition to ISO 9276-2:2014.

R1-2.2 There are two types of PSD graphical representation: differential and cumulative. The differential distribution is obtained by differentiating the cumulative distribution curve and vice versa. The figures below show both differential and cumulative distributions for the same theoretical CMP slurry. Any number of statistical parameters can be calculated from this data. In this example a unimodal distribution (only one CMP material) is shown. The distribution is not 'normal' as it is skewed towards larger sizes. Mode, mean and median diameters can all be calculated as well as the cumulative diameters at D10 (the diameter of 10% of the total distribution), D25, D50, D75 and D90. The full width half max (FWHM) value is a measure of the width of the distribution. For CMP slurries there are different ways of measuring the width of the distribution including the geometric standard deviation, polydispersity, span, etc. This Guide does not comment on advantages or disadvantages of a specific methodology to measure width of a PSD distribution.