INLINE LIQUID REFRACTIVE INDEX MEASUREMENTS UNIFY ENGINEERS ACROSS THE SLURRY SUPPLY CHAIN BY PROVIDING A STANDARD LANGUAGE FOR REPORTING SLURRY DENSITY AND H2O2 CONCENTRATION

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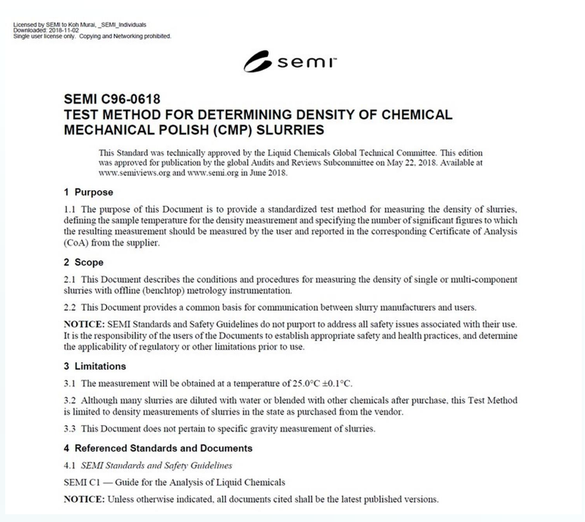
The chemical mechanical planarization CMP community has found a need for defining standards when providing technical reports or CoA of CMP related materials and consumables with respect to their properties in typical process conditions. High volume production of CMP slurries as formulated by slurry manufacturers as well as fast paced slurry blending with potential H2O2 addition and delivery to point of use, benefit from inline refractive index unit RIU measurement technology. The versatility of inline RIU technology lies in its ability to **define incoming raw slurry density, achieve slurry-water dilution ratios as well as ensure H2O2 concentration** levels in the mix of **copper, tungsten and interlayer dielectric ILD oxide slurries.**

As part of this study, an ILD oxide slurry was diluted with ultrapure water UPW in ratios from 1:0 to 1:10 resulting in a refractive index unit RIU to density g/cm3 correlation with a coefficient of determination R^2 exceeding 0.99. The RI sensors in full production were **installed in a CMP slurry loop** with a feed tank as well as a collection tank returning from the polisher. The inline RI sensor signal was used in slurry dilution control functions to achieve and ensure slurry-to-water ratio repeatability, providing means to rule out slurry composition excursions in the planarization process.

Introduction

The CMP community has called for standards when reporting CMP slurry density g/cm^3 by offline methods as delivered by the vendor.

This paper presents **inline** methods for the same CMP slurry density g/cm^3 monitoring. Tool onboard metrology suitable for slurry manufacturing QC in slurry product filling lines, as well as incoming slurry characterization in slurry delivery systems SDS become highly valuable.



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SEMI C96-0618
TEST METHOD FOR DETERMINING DENSITY OF CHEMICAL MECHANICAL POLISH (CMP) SLURRIES

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 May 22, 2018. Available at www.semiviews.org and www.semi.org in June 2018.

1 Purpose

1.1 The purpose of this Document is to provide a standardized test method for measuring the density of slurries, defining the sample temperature for the density measurement and specifying the number of significant figures to which the resulting measurement should be measured by the user and reported in the corresponding Certificate of Analysis (CoA) from the supplier.

2 Scope

2.1 This Document describes the conditions and procedures for measuring the density of single or multi-component slurries with offline (benchtop) metrology instrumentation.

2.2 This Document provides a common basis for communication between slurry manufacturers and users.

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 The measurement will be obtained at a temperature of 25.0°C ±0.1°C.

3.2 Although many slurries are diluted with water or blended with other chemicals after purchase, this Test Method is limited to density measurements of slurries in the state as purchased from the vendor.

3.3 This Document does not pertain to specific gravity measurement of slurries.

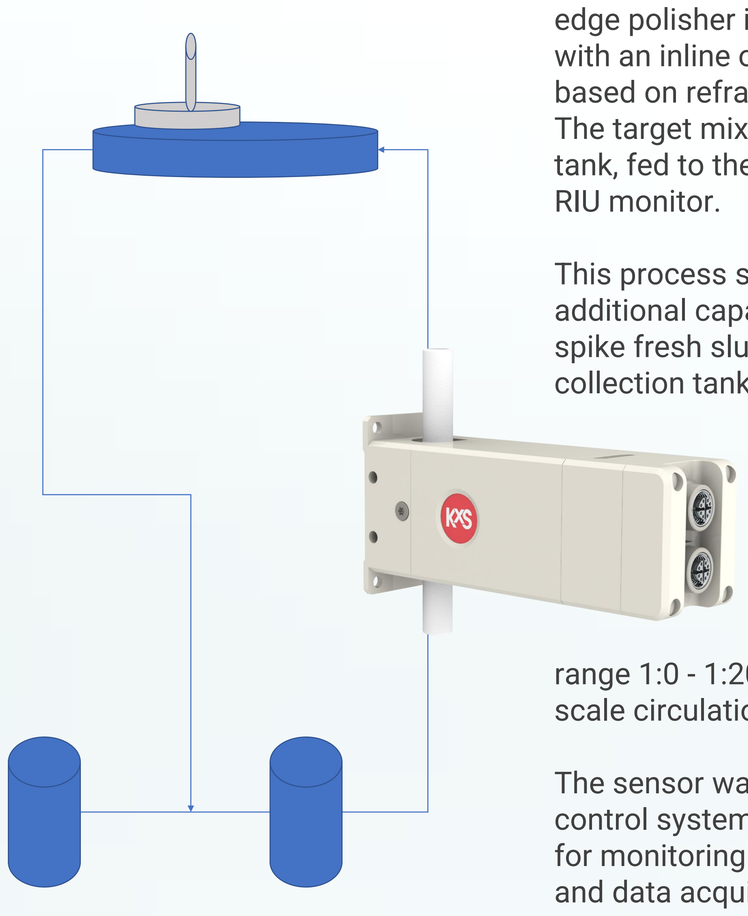
4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

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

Methods



An ILD oxide slurry-to-DIW ratio in a wafer edge polisher is targeted to be controlled with an inline optical concentration monitor based on refractive index RI technology. The target mix is prepared in a fresh slurry tank, fed to the polisher through the inline RIU monitor.

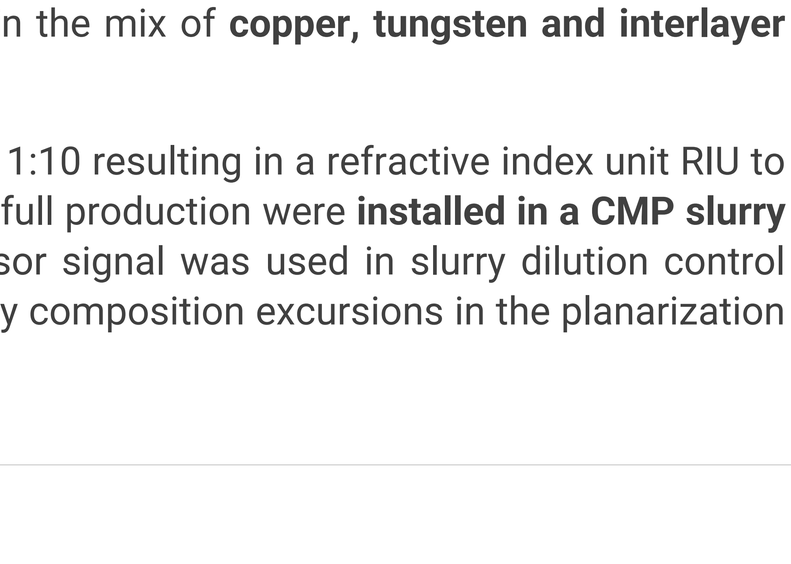
This process setup was designed with the additional capability to reclaim, filter and spike fresh slurry for reuse through a collection tank.

In order to correlate the RIU with slurry density and dilution ratio, a densitometer accurate to 10^{-4} g/cm^3 was used. The slurry:UPW samples in range 1:0 - 1:20 were prepared in a lab scale circulation test bench.

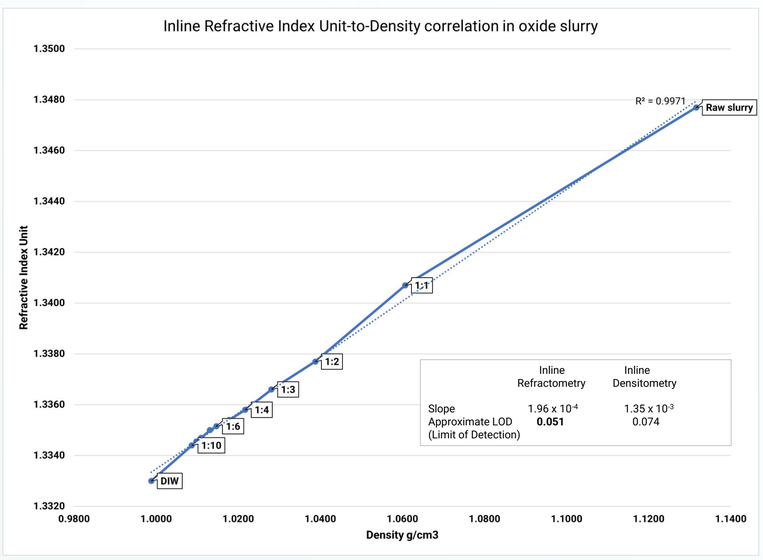
The sensor was connected to both the control system and sensor user interface for monitoring of measurement diagnostics and data acquisition.

Results

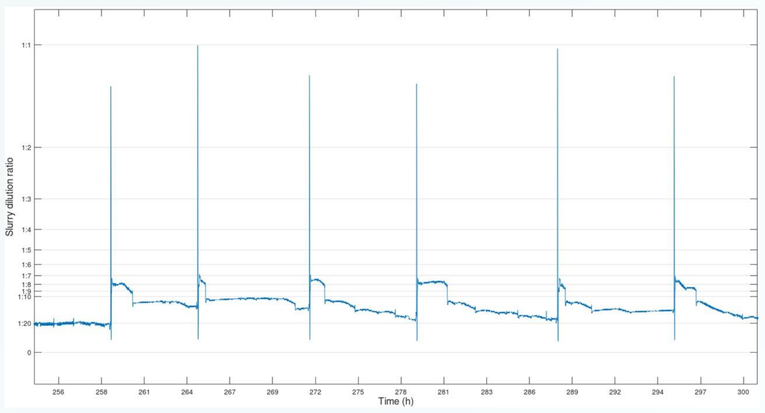
Inline refractive index unit measurements are proven in ensuring H2O2 conc% levels in tungsten and copper slurries. The same was here presented to be true **for density g/cm^3 measurements of tungsten, copper and ILD oxide slurries.**



It is known that slurry batches as delivered by the vendor present a density range. As shown in the upper chart, respective slurry type has its typical density level. The middle chart again shows that for the ILD oxide slurry, **inline RIU detects UPW %m/m** additions with an approximate **Limit of Detection LOD = 0.051**, while an **inline densitometer** with an approximate **LOD = 0.074** (lower is better).



In the wafer edge polishing process, the measurement signal confirmed the slurry:UPW ratio going to the polisher repeatable. Further work will focus on defining a process control function to achieve and ensure a stable slurry ratio at target level.



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