Application of HORIBA Instrument in Chemical Mechanical Planarization (CMP) : A Focus on Slurry Analysis and Optimization Strategies

化学機械研磨（CMP）プロセスにおけるHORIBAのアプリケーション：スラリー分析と最適化戦略を中心に

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Optimizing CMP for next-gen devices requires a holistic approach. This involves analyzing physicochemical properties, assessing removal rates, selectivity, planarity, and other relevant factors. In the dynamic landscape of semiconductor technology, analytical tools play a vital role in both assessment and optimization, underscoring their crucial importance in this intricate process. This article provides a summary of measurements conducted on the CMP process in HORIBA's application lab utilizing in-house scientific instruments. It delves into the crucial relationship between CMP slurries and particle size distribution in semiconductor manufacturing. And HORIBA's advanced instruments, such as the CN-300 Partica CENTRIFUGE, provide high-resolution capabilities and possess the ability to measure a diverse range of slurry samples. The document also underscores the role of zeta potential, controlled through sample formulations, and introduces the SZ-100V2 for simultaneous measurement of zeta potential and pH value, aiding in slurry design and quality control. Additionally, spectroscopic ellipsometry are presented as effective tools for determining removal rates and surface change. These contribute to the achievement of precise and robust CMP processes.

次世代デバイス向けのCMPプロセスの最適化には物理化学的特性，研磨レート，選択比，平坦性およびそれらに関連する要素の分析・評価など，広く総合的なアプローチが求められる。半導体技術が急激に進歩するにつれて，複雑なプロセスにおける分析の重要性が高まり，評価や最適化の両面において大きな役割を果たしている。本稿ではHORIBAの分析装置を用いたCMPプロセスに関する分析の概要を示す。まず，CMPスラリーと粒度分布の関係について述べ，多様なスラリーの粒度分布を高分解能で測定できるCN-300 Partica CENTRIFUGEを用いて測定した結果を示す。次に，スラリーの組成によって制御されるゼータ電位測定の重要性およびスラリー設計と品質管理に有用なゼータ電位とpHを同時測定できるSZ-100V2を用いた測定結果を示す。最後に研磨レートや表面状態変化を評価する手法として分光エリプソメトリーについて紹介する。これらの技術は精密でロバストなCMPプロセスの実現に貢献するものと考えている。

Introduction

Chemical Mechanical Planarization (CMP) originated in the 1980s at IBM[1] and has since become a key role in the integrated circuit (IC) manufacturing. CMP is applied in three main areas of IC manufacturing: forming transistors (FEOL), establishing local electrical connections between transistors (MEOL), and creating interconnect structures (BEOL)[1][2]. The FEOL process involves building the device architecture with various CMP steps for layers like SiO_2, Si_3N_4, poly Si stop layers, SiC, SiCN, high-k/metal gate structures, etc. W and Co have attracted attention for local interconnects in the MEOL process. In BEOL, multiple devices are interconnected by sequentially constructing multilevel Cu wires and insulating layers[1][2].

CMP operates through a synergistic interplay of chemical and mechanical interactions to achieve desired removal rates, selectivity, and planarity across different substrate materials in FEOL, MEOL, and BEOL. During the process, the substrate is pressed against a polymeric pad, while a slurry is transported on the pad and undergo three-body (slurry/polishing pad/wafer) interactions (Figure 1). Several factors, including CMP consumables such as slurry, pad, conditioner, and various process conditions like rotating speed and downforce, can influence the polishing performance[2].

To achieve optimized CMP performance tailored for next-generation devices, a comprehensive approach is necessary. This includes understanding the physicochemical properties of CMP slurries, evaluating removal rates, and selectivity, as well as planarity. Given the continuous evolution of semiconductor technology and the heightened demands on CMP performance, analytical tools play a dual role by both assessing and optimizing CMP performance. Therefore, it is evident that having appropriate analytical tools is crucial in this intricate process.

The impact of particle size on CMP performance

CMP slurries are composed of various components, and the formulation is adjusted according to the specific materials being polished. In general, the slurry comprises abrasives, oxidizers, organic compounds such as dispersants and passivation agents, and deionized water. The choice and concentration of these components influence the physicochemical properties of the slurry, impacting the overall CMP performance[2][3].

Particle size is a critical factor in the CMP process, influencing factors like abrasion efficiency and material removal rates. While past literature[3]-[5] extensively delves into the effects of particle size on CMP processes, comprehending its impact on CMP performance poses challenges, given the diverse influences of particle manufacturing processes, raw materials, and morphology[3].

Numerous research continues to seek a clearer mechanism describing the influence of particle size on CMP. As D. Tamboli et al. mentioned, when removal rates per abrasive particle are plotted against particle size, a clearer trend emerges, indicating an increase in removal efficiency per particle with size[3]. The contact area and size of particles play a crucial role in CMP processes, with larger particles exhibiting higher removal rates when the slurry's particle count is fixed[3][4]. However, spiking coarser particles in the slurry can lead to separation between the wafer and the pad, reducing pressure on smaller abrasive particles and resulting in decreased interaction with the substrate[5]. Observations from literature[5] also indicate that contamination by larger particles not only affect removal rate but also cause surface defect, such as scratching.

Advantaging of utilizing HORIBA Partica CENTRIFUGE CN-300 for CMP slurry analysis

Achieving optimal particle size distribution is crucial for ensuring uniform polishing and maintaining desired planarity in semiconductor manufacturing. Additionally, controlling particle size contributes to the prevention of scratching or damage to the polished surfaces, further emphasizing its pivotal role in the CMP process. Therefore, understanding and managing particle size within CMP slurries become essential for achieving precision and reliability in semiconductor fabrication. HORIBA offers multiple instruments for analyzing the wide range of particle size of slurry (Figure 2), employing various analytical techniques such as laser diffraction (Mie Theory), dynamic light scattering, and nanoparticle tracking analysis (NTA)[6]. And in response to the increasing demand for high-resolution CMP Slurry analysis in semiconductor factories, HORIBA has

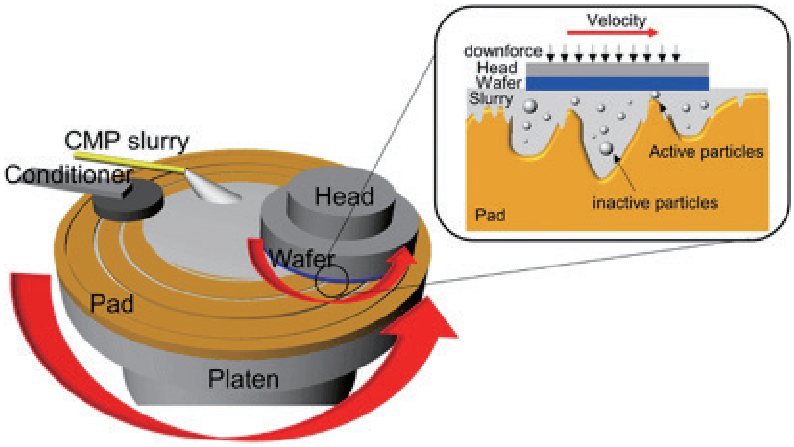

Figure 1 Three-body interactions in CMP process[2].

introduced the innovative Partica CENTRIFUGE CN-300. The CN-300 utilizes centrifugal sedimentation technology, with its primary advantage being high resolution. Furthermore, the CN-300 centrifuge presents several additional advantages, with a key focus on its two distinctive modes, the line-start and homogeneous mode, that significantly broaden the range of sample types and concentrations that can be accurately measured.

The Line-start mode enables the classification of particles based on their size, delivering precise and high-resolution results across a diverse range of samples in a single analysis. To evaluate the resolution of the Line-start mode method, a composite sample containing fourteen different sizes of monodispersed polystyrene latex (PSL), ranging from 100 nm to 30 µm[7], was utilized. As illustrated in Figure 3, a Particle Size Distribution (PSD) featuring fourteen distinct peaks, classified by centrifugal force, was observed. Significantly, these peaks closely aligned with the nominal values of the particle size standards in terms of size positions. The assessment of the mixed PSL sample has confirmed that CN-300 centrifugal sedimentation method demonstrates exceptional resolution and high accuracy capabilities.

The Homogeneous mode significantly broadens the CN-300's capabilities by facilitating the measurement of particle concentrations across a wider range. As shown in Figure 4, the Homogeneous mode enables the measurement of particle size across a broad concentration range, spanning from the undiluted sample to a dilution of up to 200 times. This implies that the CN-300 Homogeneous mode is applicable for measuring not even Point-of-Use (POU) slurry but also abrasive raw materials. Additionally, this mode proves highly effective for samples that react with Gradient Solution, highlighting the instrument's versatility in handling a diverse array of challenging samples. Such capabilities confer significant advantages in addressing the

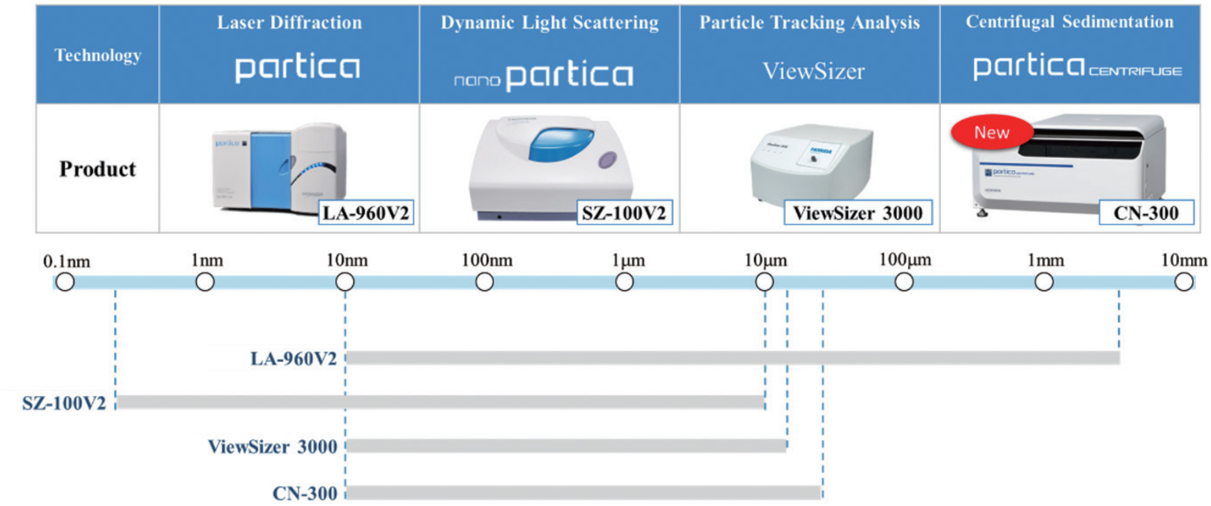

Figure 2 HORIBA particle size characterization techniques.

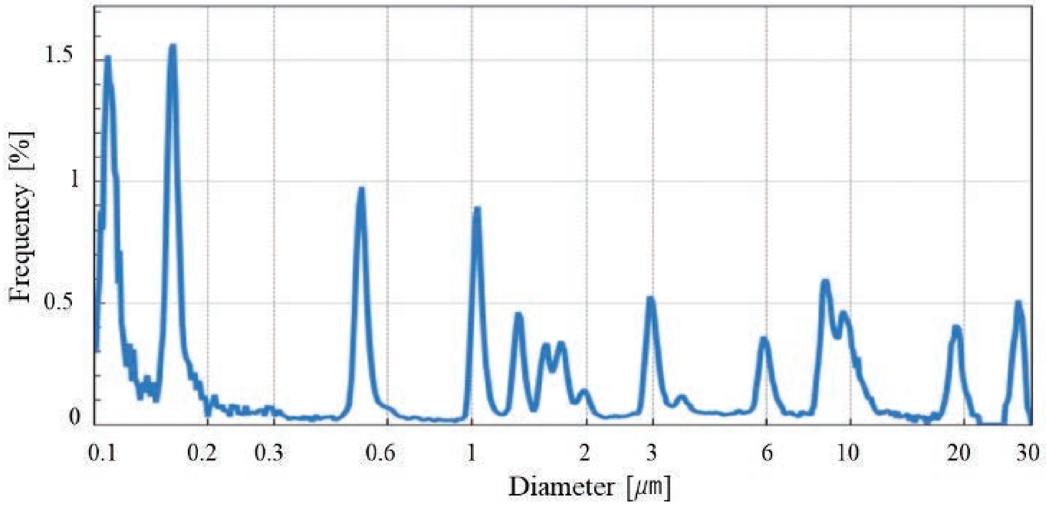

Figure 3 Measurement of mixed samples with different particle sizes with high resolution[7].

progressively complex CMP slurry formulations observed in recent years.

The influence of zeta potential on CMP slurry formulations

The impact of zeta potential on CMP is a crucial area of research, particularly concerning the interactions between particles and the substrate. The zeta Potential of particles can be further controlled through adjustments in sample formulations, allowing for precise regulation of the removal rate.

According to literature, key factors to increase friction energy in CMP processes include the number of active particles, the contact area of each particle, and the friction coefficient related to particle shape, surface morphology, and material type[4]. Increasing the number of active particles can be achieved not only by elevating particle concentration but also by facilitating particle adhesion onto the substrate surface. The constant adsorption and desorption of particles onto the substrate surface in CMP slurries distribution to the platen underscore the importance of inducing attractive forces between particles and the substrate by controlling the zeta potential of each material. The force acting between particles and the substrate in a liquid can be calculated based on the sum of electrostatic force due to electrical double layers and Van der Waals force, as per the DLVO theory. Furthermore, zeta potential adjustment can be achieved through various means, such as pH modification and the introduction of additives[8][9].

The monitoring of zeta potential is critical in both the design of CMP slurry formulations and the quality control of CMP slurry, as it plays a vital role in influencing various aspects of CMP processes. These include the efficiency of the formulation in enhancing removal rates, selectivity, and wafer planarity. HORIBA addresses this need by offering a solution that allows simultaneous measurement of the zeta potential and pH value of abrasives[10][11]. This instrument SZ-100V2 provides users with the capability to understand the zeta potential of abrasives under different pH conditions or the synergistic effects between abrasives and additives. Such insights are particularly valuable for scientists involved in designing slurry formulations and for quality control purposes, ensuring the robustness of the manufacturing process.

Additionally, SZ-100V2 offers the reliable measurement of ultra-diluted samples, enabling the acquisition of accurate zeta potential data. This capability not only ensures precision in measurements but also supports a Post CMP cleaner design approach by facilitating the exploration of interactions between abrasives and cleaners. For example, simulating the initial stages of the Post CMP process involves immersing ultra-diluted abrasives into cleaners. As shown in Figure 5, the zeta potential can be accurately measured even when the sample concentration is as low as 0.05wt%. This capability allows for the measurement of ultra-diluted abrasives in cleaners. Research efforts can then ascertain whether there is a repulsive or attractive force between the slurry abrasive and the wafer. The data obtained provides valuable insights into the formulation design direction, facilitating the pursuit of more challenging targets.

The Role of HORIBA UVISEL Plus Phase Modulated Ellipsometry in Thin Film Characterization

Spectroscopic ellipsometry is a non-destructive, non-contact, and non-invasive optical technique which is based on the change in the polarization state of light as it is reflected obliquely from a thin film sample. Ellipsometry uses a model-based approach to determine thin film, interface, and surface roughness thicknesses, as well as optical properties for thin films ranging in thickness from a few Å to several tens of microns. Therefore, it proves to

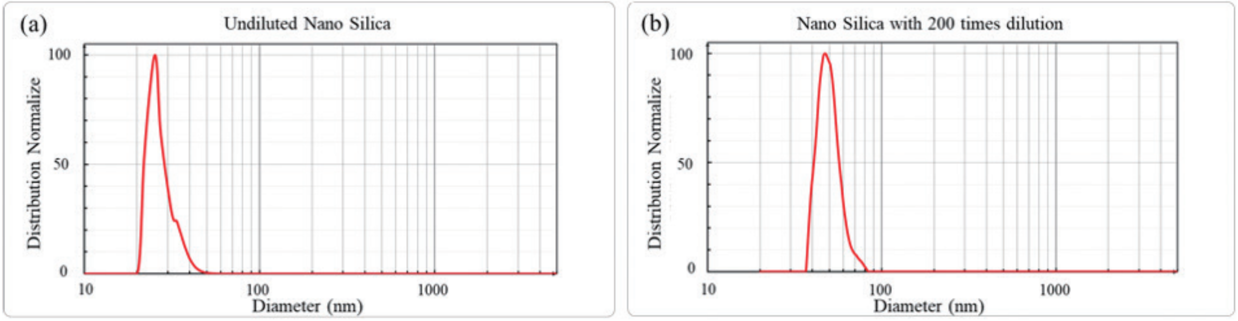

Figure 4 Using CN-300 Homogeneous mode to determine the particle size for both (a) the undiluted sample and (b) the sample diluted 200 times.

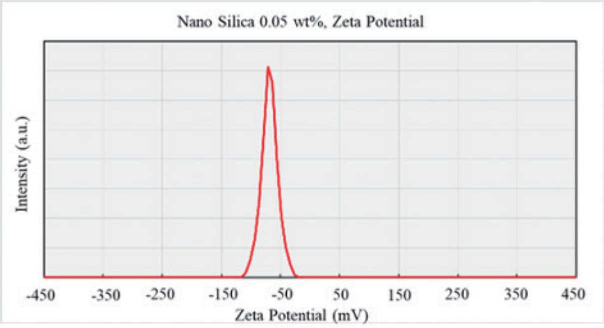

Figure 5 The zeta potential can be measured even when the sample concentration is as low as 0.05wt%.

be a valuable technique in the field of CMP as it facilitates the determination of removal rates for specific wafers. To gauge these removal rates accurately, it becomes imperative to measure the thickness of wafers both before and after CMP processes. Employing multiple thickness measurements enable verification of within-wafer uniformity.

And the HORIBA UVISEL Plus with phase-modulated ellipsometer achieves higher sensitivity and accuracy when compared to conventional ellipsometers using rotating elements. It enables the detection of single-angstrom variations or interfaces not observable by other ellipsometers. By incorporating a thin film model, users gain the capability to discern thickness as well as optical constant changes at specific sites. This functionality enables formulation determination and optimization of process parameters, allowing for the detection of even slight differences in surface conditions. For instance, in cases where certain components in the slurry exhibit strong absorption behaviors on a particular type of wafer, UVISEL Plus ellipsometry reveals spectral differences between the wafer before and after treatment. By developing a fitting model, researchers can delve into the study of absorption behaviors[12]. This capability offers users a comprehensive understanding of the intricacies of the surface by optical constant change, facilitating informed decision-making in the context of CMP processes.

Conclusion

In summary, this document highlights diverse HORIBA analytical tools applicable to the CMP process. The Partica CENTRIFUGE CN-300, with its Line-start and Homogeneous modes, offers high-resolution slurry analysis, addressing the complexity of modern CMP slurry formulations. The importance of maintaining particle size within specified ranges is underscored. The impact of zeta potential on CMP is discussed, and the SZ-100V2 is introduced as a valuable solution for simultaneous measurement of zeta potential and pH in abrasives. This instrument's capability to handle ultra-diluted samples supports precision measurements and aids in the design of Post-CMP cleaner approaches. Additionally, the document explores the role of spectroscopic ellipsometry in evaluating removal rates, providing a comprehensive understanding of CMP processes. As semiconductor technology progresses, these advanced analytical tools from HORIBA contribute significantly to achieving precision, reliability, and efficiency in CMP applications.

* Editorial note: This content is based on HORIBA's investigation at the year of issue unless otherwise stated.

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