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Correspondence to:
Hyeong-U Kim
guddn418@kimm.re.kr;
Taesung Kim
tkim@skku.edu

# These authors equally contributed to this work.

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Development of a single particle sizing system for monitoring abrasive particles in chemical mechanical polishing process

Changmin Kim[1,2,#], Sungwon Choi[3,#], Jeongan Choi[1,#], Hyunho Seok[4], Keun-Oh Park[3], Youngho Cho[3], Kihong Park[2], Sanghyuck Jeon[2], Hyeong-U Kim[1] and Taesung Kim[2]

[1]Department of Plasma Engineering, Korea Institute of Machinery and Materials (KIMM), Daejeon 34103, Korea, [2]School of Mechanical Engineering, Sungkyunkwan University (SKKU), Suwon, Gyeonggi-do 16419, Korea, [3]Korea Spectral Products Co., Ltd., 273 Digital-ro, Guro-Gu, Seoul, Korea, [4]SKKU Advanced Institute of Nanotechnology (SAINT), Sungkyunkwan University (SKKU), Suwon, Gyeonggi-do 16419, Korea

Abstract A particle sizer was developed for real-time monitoring of suspended particles in chemical mechanical polishing (CMP) slurries to prevent any damage caused by coarse particles in the CMP process. Laser-induced light scattering from individual particles was utilized to monitor the concentration and size distribution of suspended particles in the CMP slurries, because monitoring is crucial for achieving higher than 80 % of chip yields by controlling the slurries. The particle sizer, termed as PrimeSizer_SM Micro, consists of an integrated fluid system and optical sensing component. The PrimeSizer_SM Micro was calibrated using commercially available standard polystyrene-latex (PSL) particles with sizes in the range of 0.15-0.5 μm. Performance was evaluated in terms of the PSL particle size distribution. Two types of CMP slurries, containing silica and ceria particles, were analyzed using the PrimeSizer_SM Micro. The results revealed the size distributions and concentrations of the CMP slurries.

1. Introduction

Following the technological progress in semiconductor manufacturing, the design rule of line-widths dropped below 10 nm [1]. In particular, in the case of three-dimensional (3D) NAND flashes, 3D lamination in semiconductor manufacturing has advanced the possibility of integration by over hundred layers [2-4]. Thus, according to the integration rate of semiconductors, chemical mechanical polishing (CMP) has become very important [5-7]. Because layer collinearity is important for uniform multi-layer stacking, such large-area planarization can only be performed using CMP [8, 9].

However, many layer defects, such as scratches and pitches, are caused by the mechanical and chemical effects of the CMP abrasive particles. When the design rule of the semiconductor line-width was a few micrometers, the damage caused by nanometer-sized particles was not an issue. However, as the semiconductor line-width has recently decreased to a few nanometers, the risk of damage-induced serious defects and reduced product yield has increased [10, 11]. In particular, during the CMP process, particles with sizes of several tens of nanometers to several hundreds of nanometers are used as abrasive particles [12]. Owing to the aggregation of abrasive or large-sized particles, scratches or damage can occur on the wafer surface during the CMP process [13]. Because these defects may significantly affect the product yield, it is important to determine and analyze their major cause by proper process diagnosis. Although various instruments are used to measure the particles distributed in liquid samples, many limitations remain. Most particle-counting techniques have been developed for analyzing airborne and suspended particles in liquids. For example, an aerodynamic particle sizer (APS 3321; TSI Inc., USA) is often used for measuring airborne particles [14]. This device calculates the flight time of the particle based on the weight of the particle [15]. Therefore, airborne particles can be analyzed using aerodynamic particle diameters in the range of 0.5-20 μm. However, this tech-

nique is not appropriate for measuring abrasive particles inside CMP slurries, because the time-of-flight method is not applicable to suspended particles in a liquid [16, 17].

For this reason, dynamic light scattering (DLS) is generally used for analyzing CMP slurries [18]. DLS estimates the extent of the Brownian motion of particles in a liquid suspension, according to the size of the particle. DLS can be used to determine the size distribution and zeta potential of a particle-suspended liquid [19]. Particles with sizes in the range of 0.6-10000 nm can be measured. However, this measurement range is not applicable to aggregated particles with sizes exceeding 100 μm, as is typical for CMP slurries [20]. Moreover, particle sizing errors are likely to occur, owing to the sedimentation problem, and multiple light scattering is observed when the scattered light from one particle is scattered by another before reaching the detector. Therefore, DLS is typically used for measuring the average particle size and zeta potential of the abrasive particles rather than for particle counting in CMP.

Single-particle optical sizing (SPOS) is an optical sensing technique that can accurately detect suspended liquid particles even at low particle concentrations [21]. The SPOS technique allows single-particle detection by exploiting scattered light signals from particles inside a liquid suspension [22]. The advantage of the SPOS method is that it can measure the sizes and numbers of small particles in real time with a high resolution. In addition, an SPOS measurement system can be developed based on a small optical system consisting of optical lenses, an excitation laser, and an optical sensor.

In this study, we developed a simple optical system (PrimeSizer_SM Micro) for measuring particles in a liquid sample. Theoretical studies and experiments on the particle size and number concentration have been conducted to develop a particle-measurement algorithm. The developed equipment was calibrated using polystyrene-latex (PSL) standard particles, and the size measurement accuracy and counting accuracy were measured for 0.15, 0.2, 0.3, 0.4, and 0.5 μm channels. In addition, the particles that were used for calibration were measured using DLS, and the measurement accuracies of the two methods were compared. Furthermore, by measuring abrasive particles in actual CMP process, the possibility of monitoring large particles in a slurry was confirmed.

2. Theoretical background

The intensity of particle scattering depends on multiple factors, such as particle size, shape [23]. Two types of particle-based scattering, Mie scattering and Rayleigh scattering, are often used for explaining particle scattering patterns [24, 25]. The size parameter, x, is used for characterizing particle scattering. The size parameter is defined as follow Eq. (1):

$$x = \frac{2\pi r}{\lambda} \tag{1}$$

where r is the particle radius and λ is the wavelength of the incident light. Rayleigh scattering occurs for values of x smaller than 0.2. Mie scattering occurs for size parameters in the 0.2-2000 range. Geometric optics, a domain dominated by the ray-like characteristics of light can be employed for x > 2000 [26]. The theory of Mie scattering established by Gustav Mie is applicable to particles with diameters in the 0.05-100 μm range. The Mie scattering intensity [27] I_{scat} is given as follow Eq. (2):

$$I_{scat} = I_0 \frac{1}{d^2} \sigma'_{scat} \tag{2}$$

where I^0 is the intensity of the incident light; σ'_{scat} is the cross-section of the differential scattering; and d is the distance between the particle and photo detector [28]. In addition, the cross-section of differential scattering is as follow Eq. (3):

$$\sigma'_{scat} = \frac{\lambda^2}{8\pi^2}(i_1 + i_2) \tag{3}$$

where λ is the wavelength of the incident light, and i_1 and i_2 are scattering angular functions representing vertical and horizontal components, respectively. In addition, the angular function is the component necessary for calculation according to the direction of scattered light.

In addition, to measure particles passing through a specific sensing volume, it is necessary to know the scattering cross-sectional area, which represents the number of particles scattered at a specific angle when two particles are scattered as a result of repulsion owing to a distant interaction [29]. To determine the scattering cross-sectional area, it is necessary to calculate the extinction efficiency and scattering intensity. The extinction is the sum of absorption and scattering that represents total effect of medium on radiation passing the specific medium. Also, the extinction efficiency is a property that determines how strongly a species reflects or absorbs light or radiation at a particular wavelength. The scattering cross-sectional area can be obtained using the following formula as follow Eq. (4):

$$\sigma_{ext} = \left(\pi D^2 / 4\right) Q_{ext} \tag{4}$$

where D is the diameter of particle, and Q_{ext} is the extinction efficiency. The extinction efficiency in Fig. 1(a) captures how strongly a particle reflects or absorbs incident light at a certain wavelength. The scattering intensity represents the amount of radiation scattered from any particular angle, as shown in Fig. 1(b). The extinction efficiency and scattering intensity are proportional to the particle size for particles with diameters in the 0.1-0.5 μm range [30-32]. Thus, it is easy to measure large particles using an optical system with a simple optical design. Therefore, the particle size can be estimated based on the scattering intensity [33].

The scattering intensity was different depending on the size

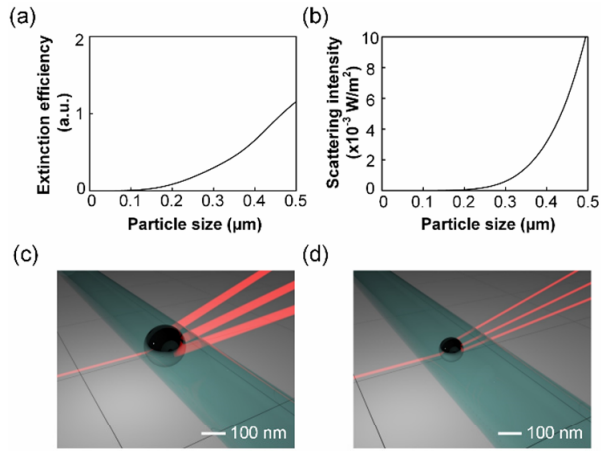

Fig. 1. (a) Extinction efficiency vs. particle size of polystyrene latex (PSL) particles; (b) scattering intensity in terms of PSL particle size. Difference of scattering intensity according to size of particle; (c) large particle scattering intensity; (d) small particle scattering intensity.

of the particles, the scattering intensity of large particles is much larger than small particles, as shown in Figs. 1(c) and (d). Based on the theory and actual experimental results, strong scattering intensity occurs in large-sized particles, and weak scattering intensity occurs in small-sized particles.

3. Experimental setup

Deionized water (DIW) was prepared using a water purification system and fed into a vessel through a water purification filter for sample preparation. PSL particles (Duke Scientific, Inc., USA) were used for calibrating the PrimeSizer_SM Micro device. The diameter of the PSL particles ranged from 150 nm to 500 nm, and the concentration of the samples was 0.1 wt.%. In addition, two CMP slurries (pH, 9; 30 wt.% of silica and ceria particles) were prepared for evaluating the performance of the PrimeSizer_SM Micro device. The abrasive particles in the CMP slurries had diameters in the 100-120 nm range.

A DLS particle analyzer (ELSZ-2000, Otsuka Electronics Co., Ltd., Japan) was used for measuring the particle size distribution. The DLS particle analyzer utilized the Brownian motion of suspended particles to measure their average size and size distribution. The PSL and commercial CMP slurry particles were measured using a DLS particle analyzer. The actual size distributions of the samples were compared with the measurement results of the PrimeSizer_SM Micro device.

The PrimeSizer_SM Micro device consists of an air valve, drain and filter parts, two pumps, a vessel, and a sensor, as shown in Fig. 2. The detailed specifications of the PrimeSizer_SM Micro device are shown in Table 1. The sample feed rate was 5 mL/min, using two peristaltic pumps. The vessel consists of DIW, a drain, an air nozzle, and a sample inlet, as shown in Fig. 2(a). In addition, the sensor was connected to the lower part of the vessel. The sensor consists of a laser diode, photodiode, and flow cell, as shown in Fig. 2(b). The

Table 1. Specification of the PrimeSizer_SM Micro.

Optical system	Single optical sizing system (SPOS)
Light source	Diode pumped solid state laser (wavelength, 532 nm; power, 200 mW; CNI laser)
Flow cell material	Quartz (fused silica)
Flow rate	5 mL/min
Measurement time	2 min
Measurement size range	0.5 > x > 0.15 μm

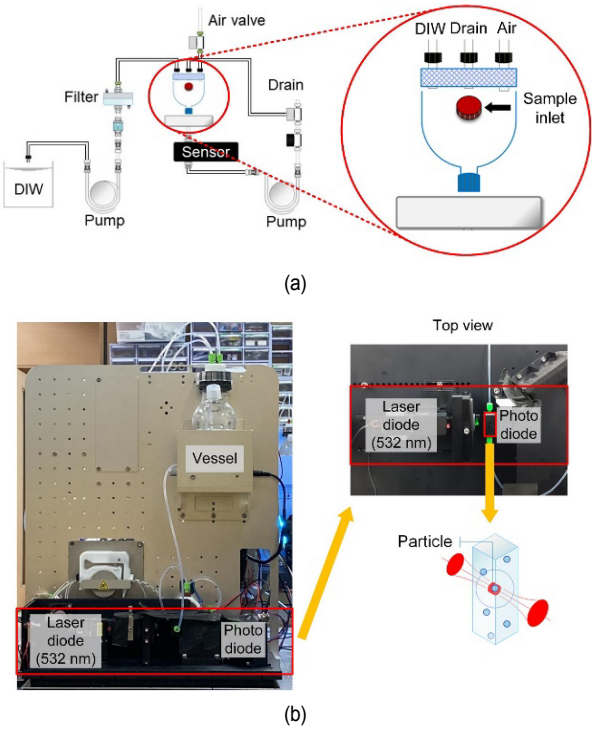

Fig. 2. (a) Schematic of the PrimeSizer_SM Micro that consist of two pumps, a water filter, a vessel, and a particle sensing unit; (b) front image of the particle sizer integrated with the single particle optical sizing system equipped with a 532 nm laser diode and a photo diode and top view image of the particle sensing part.

wavelength and power of the laser diode were 532 nm and 200 mW, respectively. A photodiode was employed to detect the intensity of the light scattered from the particles. The sampling rate and digital output of the photodiode were 1 MHz and 16 bits, respectively. The quartz flow cell was installed for measuring the particles, and it measured the particles passing through the flow cell. The material of the flow cell was fused silica. Fig. 2(b) shows an image of the prototype of the PrimeSizer_SM Micro. The flow rate of the entire system was 5 mL/min, and the measurement time was 2 min.

4. Results and discussion

4.1 DLS measurement

DLS analysis was performed to characterize the PSL parti-

Table 2. Average particle size and measurement error for different PSL particle sizes measured by DLS.

PSL (μm)	DLS-1 (μm)	DLS-2 (μm)	DLS-3 (μm)	Average size (μm)	Sizing error
0.15	0.17	0.17	0.18	0.17	14.80 %
0.20	0.23	0.22	0.22	0.22	12.05 %
0.30	0.33	0.34	0.34	0.33	10.37 %
0.40	0.43	0.44	0.43	0.43	7.75 %
0.50	0.49	0.49	0.49	0.49	1.00 %

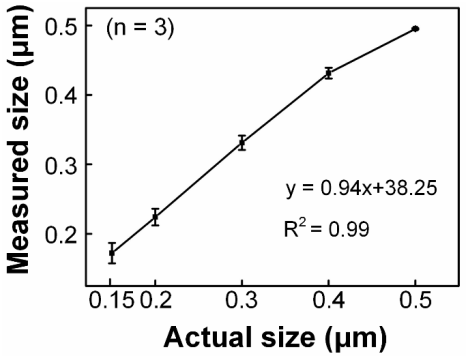

Fig. 3. DLS-measured vs. actual PSL particle size.

cles used for calibrating the PrimeSizer_SM Micro device. DLS can yield the average particle size of abrasive particles and zeta potential of the CMP slurry, which play important roles in the CMP process. Despite these advantages, DLS cannot accurately measure the size and number of individual particles. The standard particles used in the experiment were particles of Duke Scientific and had a size distribution recognized as an international standard, the actual size was identified using an official test report.

The actual and DLS-measured particle sizes are compared as shown in Fig. 3 and listed in Table 2. Five different PSL particle sizes (0.15, 0.2, 0.3, 0.4, and 0.5 μm) were measured by the DLS analyzer. To measure the error between the particle size measured by DLS and the actual PSL particle size, each PSL sample was measured three times to determine the average size and measurement error. The average sizes of the PSL particles were 0.172, 0.224, 0.331, 0.431, and 0.495 μm, and the discrepancies between the actual and measured sizes were 14.80 %, 12.05 %, 10.37 %, 7.75 %, and 1 % for each PSL sample.

Although the average size of the particles in a sample can be measured using the DLS method, large particles that are likely to be associated with defects during CMP may not be adequately detected, because it is impossible to measure the number of particles included in a sample using DLS. Therefore, detection of abnormal-sized particles in CMP should be done using a single-particle detection system.

4.2 Calibration of the prime-sizer_SM micro

The optical particle-sensing component of the PrimeSizer_SM Micro device is a laser-beam-focused optical system. The focused-type optical sensor is suitable for measuring small-sized particles because it can yield higher power density compared with that of the line beam-type optical sensor. Owing to this important advantage, a focused-type optical sensor was adopted for the PrimeSizer_SM Micro device.

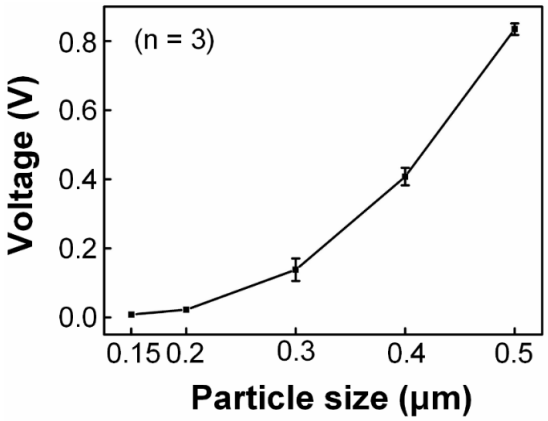

Fig. 4. Calibration of the PrimeSizer_SM Micro using PSL particles.

The PrimeSizer_SM Micro device was calibrated using PSL particles. The intensity of light scattered from PSL particles was converted to voltage using a photodiode, as shown in Fig. 4. The voltage signal was used as a critical point for determining the individual particle sizes. The voltage signal was used as a critical point for determining the individual particle sizes. The critical point values for the PSL particles with diameters of 0.15, 0.2, 0.3, 0.4, and 0.5 μm were 0.008, 0.022, 0.137, 0.407, and 0.834, respectively. The PrimeSizer_SM Micro device has five channels (0.15, 0.2, 0.3, 0.4, and 0.5 μm) for sizing resolution determined by the critical point values as listed in Table 1. Therefore, voltage outputs above each critical value can be considered to correspond to the same particle size in the PrimeSizer_SM Micro.

4.3 Size distribution of PSL particles

The performance of the PrimeSizer_SM Micro device was evaluated using monodisperse PSL particles of five different sizes. Particles with diameters of 0.15, 0.2, 0.3, 0.4, and 0.5 μm were measured using each of the five channels of the PrimeSizer_SM Micro device, and the results are shown in Fig. 5. The numbers of the 0.15 and 0.2 μm-diameter particles were 3746 and 16107 /mL, respectively, as shown in Figs. 5(a) and (b). The results in these figures exhibited no significant differences with respect to the number of particles measured in the neighboring measurement channels. This was because the intensity of the light scattered from small particles was very weak. However, it was possible to distinguish the particles by size based on the position of the peaks in the different channels. The numbers of particles with diameters of 0.3, 0.4, and 0.5 μm were 20970, 24286, and 34006 /mL, respectively, as shown in Figs. 5(c)-(e). For the channels

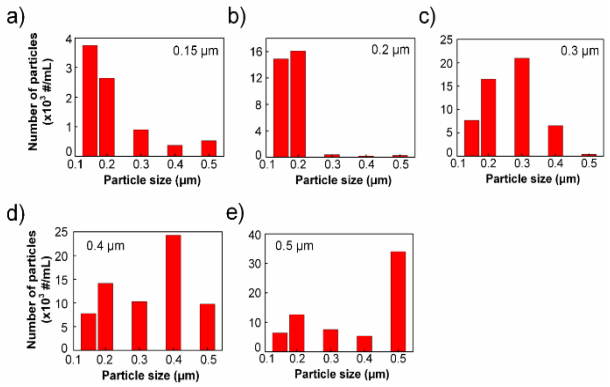

Fig. 5. Size distributions and number concentrations of the monodisperse PSL particles measured by the PrimeSizer_SM Micro.

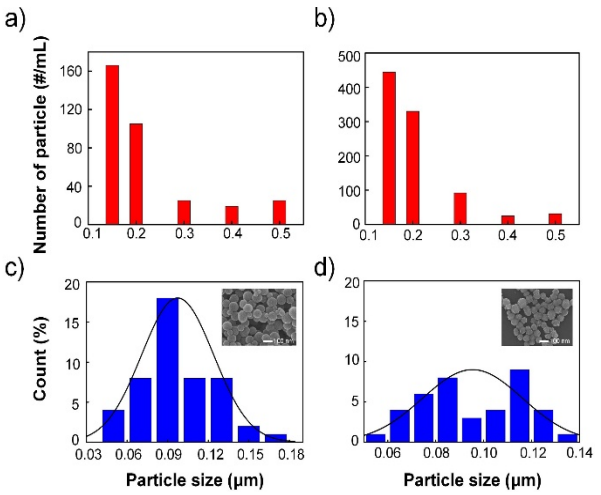

Fig. 6. Particle number concentrations of two types of CMP abrasive particles: (a) colloidal silica particles; (b) ceria particles, measured by the PrimeSizer_SM Micro. Number count distributions of the; (c) colloidal silica particles; (d) ceria particles measured from SEM images.

measuring 0.3, 0.4, and 0.5-μm-diameter particles, several particles were measured for each channel, and the particles could be unambiguously categorized in terms of their size. The size distributions measured using the PrimeSizer_SM Micro device were wider than those measured by the DSL analyzer. The discrepancy could be attributed to the measurement technique used by the optical-sensing component. Because the beam size of the light source used in the focused-type sensor has a non-uniform Gaussian distribution, the pulse height distribution, corresponding to the scattering-based size distribution of the particles, was wide. However, it was also possible to unambiguously categorize the larger particles, resulting in many defects, using the PrimeSizer_SM Micro. These results show that abnormal-sized abrasive particles with diameters exceeding 0.5 μm, can be easily monitored using the PrimeSizer_SM Micro device. Overall, the PrimeSizer_SM Micro device yielded correct results for the different measured PSL particles.

4.4 Measurement of abrasive particles

The abrasive particles that were added to the initial CMP slurry were usually well distributed within the slurry. In addition, the average sizes of colloidal silica and ceria particles in the initial state (before the treatment) were in the 0.08-0.1 μm range. However, after a certain time, the effect of the additives decreased, and the abrasive particles started to aggregate. Aggregation of particles with diameters larger than approximately 0.1 μm may cause scratches and/or defects on the wafer surface during the CMP process. Therefore, it is important to measure the size and number of particles responsible for defects.

The number concentrations of the two abrasive particle types were measured using the PrimeSizer_SM Micro device, and the results are shown in Fig. 6. The measured abrasive particle types are colloidal silica and ceria, as shown in the SEM images in Figs. 6(c) and (d), respectively. The number of colloidal silica particles measured were 161, 115, 26, 19, and 27 in the 0.15, 0.2, 0.3, 0.4, and 0.5 μm measurement channels, respectively, while the numbers of ceria particles measured were 451, 321, 98, 25, and 30 those same channels.

A large number of particles were measured in the 0.15 and 0.2 channels, because the particle sizes were approximately in the 0.15-0.2 range, even if only a small number of particles were aggregated. And, the particles measured in the 0.3 to 0.5 channels suggests that the number of aggregated particles was measured. This allows to determine the number of aggregated particles in a measured sample, and a correlation with defects occurring during CMP can also be estimated.

In addition, the number concentrations of the abrasive particles measured by the PrimeSizer_SM Micro were verified by directing particle counting using SEM images. Figs. 6(c) and (d) show the number counts of the two CMP abrasive particles processed by direct size measurement using the two inset images. In the case of colloidal silica, particles in the size range of 0.03 to 0.18 μm were measured, and in the case of ceria, particles in the size range of 0.06 to 0.14 μm were measured. The initial size distribution of both abrasive grains was about 0.08 to 0.1 μm. There were no particles larger than 0.2 μm in the SEM images. These results support the fact that there were agglomerated or aggregated abrasive particles in the CMP slurries as shown in Figs. 6(a) and (b). Therefore, the developed particle sizer can provide good performance for abrasive particle monitoring in CMP slurries.

5. Conclusion

In this paper, we developed a real-time particle counter (PrimeSizer_SM Micro) for analyzing the abrasive particles in CMP slurries. A low-cost and custom-built optical set-up was combined with a fluidic system, to collect the light scattered from particles in the analyzed slurries. The PrimeSizer_SM Micro device was carefully calibrated using standard PSL parti-

cles for the purpose of particle sizing. In addition, the PrimeSizer_SM Micro device was utilized for measuring the number of particles and size distributions of two commercial abrasive particle types in CMP slurries. The results suggested agglomeration and/or aggregation of particles in the studied CMP slurries. The developed particle counter can be used for overcoming the limitations of DLS, which is conventionally used for measuring the average sizes of particles during CMP. Therefore, the PrimeSizer_SM Micro device is likely to constitute a cost-effective diagnostic solution for mitigating particle-related damages in CMP.

Acknowledgments

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Nomenclature

x	: Size parameter
λ	: Wavelength
r	: Radius of particle
d	: Distance between the particle and photo detector
I	: Mie scattering intensity
I^0	: Intensity of incident light
σ'_{scat}	: Cross-section of differential scattering
σ_{ext}	: Scattering cross-sectional area
D	: Diameter of particle
Q_{ext}	: Extinction efficiency

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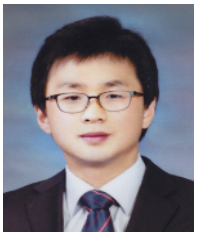
Changmin Kim received his Bachelor of Science degree in mechanical engineering from Kangwon National University, Korea, in 2015. He received his Master of Engineering degree in 2017 from the Department of Convergence Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea. He is currently Ph.D. candidate in the Department of Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea.



Sungwon Choi received his M.S. from Sogang Univesity in 2014. He is currently works as a resercher at Korea Spectral Products. His research interests are light scattering and optical analysis using machine learning.

Jeongan Choi is a Senior Researcher at the Korea Institute of Machinery and Materials (KIMM). He received the Ph.D. degree in mechanical engineering from the University of Illinois Urbana Champaign, Champaign, IL, USA, in 2021. His current research interests include plasma technologies for renewable energy devices and laser diagnostics for reacting flows.

Hyunho Seok is a Ph.D. candidate in SKKU Advanced Institute of Nanotechnology (SAINT) at Sungkyunkwan University. He received his B.S. degree in the Department of Mechanical Engineering from Sungkyunkwan University in 2019. His research interest is plasma-assisted 2D materials and 2D heterostructures synthesis and mechanism investigation for their electrical and electrochemical applications.

Keun-Oh Park received his master's degree at Pusan National University in 1993, worked at SK Hynix (device company), TES (equipment company), and now works as a researcher at Korea Spectral Products. His research interests focus on process diagnosis and LPC of semiconductor equipment.

Youngho Cho received his Ph.D. from Kookmin Univesity in 2019. He is currently works as a resercher at Korea Spectral Products. His research interests are spectroscopy, especially Raman and plasma spectroscopy.

Kihong Park received his Bachelor of Engineering degree from the Department of Chemical Engineering, Changwon National University, Republic of Korea, in 2015. He received his Master of Engineering degree in 2017 from the Department of Convergence Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea. He is currently Ph.D. candidate in the Department of Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea.

Sanghyuck Jeon received his Bachelor of Engineering degree from the Department Materials Science & Engineering, Seoul National University of Science and Technology, Republic of Korea, in 2015. He received his Master of Engineering degree in 2017 from the Department of Convergence Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea. He is currently Ph.D. candidate in the Department of Mechanical Engineering at Sungkyunkwan University (SKKU), Republic of Korea.

Hyeong-U Kim received his B.S. from Hanyang University in 2013 and Ph.D. degree in 2019 from Sungkyunkwan University. He is currently a Senior Researcher in the Department of Plasma Engineering from Korea Institute of Machinery & Materials (KIMM). His research field is plasma-based 2D semiconductor and heterostructure synthesis and etching for various applications

Taesung Kim received his B.S. from Seoul National University in 1994 and a Ph.D. degree in 2002 from the University of Minnesota. He is currently a Professor in the Department of Mechanical Engineering and SAINT at Sungkyunkwan University. His research interest is aerosol technology, semiconductor process (chemical mechanical planarization and cleaning), and plasma process for 2D material synthesis.