euspen's 22nd International Conference & Exhibition, Geneva, CH, May/June 2022
www.euspen.eu

Absolute longitudinal distance measurement verification of a standard polystyrene nanoparticle near a surface in water by means of multi-wavelength evanescent field

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Abstract
In the wet semiconductor process, abrasive nanoparticles are widely used in the chemical mechanical polishing (CMP) process to polish wafer surfaces for high planarization. During polishing, nanoparticles move with a high-speed three-dimensional (3D) position in the solution. In the 3D particle's moving positions, Z is a perpendicular longitudinal distance between the surface to the nanoparticle that plays a significant role in polishing, which could not be measured by general microscopy without longitudinal scanning. To understand nanoparticle phenomena near-surface, we have proposed and developed the apparatus with real-time fast-motion tracking on optical multi-wavelength evanescent fields microscopy to measure the 3D motion of the nanoparticle in water without scanning. In euspen's 2020, we could verify the relative distance ΔZ of nanoparticles in water, but the absolute distance Z is unknown yet. In this manuscript, we have therefore developed an invisible nano-step height in water to verify the absolute height Z of an individual nanoparticle. The invisible nano-step height in water is made from an optical resin with a refractive index of 1.35 that is close to a refractive index of water to avoid unnecessary light during experimental verification. The nano-step height was less than 100 nm on a glass surface, fabricated using nanoimprint lithography. Polystyrene ϕ100 nm standard particles in water were used to verify the height Z. The results of height Z of nanoparticles could be measured less than 100 nm with the measurement uncertainty ±10 nm (2σ), which is related to the interferometer result of the nano-step height.

Absolute longitudinal distance measurement, standard polystyrene nanoparticle, invisible nano-step height, multi-wavelength evanescent fields microscopy, wet condition

1. Introductions

An abrasive nanoparticle behaviour in the chemical mechanical polishing (CMP) process is a three-dimensional (3D) motion near a substrate or reference surface. The height Z is a longitudinal distance between the reference surface and the nanoparticle in z-direction that plays a significant role in polishing [1], which general microscopy could not measure without longitudinal scanning. Evanescent field microscopy has been proposed to detect the scattering light from nanoparticles near a surface with high contrast [2-8]. Our previous studies proposed multi-wavelength evanescent fields to measure the 3D motion [9-10] and the relative height of polystyrene 100 nm particle, but the absolute height was not confirmed yet [11]. In this manuscript, we proposed a design concept of the nano-step heights to verify the height Z of the nanoparticle [12]. The invisible nano-step height is made from an optical resin (NOA1348) with a refractive index (n ≈1.35) that is close to a refractive index of water to decrease the unnecessary light during observing nanoparticles in water [11]. The height Z of the nano-step height resin was approximately less than 100 nm from the reference surface, which was fabricated by the nanoimprint process [13]. In the experiment, polystyrene (PS) f100 nm particles randomly adhered to the nano-step height and reference surface in water, which were used to measure the absolute height Z of the nanoparticle by applying multi-wavelength evanescent fields microscopy.

2. Nanoparticle observation in the evanescent field microscopy

2.1. Single-wavelength evanescent field

The evanescent field generated from the total internal reflection (TIR) occurs when light is internally reflected off an interface from a higher refractive index n_1 material to the material with lower index n2 at an incident angle i greater than the critical angle: $\theta_c = \sin^{-1}(n_2/n_1)$. The evanescent field occurs at a lower refractive index n_2 side, generated only near the reference surface in a few hundred nanometers, as shown in figure 1.

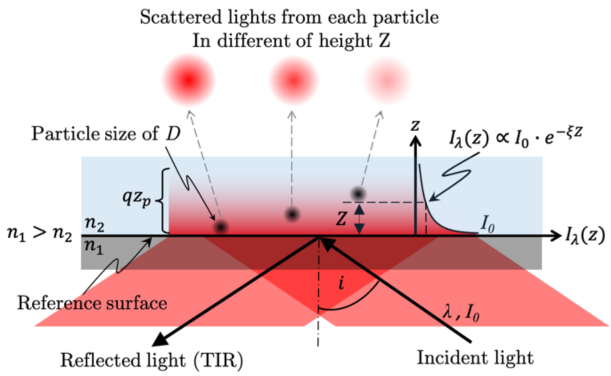

Figure 1. Single-wavelength evanescent field

The observation occurs when the particle size of *D* moves into the evanescent field. The height Z is a longitudinal distance between the reference surface to the nanoparticle. The intensity of the evanescent light decays exponentially with the distance of height Z from the reference surface [8]. The scattered light intensity I_λ from the particle at any height Z can be accumulated by an objective lens with a numerical aperture NA, which can be approximated by equation (1). Where I_0 and λ are the laser intensity and laser wavelength, respectively, ξ is attenuation coefficient given by equation (2).

$$I_\lambda(D,Z) \propto \int_{\theta=\cos^{-1}NA}^{\theta=\frac{\pi}{2}} I_\lambda\,[z_0(\theta)+Z]\times \pi D\cos\theta \times \frac{D}{2}\,d\theta$$

$$I_\lambda(D,Z) \propto I_0 \cdot \frac{1-\sqrt{1-NA^2}}{2}\cdot \pi D^2 \exp[-\xi Z] \qquad (1)$$

$$\xi = (4\pi/\lambda)\sqrt{n_1^2 sin^2 i - n_2^2} \qquad (2)$$

2.2. Multi-wavelength evanescent

We have added one more laser source in different wavelengths to generate multi-wavelength evanescent fields for determining the height Z of the particle at a higher resolution than single-wavelength [9-11]. Figure 2 demonstrates the scattered light intensities from nanoparticles at different heights Z in multi-wavelength evanescent fields. Hence, the scattered light intensities for both wavelengths I_{λ_1}, I_{λ_2} are expressed in equations (3) and (4). The height Z of the unknown particle size *D* can be obtained by substituting the ratio of the scattered light intensities ($I_{\lambda_1}/I_{\lambda_2}$) into equation (5), where k is a constant compensating for the height Z from the experiment. I_{0_1} and I_{0_2} are laser intensities, irradiated at incident angles i_1, i_2 (deg), in which both laser beams are total internally reflected from the reference surface. ξ_1, ξ_2 are attenuation coefficients for both wavelengths. Thus, the height Z of the particle can be determined by a two-dimensional image of the scattered light on optical multi-wavelength microscopy.

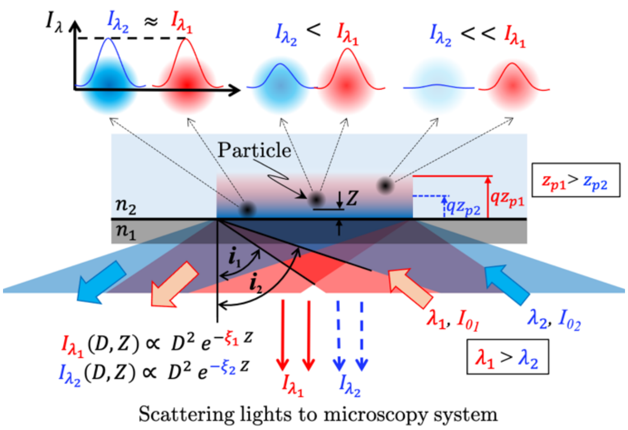

Figure 2. Multi-wavelength evanescent fields microscopy for height measurement of an individual nanoparticle

$$I_{\lambda_1}(D,Z) \propto I_{01} \cdot \frac{1-\sqrt{1-NA^2}}{2}\cdot \pi D^2 \exp[-\xi_1 Z] \qquad (3)$$

$$I_{\lambda_2}(D,Z) \propto I_{02} \cdot \frac{1-\sqrt{1-NA^2}}{2}\cdot \pi D^2 \exp[-\xi_2 Z] \qquad (4)$$

$$Z = \frac{1}{\xi_2-\xi_1}\ln\left\{k\cdot\frac{I_{02}}{I_{01}}\cdot\frac{I_{\lambda_1}}{I_{\lambda_2}}\right\} \qquad (5)$$

3. The frabrication process of the invisible nano-step height

An invisible nano-step height in water was proposed to verify the absolute height Z of the nanoparticle in water. Therefore, an optical resin (NOA1348) with a refractive index (n≈1.35) close to a refractive index of water is used to fabricate the nano-step height because it is an invisible object underwater [11]. The stamping mould made from polydimethylsiloxane (PDMS) was used to form the nano-step height. The height and width dimensions of the stamp mould were approximately 100 nm and 10 μm, respectively, measured by atomic force microscopy (AFM), as shown in figure 3.

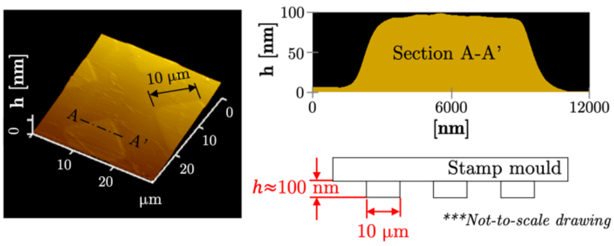

Figure 3. The AFM result of the PDMS stamp mold for nano-step height

The nanoimprint lithography process was employed to fabricate the invisible nano-step height resin on the glass surface, in which there are five main procedures, as shown in figure 4. After the nanoimprinting process, the nano-step height's dimension could be measured less than 100 nm in several heights using an interferometer, as shown in figure 5. Thus, the nano-step height resin on the glass surface was accepted to verify the nanoparticle's height in water (Z <100nm).

The interferometer result is used to evaluate some areas of the nano-step height only, which could exhibit the inclined surface, as shown in cross-section B-B' of figure 6. However, the result could not be used to validate the absolute distance of nanoparticles because we could not measure the nano-step height at the same area and time. Furthermore, the nano-step height resin (NOA1348) gradually evaporated on the glass surface, leading to thin layer aggregations and failures, depending on several conditions [14].

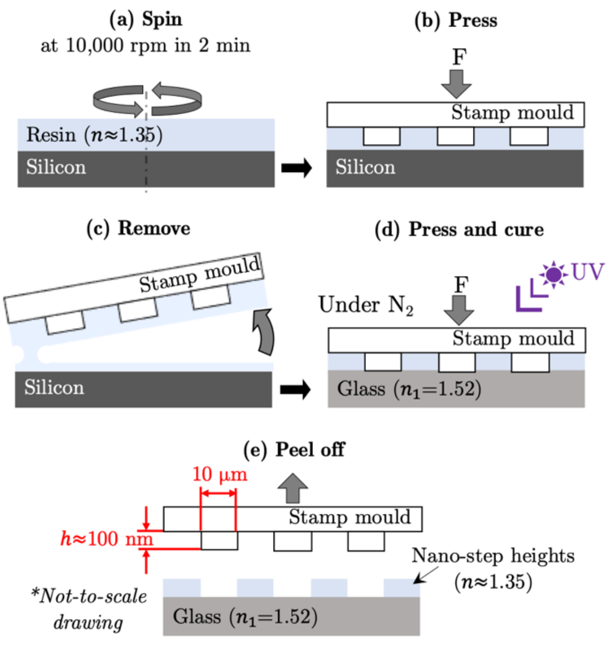

Figure 4. The frabrication process of the nano-step height resin

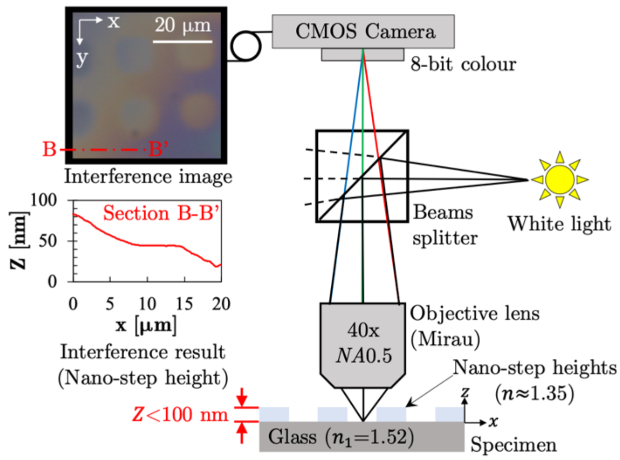

Figure 5. Illustration of the measurement of the nano-step height on glass surface using interferometer

4. Verification results of the absolute height Z of nanoparticles

The fabricated nano-step height (Z < 100nm) was employed to verify the height Z of nanoparticles in water by using multi-wavelength evanescent fields microscopy. Polystyrene (PS) ϕ100 nm standard particles (0.001wt%) in water were used to verify the absolute height Z because of the highly uniform particle size and high dispersity. Figure 6 demonstrates the developed apparatus for detecting scattering light intensities from nanoparticles on an optical multi-wavelength evanescent field microscopy. The experimental parameters and conditions of the optical microscopy system are listed in Table 1.

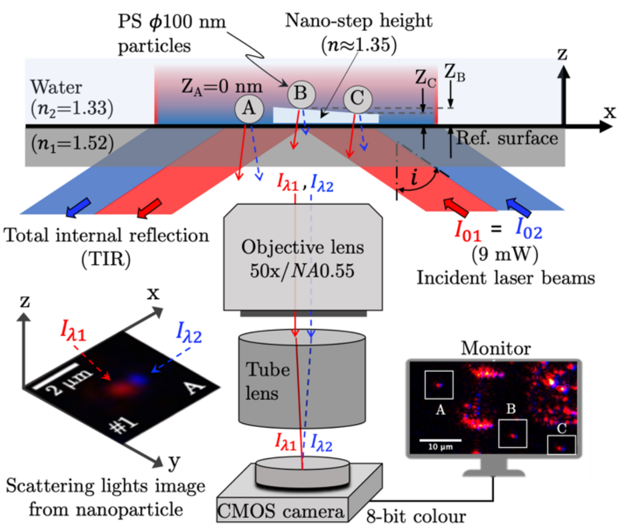

Figure 6. Illustration of the developed apparatus on optical multi-wavelength microscopy for nanoparticle height measurement

Figure 7 (a) shows the nano-step height on the glass or reference surface in the air, observed by the bright-field imaging mode on the developed apparatus. PS ϕ100 nm particles in water were dropped on the evanescent field area. Then they adhered to the glass and nano-step height surface in water randomly, in which there were three particles located on the verification surface, as shown in Figure 7 (b). Figure 7 (c) shows the scattered light image of nanoparticles in multi-wavelength evanescent fields. In the experiment, the nano-step height almost disappeared after it was coated with water, which could not detect the scattering light from the nano-step height. Therefore, it is accepted in experimental verification for nanoparticle height Z. Figure 7 (d) shows the sketched side-view of nanoparticles' location. Particle A adhered to the glass surface, and particles B and C adhered to the nano-step height surface. However, the actual value of the nano-step height is unknown yet, but it could be evaluated less than that of 100 nm by using the interferometer.

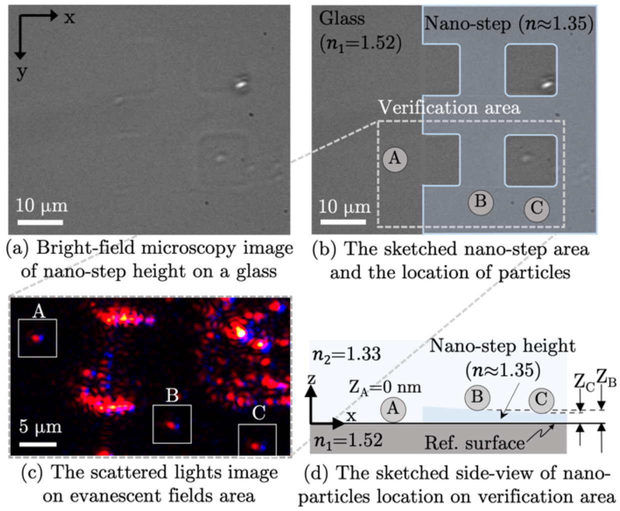

Figure 7. Illustration of nanoparticles locations that adhered on the glass and nano-step height surface, and also demonstrates the scattered light intensities from nanoparticles in the water in optical multi-wavelength evanescent fields

Table 1 Optical systems specifications and conditions in experimental verification of nanoparticle height

Laser Illumination		λ_1	λ_2
-Wavelength: λ	nm	642	450
-Power laser: $I_{01}=I_{02}$	mW	9	9
-Incident angle: $i_1=i_2$	deg	75	
Refractive index: n			
-Glass: n_1		1.52	
-Water: n_2		1.33	
-Nano-step height (NOA1348)		1.35	
-Polystyrene nanoparticle		1.59	
Optical microscopy system			
-Total magnification		76x	
-Numerical aperture (NA)		0.55	
CMOS camera (8-bit colour)			
-Pixel size	μm²	6.9×6.9	
-Frame rate	fps	20	
-Exposure time	ms	50	
-Recording images	frame	100	

In the experiment, the scattered light intensities from nanoparticles were recorded in 100 images at a frame rate of 20 fps with an exposure time of 50 ms. The scattered light intensities from the nanoparticle (particle A) was split into two images to integrate intensities of each wavelength, as shown in figure 8. Then, the integrated scattered light intensities ratio for both wavelengths ($I_{\lambda_1}/I_{\lambda_2}$) were substituted into equation (5) to determine the height Z of the nanoparticle. The constant compensation k was 0.84, which is defined by the ratio of the scattering light intensity ($I_{\lambda_1}/I_{\lambda_2}$ = 1) from particle A because the height Z of particle A on glass or reference surface was assumed that equal to zero (Z=0), which is used to calibrate particles B and C, respectively.

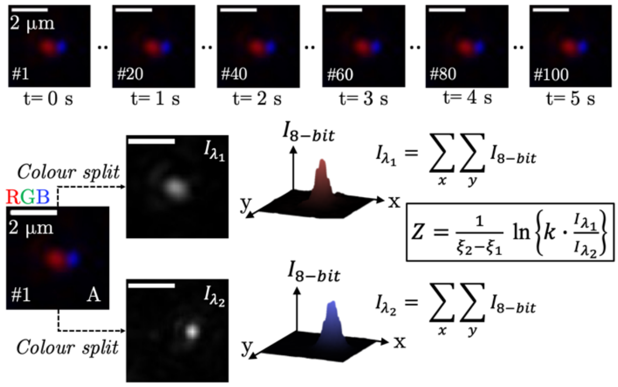

Figure 8. Calculating process for height Z of particle A

After compensation, the calculated height Z of three particles were plotted with time, as shown in figure 10. The averaged height Z of particles A, B and C were 0, 50, and 31 nm, respectively. Moreover, we found that the measurement uncertainty of the height Z was less than ±4.2 nm (two times the standard deviation: 2σ from 100 measurements). Figure 10 shows the exponential relationship between the nanoparticle height Z and the multiplied scattered light intensities of three particles, implying that the increased height Z decreased the scattering light intensity. However, the absolute height Z of nanoparticles is unknown yet, but it could be calculated less than 100 nm, related to the interferometer results. Furthermore, we found that the nano-step height resin evaporated after experimental height Z verification, which could not repeat the experiment.

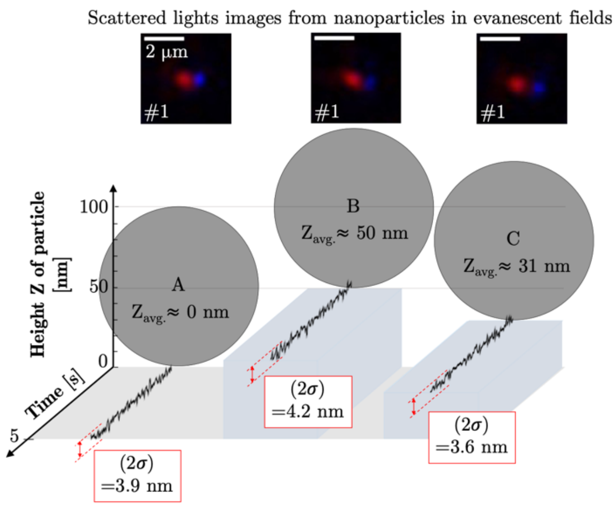

Figure 9. The calculated height Z results of three nanoparticles

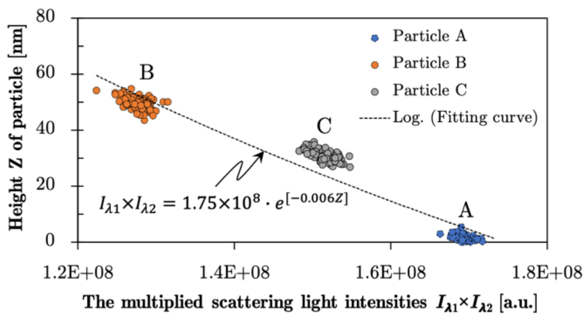

Figure 10. The exponential relationship between the nanoparticle height Z and the multiplied scattering light intensities of three particles

5. Conclusion

The nano-step height made from optical resin ($n \approx 1.35$) was proposed to verify the absolute heigh Z of polystyrene ϕ100 nm particle in water because it was invisible underwater. The height Z of nanoparticle were estimated based on a calculation to evaluate the possibility of the proposed method on the optical multi-wavelength microscopy. After calculating the height Z, we found that:

i. The height Z of nanoparticles was less than 100 nm, which agreed with the interferometer trend of nano-step height.
ii. The measurement uncertainty of height Z of nanoparticles was less than ±4.2 nm (2σ) from 100 measurements.
iii. The results of heights Z and the scattering light intensities of three particles were agreed, as the exponential trends.

However, the absolute height Z of nanoparticles are not confirmed yet due to the unstable of the nano-step height. In our future work, we attempt to repeat and validate the height Z of the particle and nano-step height at the same position. The outcome of the proposed method would be applied to clarify some phenomena of the nanoparticle near-surface in wet conditions during precision manufacturing processes.

Acknowledgements

This work had been supported by KAKENHI Grant-in-Aid for Scientific Research (C) 16K06015. The authors would also like to express our sincere to the Center for Microelectronic System (CMS), Kyushu Institute of Technology, to fabricate the master mould silicon of nano-step height.

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