

Article

Detachment Energy Evaluation in Nano-Particle Cleaning Using Lateral Force Microscopy

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Abstract: It has been difficult to detach abrasive particles smaller than 50 nm from polished surfaces in post-CMP cleaning. During the cleaning process, the residual nano-particles exert shear force in the inevitable shear flow. In order to understand the cleaning mechanism, it is indispensable to investigate not only the force but also the energy acting on different-sized nano-particles. In this article, we proposed the evaluation of detachment energy (the energy required to detach nano-particles) by using Lateral Force Microscopy. As an example, the dominant detachment energy of the silica nano-particle between the oxide film is the potential energy of the hydrogen bond. It suggested that the silica nano-particle detachment involves the breaking of hydrogen bonds.

Keywords: detachment energy; hydrogen bond; shear flow; lateral force microscopy; post-CMP cleaning; SiO₂ nano-particle; oxide film



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1. Introduction

In recent years, abrasive particles smaller than 50 nm have been applied in Chemical Mechanical Polishing (CMP) techniques to achieve a surface with nanoscale flatness within the depth of focus in a lithography process in each layer. This advancement is crucial for realizing multilayer wiring structures and reducing wiring widths down to 7 nm in high-performance large-scale ICs [1–3]. Due to this, the presence of residual particles on the polished film surface can be a killer defect (short or opens in electric circuits, etc.) as a mask in the lithography process [4], causing partial convexity in the subsequent film deposition process [5], etc., and then leading to lower LSI chip yields. In particular, the oxide films that serve as interlayer insulators and are polished with smaller silica nano-particles are used in the process. Hence, detaching smaller nano-particles (abrasive particles, cleaning waste/dust, etc.) from polished surfaces has indeed posed a significant challenge in post-CMP wet cleaning processes.

In general, contact cleaning by polyvinyl alcohol (PVA) brush scrubbing and non-contact cleaning by enforcing MHz waves were applied to detach nano-particles from the surface. Although the cleaning process is being reconsidered to realize optimal and efficient cleaning [2,6], the cleaning phenomenon has not yet been clearly understood because the residual contamination on the wafer surface is typically inspected only after the cleaning process in dry conditions, rather than wet conditions using the surface scanning methods (e.g., scanning electron microscopy). Consequently, it is essential to investigate the cleaning mechanisms, which involve the detachment of the nano-particle from the surface to be cleaned, and the occasional reattachment to the surface.



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Thus, we have established a direct observation method for studying the cleaning phenomena that occurred in a range of a few hundred nanometers from the surface by employing an evanescent light which generated a few hundred nanometers from the surface to be cleaned [7–13]. By using this observing method, size-dependent particle behaviors during cleaning were evaluated in the cleaning phenomenon [14–17], especially in the presence of inevitable shear flow [18]. As a result, the smaller particles required considerably more time to detach. This size-dependent detachability of the nano-particle is considered involvement with the physicochemical energy between the nano-particle and the substrate in the shear flow. However, the potential energy of the physicochemical interactions has not been investigated quantitatively.

Various research groups have evaluated the vertical force to pull the particle with a diameter of a few micrometers at the smallest size from the substrate surface by the force curve mode of AFM [19] or by numerically calculating the interaction (van der Waals force, repulsive forces by the electric double layer, etc.) of the nano-particle and the substrate under theoretical models [20]. Hence, the energy evaluation of the nano-particle detachment in the action shear force (lateral force) was indispensable.

In this article, we proposed an evaluation method of energy required to detach various-sized nano-particles from the substrate by using Lateral Force Microscopy (LFM) in order to investigate the dominant interactions. Distinctively, the energy quantity is not a vector quantity like the interacting force, but a scalar quantity at the nanoscale. Therefore, the phenomenon could be evaluated in terms of its scalar quantity as the physicochemical energy, and not only evaluated in terms of the acting behavior to nano-particles in one or two directions and at a certain moment.

As an example, the detachment energy of the silica nano-particle from the oxide film (SiO_2 film on a silicon wafer) surface was measured such as in post-CMP cleaning as shown in Figure 1. The dominant energy to detach the particle which involves breaking the bond was discovered.

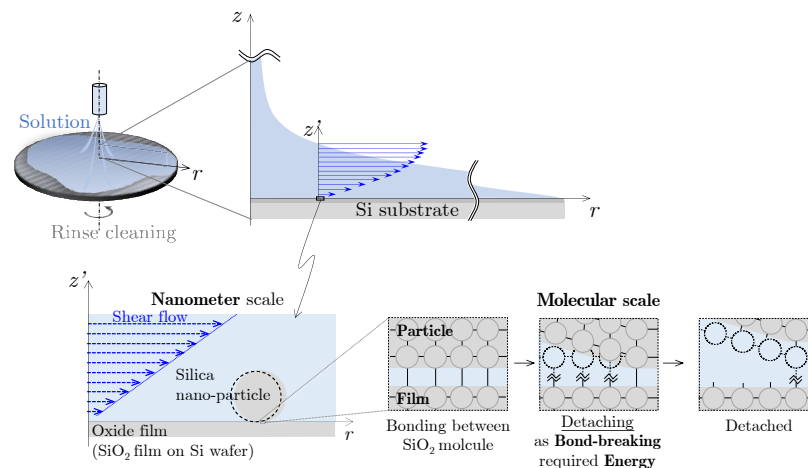


Figure 1. Nano-particle detachment involves breaking the bonding such as in post-CMP cleaning.

2. Measurement Method of Nano-Particle Detachment Energy Using Lateral Force Microscopy

We used a method that can measure the shear action force to detach the nano-particles adhered to the substrate surface [21,22]. The position and the size (height) ($H_{\text{topography}}(V_T)$) of the nano-particles were determined by Atomic Force Microscopy (AFM) before and after detachment. In order to measure the force required to detach the nano-particle from the surface, we detached the nano-particle from the surface and moved it on the surface by applying a force through contact with the tip of the cantilever using LFM (Figure 2). The elastic torsion of the cantilever was detected as a voltage signal (V_L) by the position-sensitive detector (PSD). This signal indicates the variation in the lateral force F_{Lateral} acting on the particle during the probe's movement in the x -direction and is converted to lateral force

through calibration [22,23] (the calibration method is in the Supplementary Information). When the torsion is released, it can be regarded as the detachment of the particles.

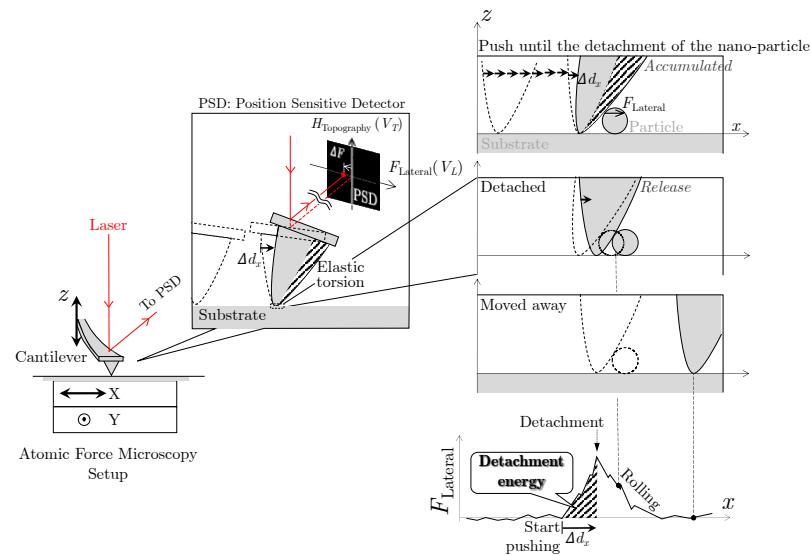


Figure 2. Measurement of lateral force variation during detachment of nano-particle.

The energy required to detach (detachment energy) can be calculated by the variation of lateral force and the displacement Δd_x of the cantilever tip while pushing the nano-particle until it detaches. Then, the surface interrelationship between the particle and the film was quantitatively investigated.

3. Experimental Results of Detachment Energy for Various-Sized Silica Particles from an Oxide Film Surface

3.1. Measurand of the Force Required to Detach

Silica standard particles with diameters of $D = 52 \pm 3$, 80 ± 3 , and 310 ± 19 nm (nanoComposix NanoXact™ Silica Nanospheres, San Diego, CA, USA) were adhered to the oxide film surface after the suspension diluted in a pH10.5 solution had naturally dried. The probe was scanned on the surface in an LFM mode (XE-300P AFM, Park Systems, Suwon, Republic of Korea) within 30 min. The scanning speed was 1000 nm/s in the x -axis, and the sampling time Δt was 1/256 s. When scanning in the x -direction at any y -coordinate is completed, the probe moves slightly in the positive y -direction and starts scanning in the x -direction from the $x = 0$ nm point again. The typical results of the images showed the horizontal movement of the $D = 80$ nm silica particle as shown in Figure 3. It was clearly confirmed that the nano-particle was detached and moved by the probe pushing.

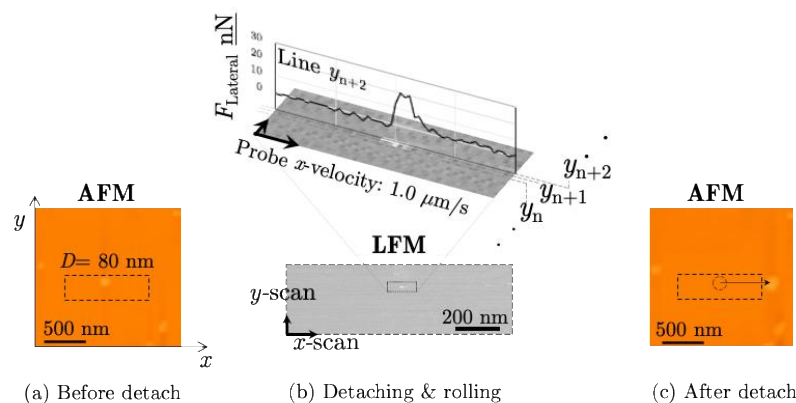


Figure 3. $D = 80$ nm silica particle's position and lateral force variation until detaching from the nano-particle.

Figure 4 shows lateral forces before acting (Line y_n) and during acting on the SiO₂ particles (Line $y_{n+1} \sim y_{n+2}$) and after detaching (Line y_{n+3}). The timing of the nano-particle detachment was known as the lateral force maximum. The lateral force was converted from the measured voltage which indicated that the displacement of the laser spot moved on the position-sensitive detector when the cantilever was twisted. The resolution of measurable lateral force in this experiment was 2 nN (2σ) which calculated using the force variation during scanning on the oxide film in line y_n . Subsequently, the work to detach W_{lfm} was analyzed on the line y_{n+2} that the particle detached from on the surface.

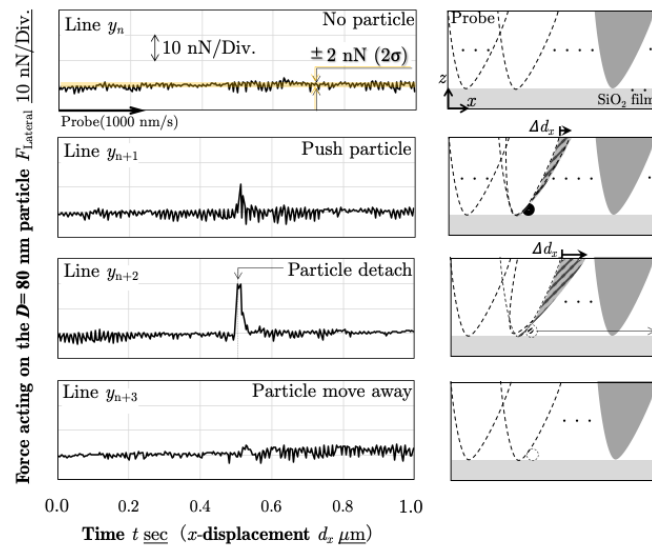


Figure 4. The force acting on $D = 80$ nm silica particle per line and particle behaviors during scanning.

3.2. Measurements of the Detachment Energy

Figure 5 shows the variation in lateral forces acting on the silica particle during detachment from the oxide film surface. The work to detach the particle W_{lfm} that was drawn by hatching was calculated by the changes in lateral force F_{Lateral} and the displacement Δd_x of the cantilever tip from the moment the probe initiated contact with the particle t_0 until the particle detached at t_n by Equation (1).

$$W_{\text{lfm}} = \sum_{i=t_0}^{t_n} (F_{\text{Lateral}, i} \cdot \Delta d_{x,i}) \quad (1)$$

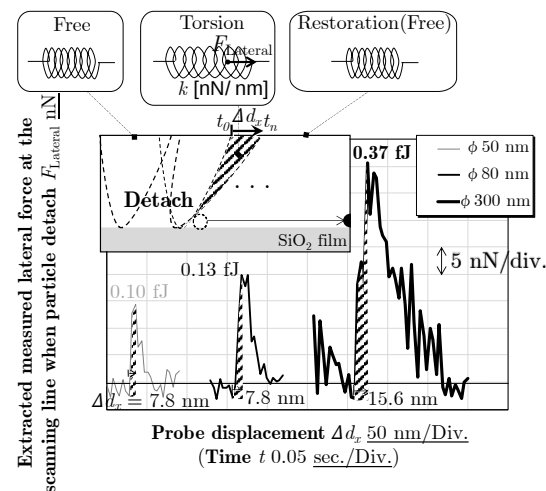


Figure 5. Lateral force variation during the detachment of various-sized silica nano-particles on the oxide film surface.

This work is equivalent to the energy required to detach a nano-particle (detachment energy).

4. Theoretical Calculation of Interaction Energy

The work to detach the silica particle from the oxide surface W_{lfm} indicates the physicochemical interaction energies. These interaction energies (siloxane bonds, hydrogen bonds, and van der Waals interactions as shown in Figure 6) were calculated below to discover the dominant energy with physicochemical parameters as shown in Table 1.

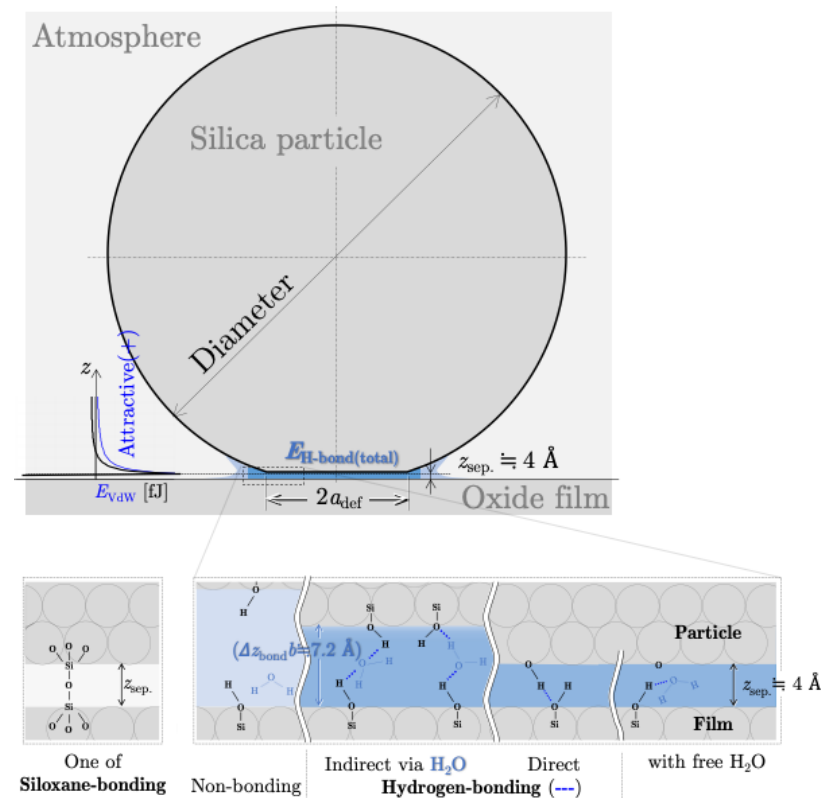


Figure 6. Calculated interaction energies of siloxane bonds, hydrogen bonds, and van der Waals interactions between the spherical elastic silica particle and the oxide film surface.

Table 1. Physicochemical parameters.

Potential Energy of Bonding Interaction			
Siloxane bond energy [24]	$E_{\text{S-bond}} @ \geq 200 [^{\circ}\text{C}]$	kJ/mol	444
Hydrogen bond energy [25]	$E_{\text{H-bond}} @ \leq 200 [^{\circ}\text{C}]$	kJ/mol	20.9
OH group concentration [26–28]	$\rho_{\text{OH}} @ \leq 200 [^{\circ}\text{C}]$	OH/nm ² ($\mu\text{mol}/\text{m}^2$)	4.6 ± 0.6 (7.64 ± 1.0)
Total length of bond chain with forming probability b [26]			
SiO ₂ /SiO ₂ interaction	$\Delta z_{\text{bond}} b$	Å	≈ 7.2
Potential energy of van der Waals interaction			
Hamaker constant [29]			
SiO ₂ –atmosphere–SiO ₂	$A_{\text{@atmosphere}}$	$\times 10^{-20}$ J	6.00
SiO ₂ @atmosphere	$A_{\text{SiO2@atmosphere}}$	$\times 10^{-20}$ J	6.00
Separated distance between the particle and the film [30,31]			
	z_{sep}	Å	4
Contact radius calculation by JKR theory			
Elastic modulus (SiO ₂ /SiO ₂) [32]	K	N/m ²	4.9×10^{10}
Poisson's ratio [33]	ν_{SiO2}	–	0.2
Young's modulus [33,34]	E_{SiO2}	GPa	70

4.1. Potential Energy of Siloxane Bonds

The SiO₂ surface was hydroxylated [35], and various silanol groups related to the siloxane bridge were formed (Si–O–Si) in an environment above 200 °C. The concentration

of the siloxane bridge increases when the silicas are heated under a vacuum because the OH group concentration on the surface decreases monotonically with increasing temperature. Its total siloxane bonding energy $E_{\text{S-bond(total)}}$ is calculated as follows [26]:

$$E_{\text{S-bond(total)}} = \rho_{\text{OH}} S_{\text{area}} E_{\text{S-bond}} \quad (2)$$

where ρ_{OH} is the concentration of the OH group, $E_{\text{S-bond}}$ is the siloxane bonding energy per mole, and S_{area} is the total interaction area between particle and substrate (film). The interaction area is calculated as follows:

$$S_{\text{area}} = \pi(a_{\text{def}})^2 + 2\pi(D/2)\Delta z_{\text{bond}}b \quad (3)$$

where the first term is the deformation area of the particle (a_{def} is the contact radius calculated by the JKR model [32]), and the second term represents the surface area of the spherical crown at a height Δz_{bond} near the contact point with a probability b . The typical calculated results are shown in Table 2.

Table 2. Typical calculated results of energy of siloxane bond and hydrogen bond.

Diameter [nm]	$E_{\text{S-bond(total)}} [\text{fJ}]$	$E_{\text{H-bond(total)}} [\text{fJ}]$	$S_{\text{area}} [\text{nm}^2]$	$a_{\text{def}} [\text{nm}]$
10	0.13	0.01	38	2.2
100	1.92	0.09	567	10.4
1000	32.5	1.53	9595	48.3

4.2. Potential Energy of Hydrogen Bonds

At temperatures below 200 °C, hydrogen bonding (physisorption) occurs as water remains on the surface [27]. The bond formed between a positively charged hydrogen atom and another negatively charged atom (in this paper, an oxygen atom) is called a hydrogen bond. As shown in Figure 6, hydrogen bonds include cases in which the oxygen and hydrogen atoms of the Si-OH groups on the silica nano-particle or the oxide film surface bond indirectly via water molecules or directly. The total potential energy of a hydrogen bond is calculated [26] as follows:

$$E_{\text{H-bond(total)}} = \rho_{\text{OH}} S_{\text{area}} E_{\text{H-bond}} \quad (4)$$

where ρ_{OH} is the concentration of the OH group, $E_{\text{H-bond}}$ is the hydrogen bonding energy per mole, and S_{area} is the total interaction area between the nano-particle and the film. The typical calculated results are shown in Table 2.

4.3. Potential Energy of van der Waals Interaction

The van der Waals force generated between atoms and molecules is generally attractive in nature due to the motion of electrons (Figure 6). For a deformed particle of a diameter D on a flat surface, the interaction energy E_{VdW} is given by the following [36]:

$$E_{\text{VdW}} = -\frac{AD}{12z_{\text{sep}}} \left[1 + \frac{(a_{\text{def}})^2}{Dz_{\text{sep}}} \right] \quad (5)$$

where A is the Hamaker constant, z_{sep} is the separation distance between the particle and the substrate, and a_{def} is the contact radius. The typical calculated results are shown in Table 3.

Table 3. Typical calculated results of energy of van der Waals interaction.

Diameter [nm]	E_{VdW} [fJ]	a_{def} [nm]
10	1.32×10^{-4}	0.5
100	1.39×10^{-3}	2.1
1000	1.56×10^{-2}	9.9

4.4. Dominant Interaction Energy for Detachment

Figure 7a and Table 4 show each calculated energy in the atmosphere using physicochemical properties (Table 1). As a result, the work to detach the particle from the film $W_{lfm} \propto D$ showed similar characteristics to the potential energy of hydrogen bonding $E_{H-bond} \propto D^{1.2}$. Therefore, the effective total interaction energies in the atmosphere were calculated by Equation (6) as shown in Figure 7b.

$$E_{req@atmosphere} = E_{H-bond(total)} + E_{VdW} \quad (6)$$

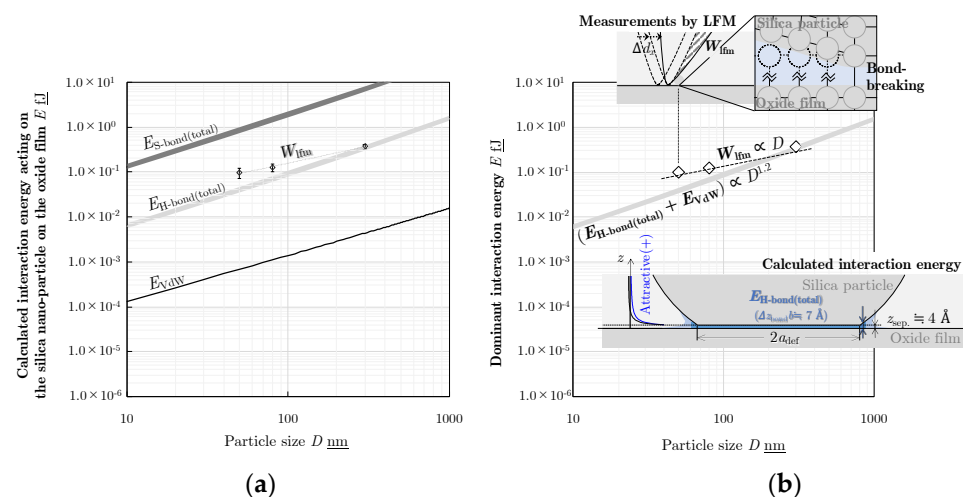


Figure 7. The potential energy of bonding and interaction between the silica nano-particle and the oxide film surface in the atmosphere. (a) Each calculated theoretical potential energy; (b) dominant total interaction energy.

Table 4. Measured results of work to detach nano-particle and calculated potential energy.

Diameter [nm]	W_{lfm} [fJ]	$E_{S-bond(total)}$ [fJ]	$E_{H-bond(total)}$ [fJ]	E_{VdW} [fJ]
50	0.10	0.84	0.04	6.81×10^{-4}
80	0.13	1.47	0.07	1.11×10^{-3}
300	0.37	7.30	0.34	4.36×10^{-3}

5. Discussion

The dominant energy required to detach the particle from the surface is the energy of the hydrogen bond (Si-OH bonding) represented as $E_{H-bond(total)}$ between the silica particle and the oxide film. This indicates that the particle detachment involves breaking the hydrogen bond between the nano-particle and the film. The possible dominance (importance) of hydrogen bonding had already been pointed out by estimation by measuring the vertical force interaction [25]; however, our experimental results measured the lateral force in the same direction as observed in the cleaning phenomenon, and a theoretical comparison **determined** it clearly. Another reason for the dominance of hydrogen bonding is due to the formation of silanol groups on the surface and enabling hydrogen bonds to occur because the surfaces of the silica nano-particles and oxide film are hydrated in the alkaline solution

(pH 10.5). In the case of the siloxane bond, it was significantly higher, suggesting that it required a higher temperature in the atmosphere, unrelated to the water reaction [27].

In addition, the detachment energy density was considered to evaluate the particle's size-dependent detachability as shown in Figure 8. It was implied that the smaller particle size is difficult to detach from the surface due to higher energy density (energy per contact area).

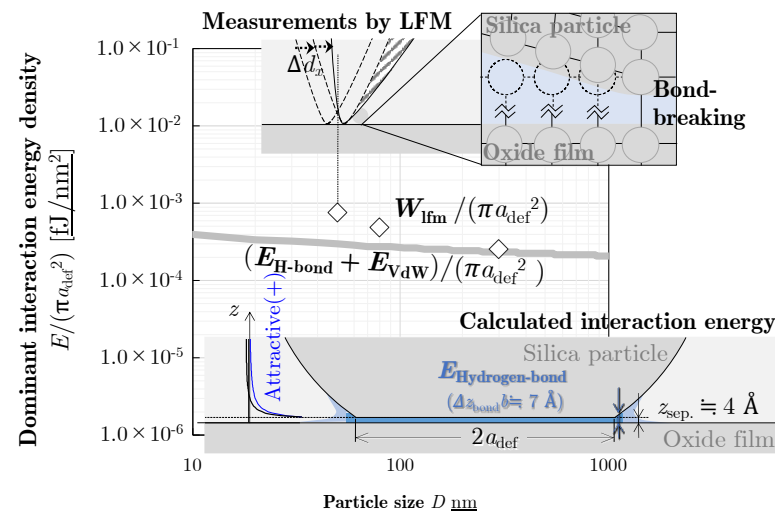


Figure 8. Detachment energy density between the silica particle and the oxide film surface.

6. Conclusions

The detachment energy evaluation method was proposed to investigate the dominant interaction in the cleaning using Lateral Force Microscopy. In this article, the detachment energies of the silica nano-particle with a diameter of 50, 70, and 300 nm from an oxide film (SiO_2 film on the silicon wafer) such as in the post-CMP cleaning were measured. It was discovered that the dominant detachment energy of the silica nano-particles from the oxide film is the potential energy of hydrogen bonding. This indicates that the particle detachment involves breaking the hydrogen bond between the silica nano-particle and the oxide film.

As described above, the effectiveness of this approach was demonstrated to discover the dominant interaction which involved the physicochemical energy between the nano-particle and the substrate. It leads to the quantitative investigation of the size-dependent detachability of the nano-particle in the cleaning phenomenon.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app14188145/s1>, Figure S1: Conversion method from the measured lateral voltage to the lateral force.

Author Contributions: Conceptualization, P.K. and S.H.; methodology, P.K.; validation (conduction of the experiment and consideration of the bonding), Y.T.; formal analysis, P.K., Y.T. and S.H.; instrumentation resources, J.S.; writing—original draft preparation, Y.T.; writing—review and editing, J.S.; investigation, Y.W. and H.H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: Authors Satomi Hamada, Yutaka Wada and Hirokuni Hiyama were employed by the company Ebara Corporation, Ltd. The remaining authors declare that the re-search was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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