

# Experimental analysis on material removal mechanism in CMP process for SiO<sub>2</sub> film with AFM observation

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# Kyutech

Kyushu Institute of Technology



# History

|      |   |
|------|---|
| 1909 | Founded in 1909 as Meiji College of Technology (4-year, private)  |
| 1921 | Became 4-year national institution  |
| 1949 | Under the national school establishment law, Meiji College of Technology was renamed Kyushu Institute of Technology.  |
| 1986 | Faculty of Computer Science and Systems Engineering was established.  |
| 2000 | Graduate School of Life Science and Systems Engineering (Independent postgraduate school) was established.  |
| 2004 | All National Universities transformed into National University Corporations; Kyushu Institute of Technology was also incorporated as a National University Corporation. |



**Dr. Kenjiro Yamakawa**  
First President



**Keiichiro Yasukawa**  
Founder



# Locations of Kyutech Campuses



**Wakamatsu Campus**  
Graduate School of Life Science and Systems Engineering

**Tobata Campus**  
School of Engineering



**Iizuka Campus**  
School of Computer Science and Systems Engineering

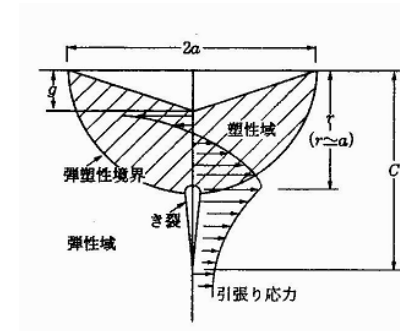
## □ 3 theories on material removal mechanism in polishing as follows :

### (1) Theory of Micro Cutting

Material removal with destruction by forcing abrasive grains into material

metal : plastic deformation

ceramics : cracks generation as Hertz crack



### (2) Theory of surface flow (Bowden, Tabor )

Heat generation at contact point → high temperature softening, melting → surface flow → formation of Beilby layer

F.P. Bowden, D. Tabor : The Friction and Lubrication of Solids, Oxford (1954)

### (3) Theory of chemical reaction

**Grebenschikov : soft gel layer on glass surface**

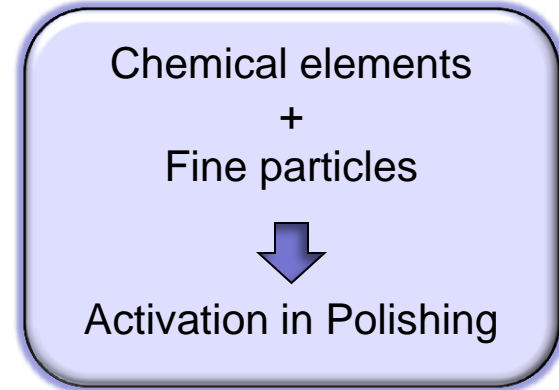
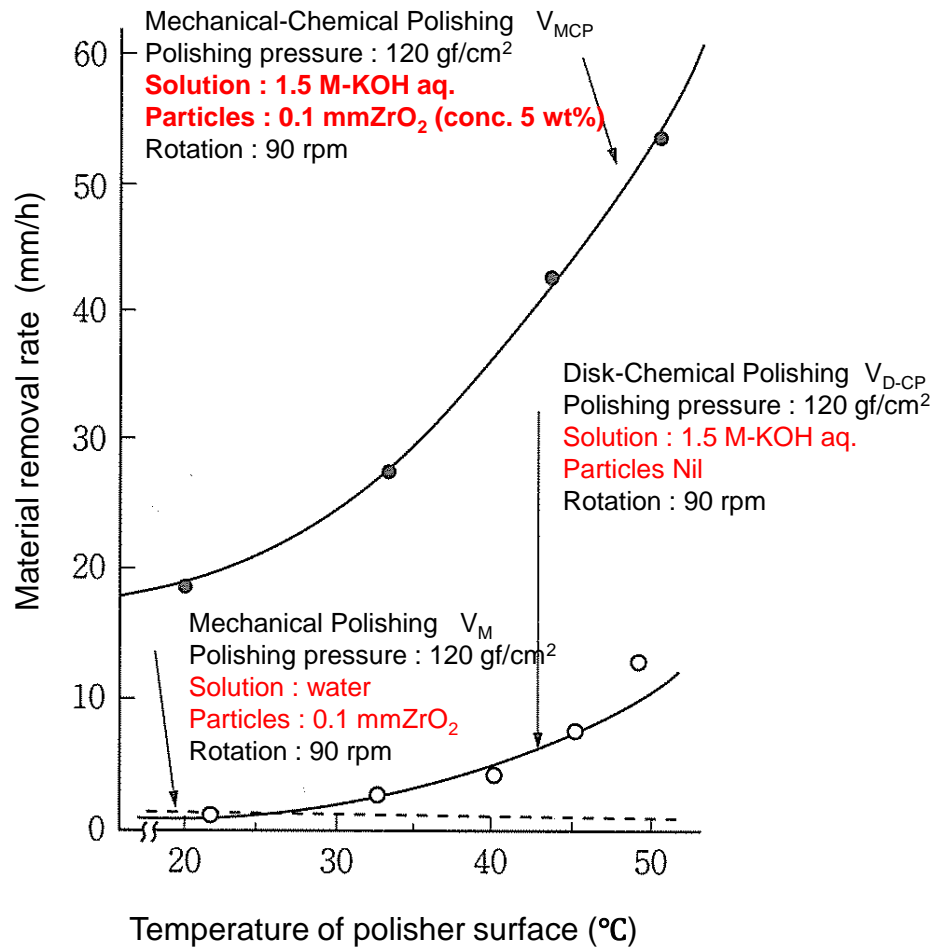
V. Grebenschikov : Keram, I Stekro, 7 [11/12] (1931)

**Kaller : chemical reaction among glass/polisher/polishing liquid/particles**

A. Kaller : Neuere Erkenntnisse über die Vorgänge beim Polieren des Glases, Mschr. Feinmech. Opt. 79 (1962) 5

**Izumitani : property of hydration layer determines polishing ability**

T. Izumitani : Treatise on Material Science & Technology, vol. 17, Academic Press, New York (1979)



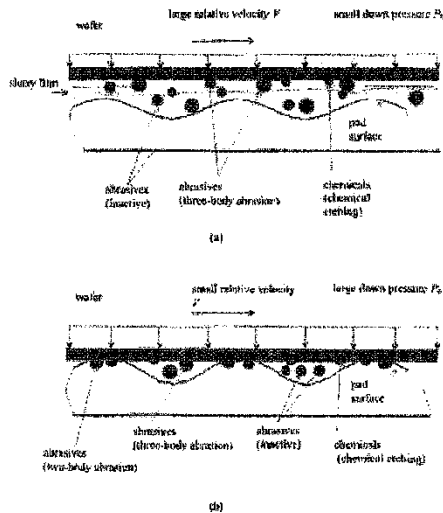
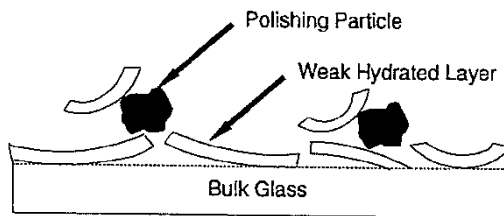
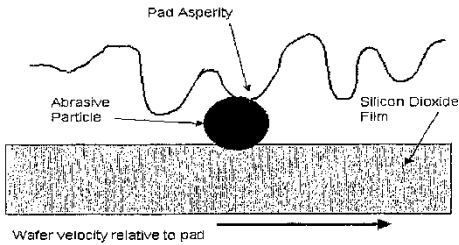
**Slurry's function**

chemical elements :  
chemical reacted layer formation

**fine particles :**  
**unidentified, but significant**

## Si single-crystal polishing

Toshiro Doi : Optical Technology Contact, 31 [10] (1993) 47



Material removal proceeds as mechanical cutting process with fine particles with backup of polishing pad

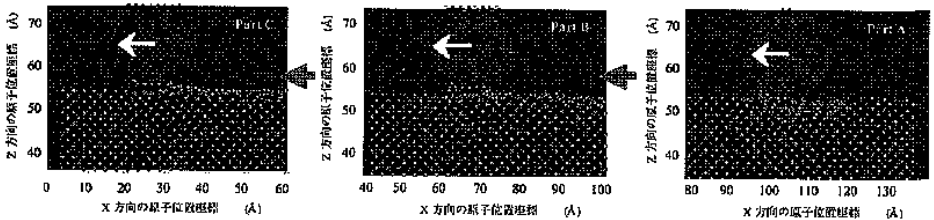
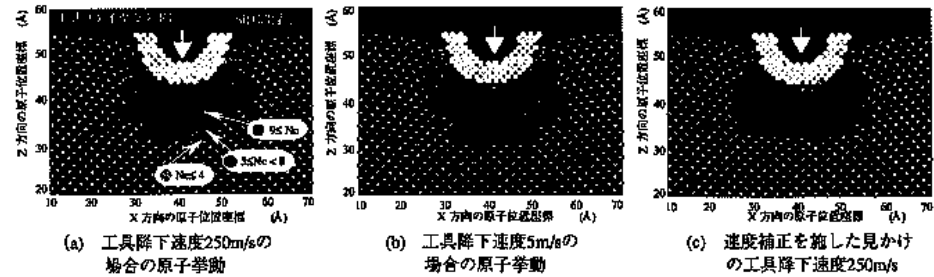
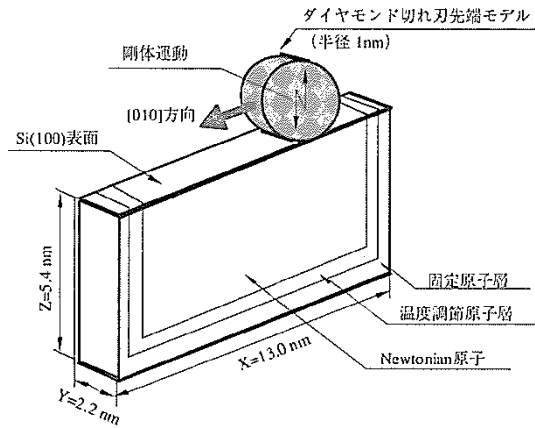
Material removal process proceeds as destruction and flaking of hydration layer on wafer surface with fine particles in slurry

N.B. Kirk, J. Wood : Glass polishing,

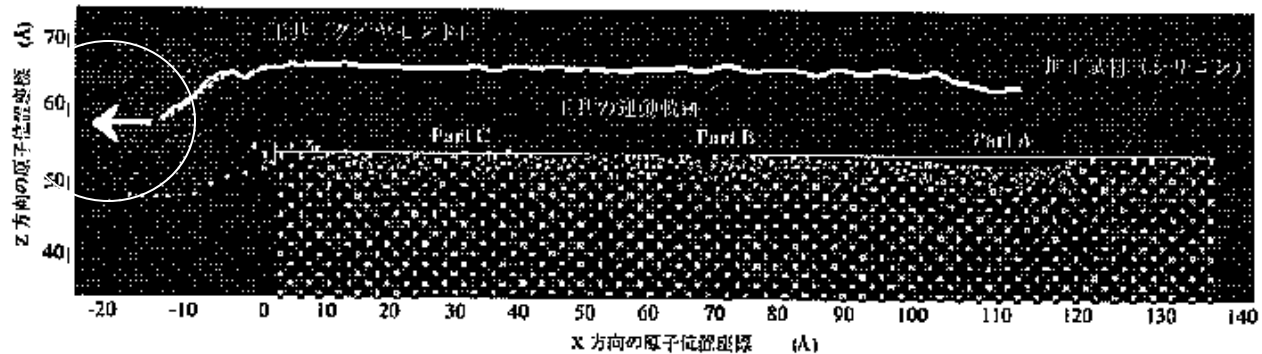
Is it realistic phenomena ?

Material removal with fine particles is the essential function !

J. Luo, D. A. Dornfeld : Material Removal Mechanism in Chemical Mechanical Polishing : Theory and Modeling, IEEE Transactions on Semiconductor Manufacturing, Vol. 14, No.2 (2001) pp112-133



(d) 工具移動距離8.0nm (Part C) での加工作用：表面原子層の配列をアモルファス化するが除去効果はない。  
 (e) 工具移動距離4.0nm (Part B) での加工作用：工具表面にSi原子が付着した状態で負の切り込みとなる。  
 (b) 工具移動距離0.8nm (Part A) での加工作用：工具表面にSi原子が付着し1原子層程度が除去される。

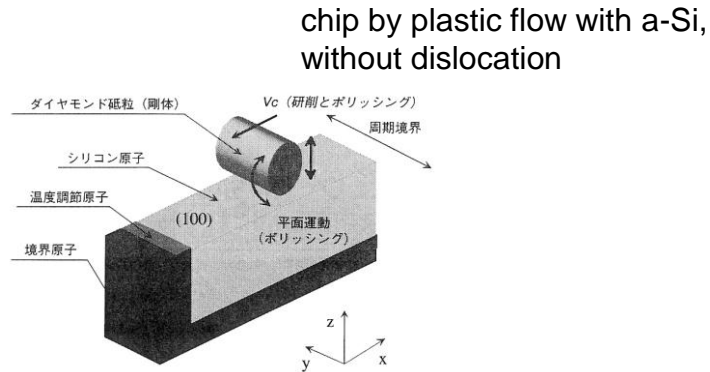


(a) 1回の工具運動と加工試料表面原子層の状態 (制御温度293K)

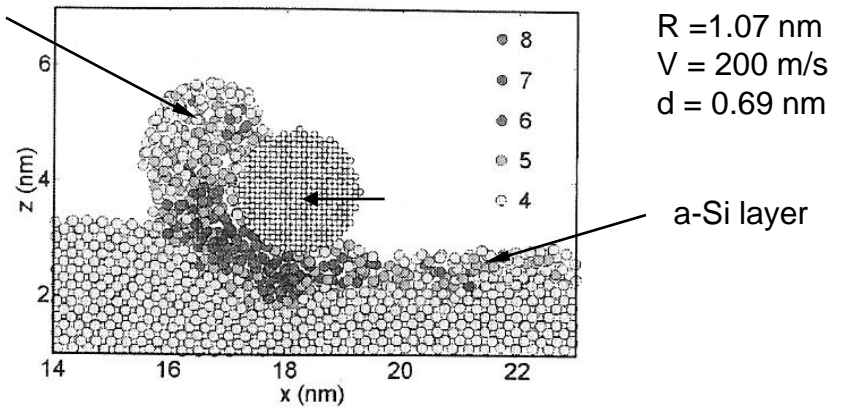
Y. Takaya, K. Kimura : Analysis on phenomena of optical radiation pressure micro-machining with molecular dynamics, J. of JSAT, 47,12 (2003) 677-683

高谷裕浩、木村景一：分子動力学による光放射圧マイクロ加工現象の解析、砥粒加工学会誌、47,12 (2003) 677-683

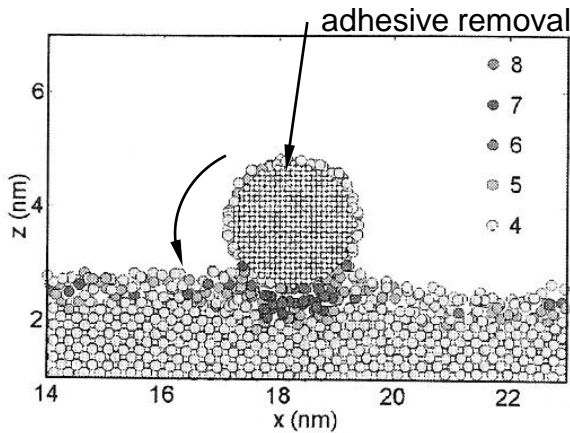




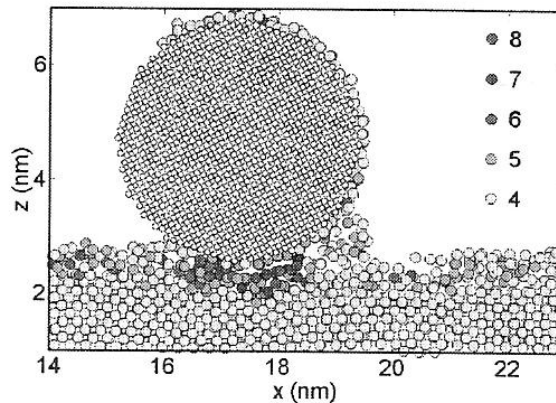
Initial model on molecular dynamics simulation



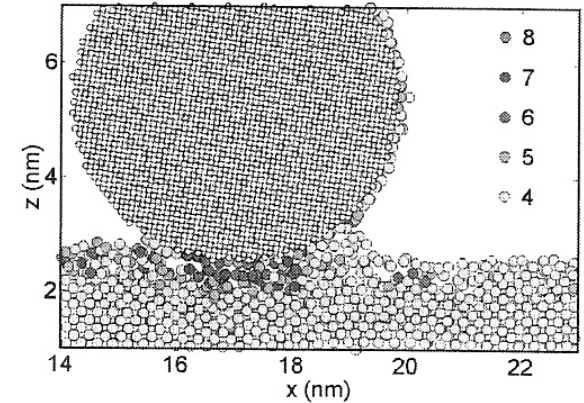
(a) Grinding ( $R=1.07\text{nm}$ )



(b) Polishing ( $R=1.07\text{nm}$ )



(c) Polishing ( $R=2.14\text{nm}$ )



(d) Polishing ( $R=2.85\text{nm}$ )

## Damaged layer on single crystal Silicon after grinding and polishing

- H. Tanaka, S. Shimada, N. Ikawa : Prediction of an ideal surface processing of monocrystalline silicon for minimal surface roughness and damage by molecular dynamics analysis, J. Soc. Grinding Engineers, 45, 4 (2001) 175-180 (in Japanese)

田中宏明, 島田尚一, 井川直哉: 分子動力学シミュレーションによる単結晶シリコン理想表面生成プロセスの予測, 砥粒加工学会誌 Vol.45, No.4 (2001) pp.175-180

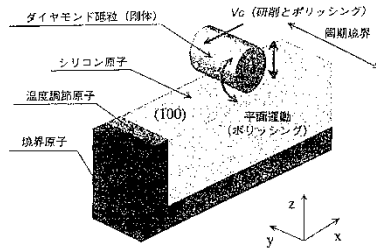
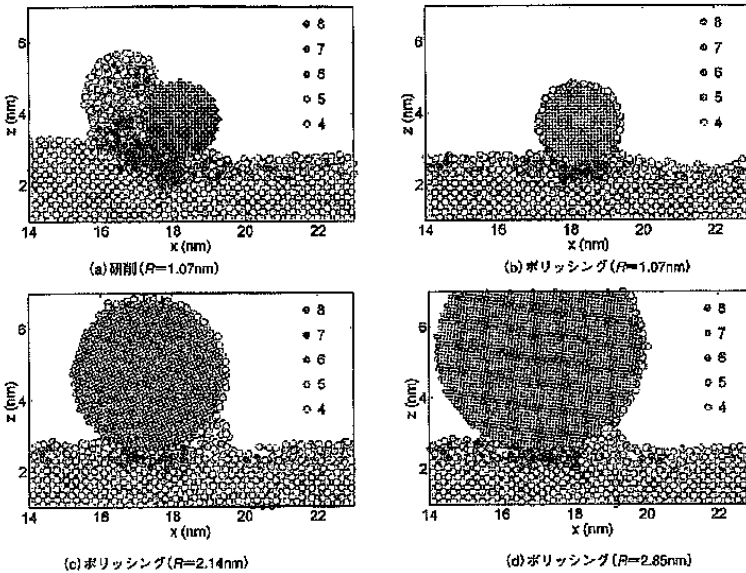


図1 分子動力学シミュレーションによる初期モデル



1. Particles' slide on the surface  
 - micro cutting  
 - amorphous layer formation

2. Atomic bonding force in amorphous layer is weak

3. Atoms in amorphous layer with weak bonding force adhere on contacting and rolling particles

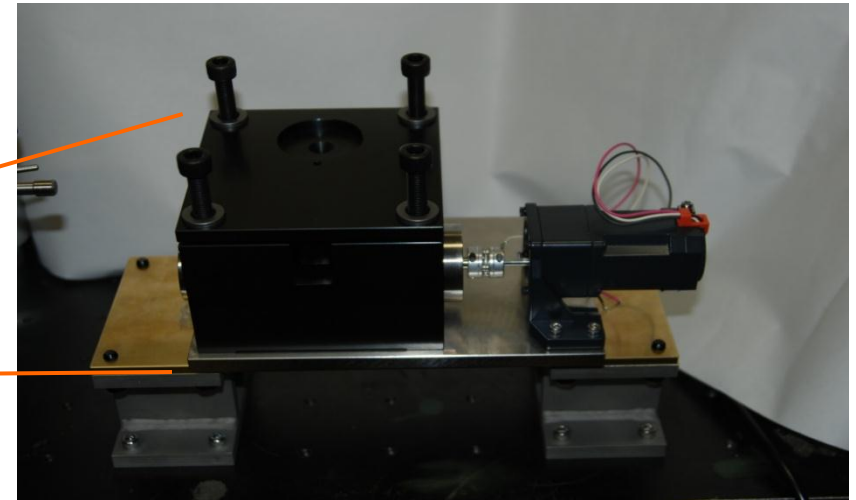
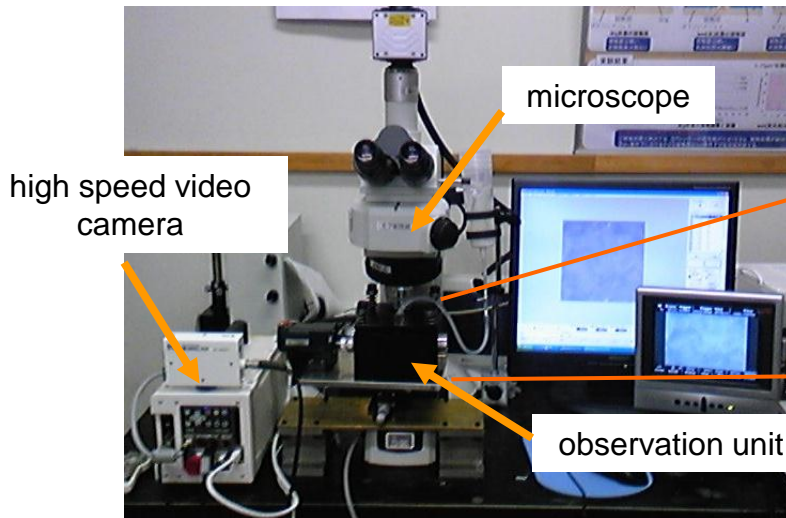
4. Material removal proceeds continuously

- H. Tanaka, S. Shimada, N. Ikawa : Prediction of an ideal surface processing of monocrystalline silicon for minimal surface roughness and damage by molecular dynamics analysis, J. Soc. Grinding Engineers, 45, 4 (2001) 175-180 (in Japanese)

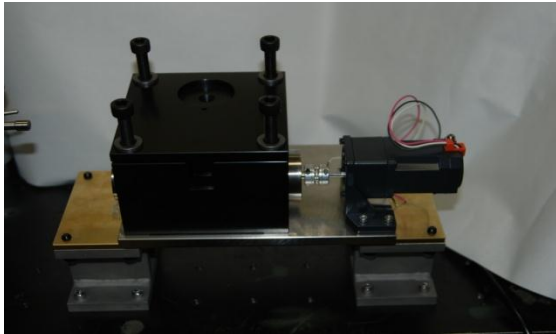
田中宏明, 島田尚一, 井川直哉: 分子動力学シミュレーションによる単結晶シリコン理想表面生成プロセスの予測, 砥粒加工学会誌 Vol.45, No.4 (2001) pp.175-180

1. Chemical elements in slurry forms chemical reacted layer on the wafer surface
2. Two suppositions for slurry particles' function as follows :
  - (1) micro cutting by embedded particles on the wafer surface
  - (2) chemical adhesion by slurry particles

***At first, contact area observation between polishing pad and wafer is attempted.***



|                         |                       |                               |
|-------------------------|-----------------------|-------------------------------|
| microscope              | Nikon                 | Industrial microscope LV150   |
| Halogen lamp            | Nikon                 | LV-KH50PC                     |
| Anti-reflective film    | Nichiyu               | ReaLook 7802UV                |
| High speed video camera | Nack Image Technology | MEMRECAM fx RX-6G<br>2000 fps |



Observation unit

contact glass plate and pad



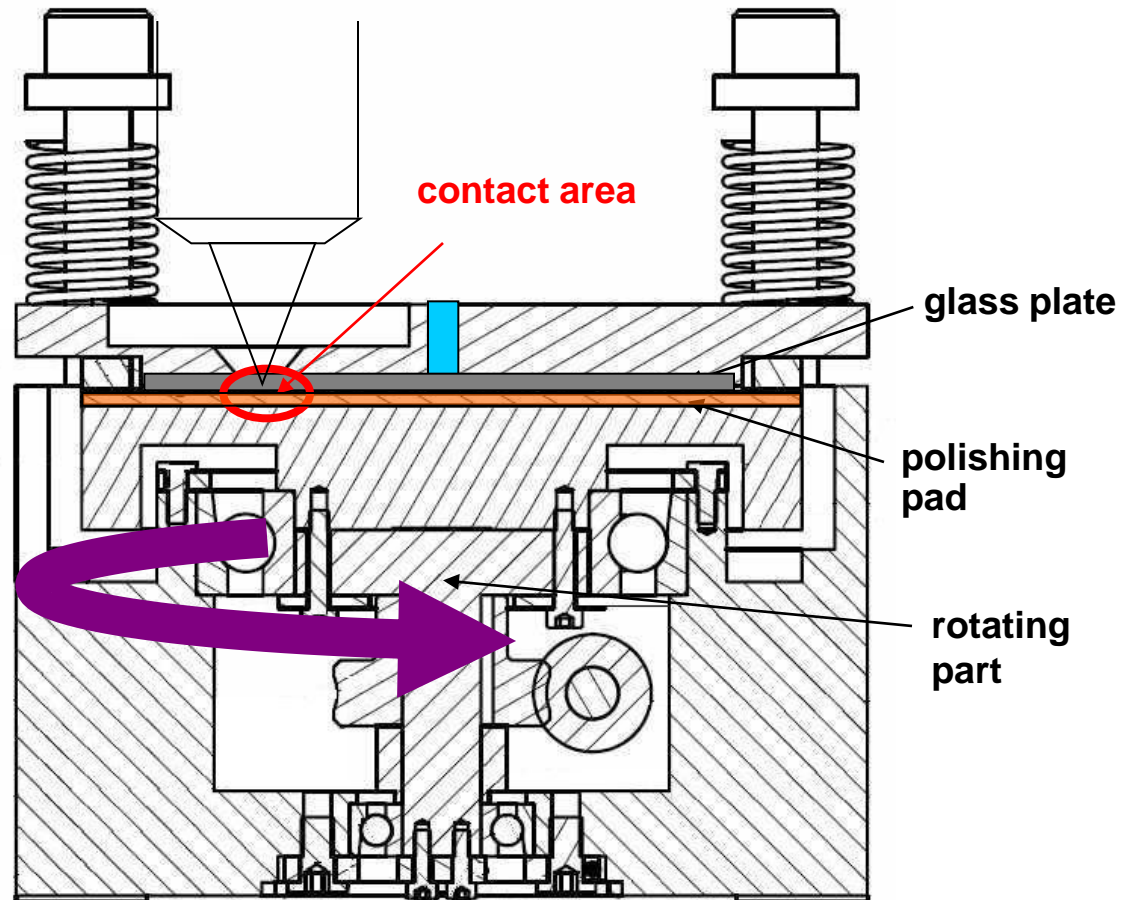
glass plate is fixed  
Pad is rotating



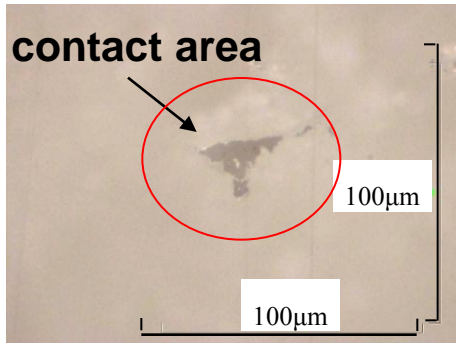
supply D.I. water at center



observe contact area



## dry observation

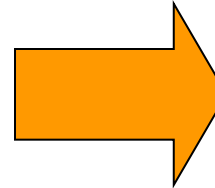
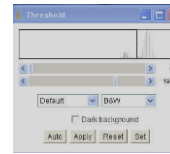


bright area :  
no-contact area

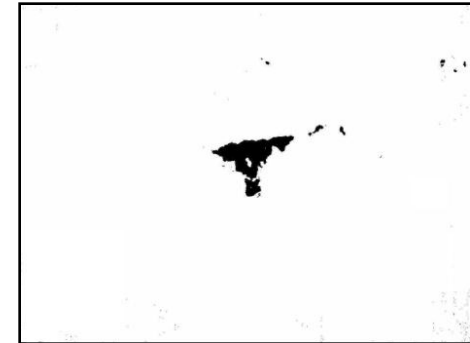
dark area :  
contact area

X 20

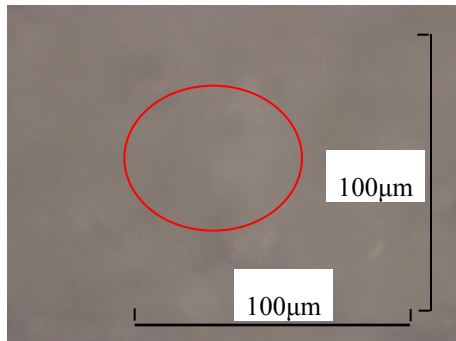
## binarization



## processed image

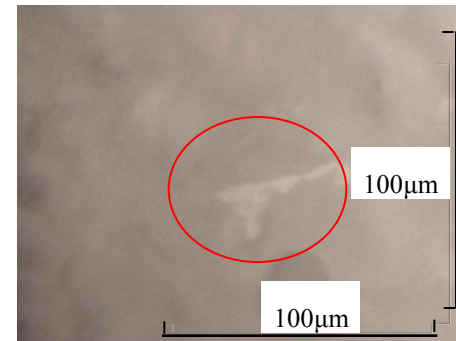


## wet observation



hard  
recognition

## with anti-reflection film



bright area :  
contact area

dark area :  
no-contact area

Contact area was taken as a movie with High speed video camera

[ example ]

conditioner : #100  
rotation : 0.95 min<sup>-1</sup>  
pressure : 3 psi  
playback speed : 0.1 times

bright area : contact area

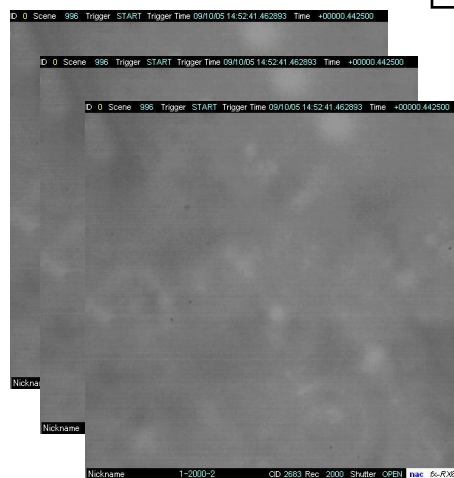
dark area : no-contact area



50μm

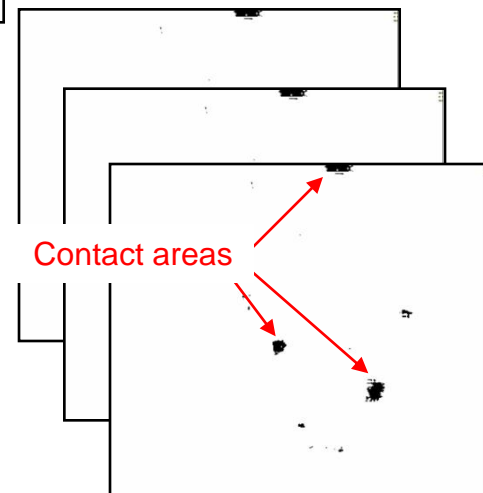
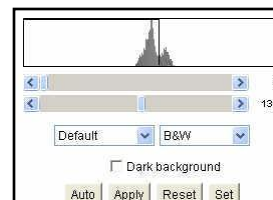


movie



still images

binarization



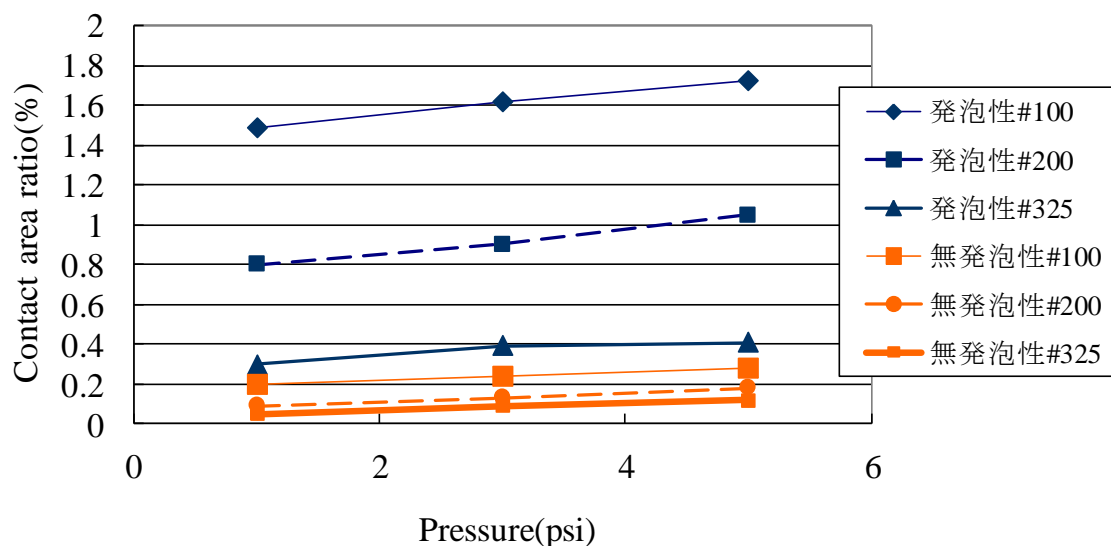
after processing

$$\text{contact area ratio} = \frac{\text{contact area}}{\text{whole area of photograph}}$$



## Calculations for “contact area ratio” with pressure variation

- (1) Observed contact areas with 6 kinds of conditioned polishing pad
- (2) Selected 10 Images at random and calculated “contact area ratio”

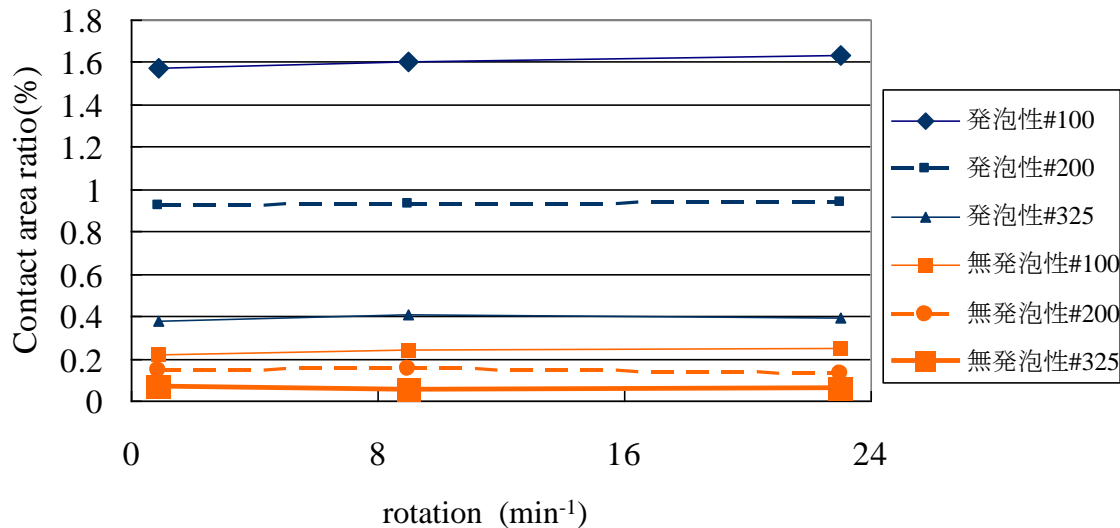


- polishing pad :  
IC1000 , no foaming type
- conditioner :  
#100, #200, #325
- Pressure : 1, 3, 5 psi
- rotation : 9.5 min<sup>-1</sup>

1. Contact area ratio increases with pressure growing
2. Contact area ratio of foaming type polishing pad is larger than that of no-foaming pad

## Calculations for “contact area ratio” with rotation speed variation

- (1) Observed contact areas with 6 kinds of conditioned polishing pad
- (2) Selected 10 images at random and calculated “contact area ratio”

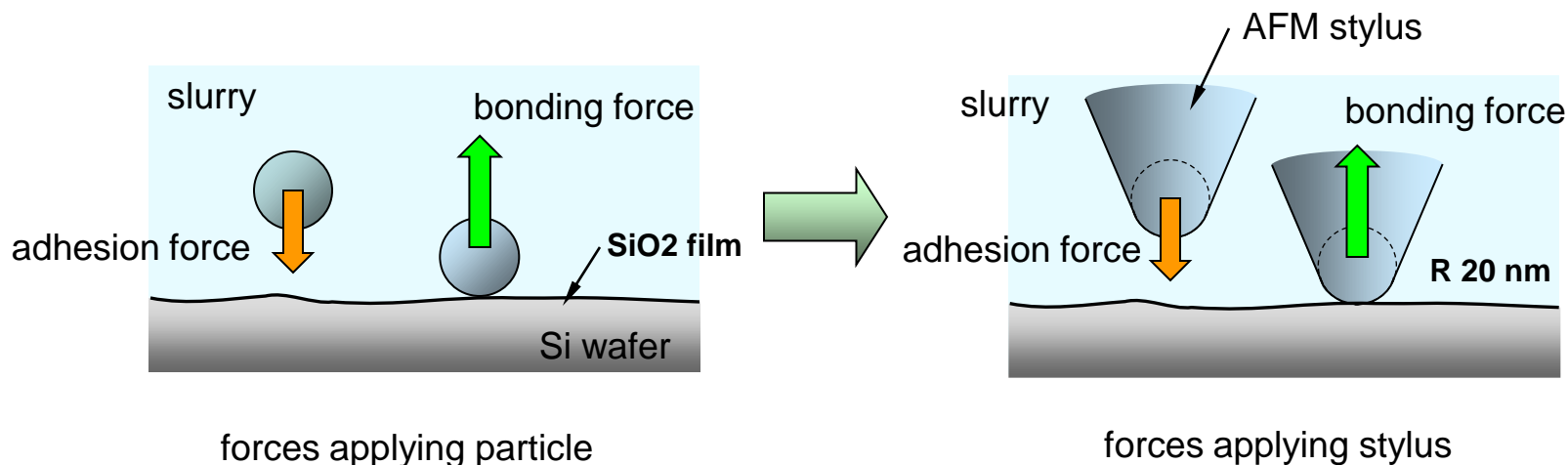


- polishing pad : IC1000 , no foaming type
- conditioner : #100, #200, #325
- Pressure : 3 psi
- rotation : 0.9, 9.5, 23.5 min<sup>-1</sup>

1. Contact area ratio has nothing to do with polishing pad rotation speed
2. Contact area ratio is less than 2 % in every normal conditions

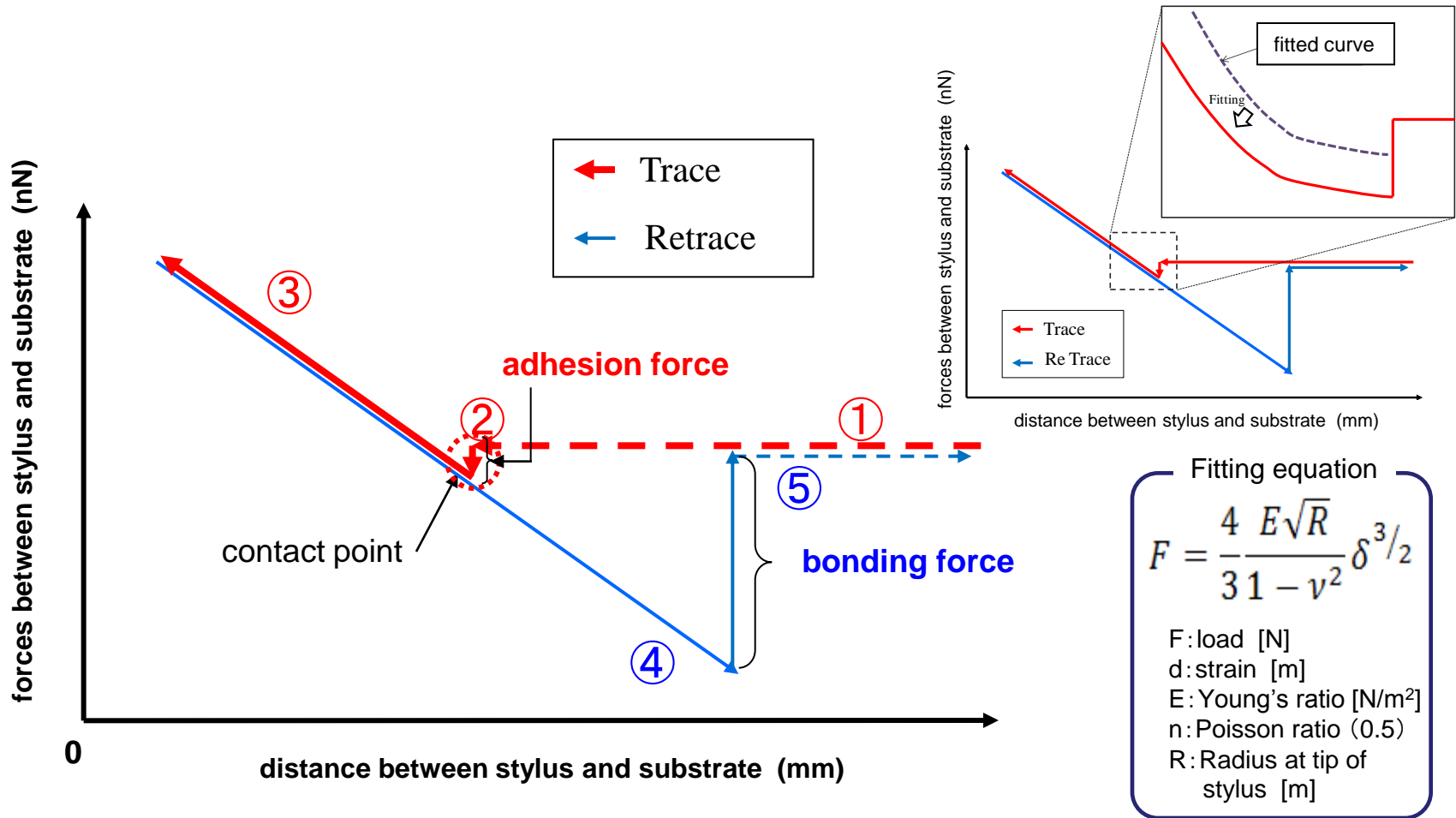
1. Contact area observation between rotating polishing pad and glass plate was attempted.
2. Contact area ratio between polishing pad and wafer is extremely small, less than **2 %**.
3. It is difficult to consider that material removal happens at the contact area with scratching by slurry particles.

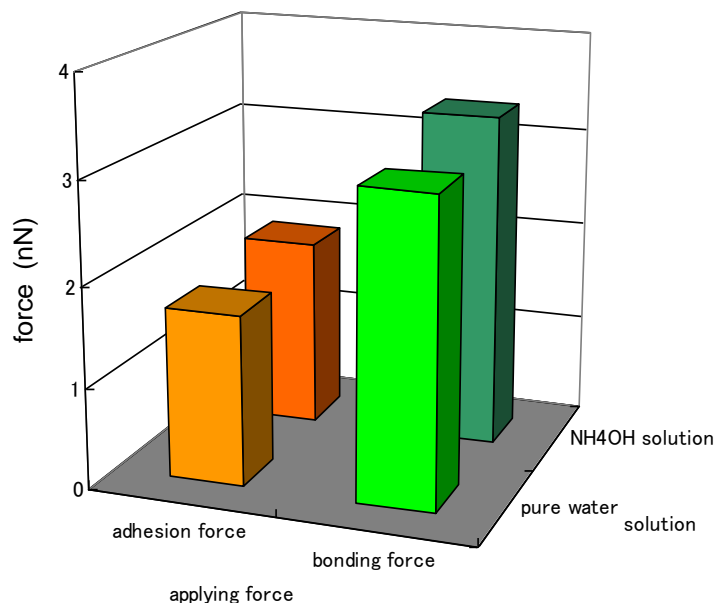
***As a second step, the possibility of particle's adhesion is investigated with AFM.***



MFP-3D (Asylum Research)

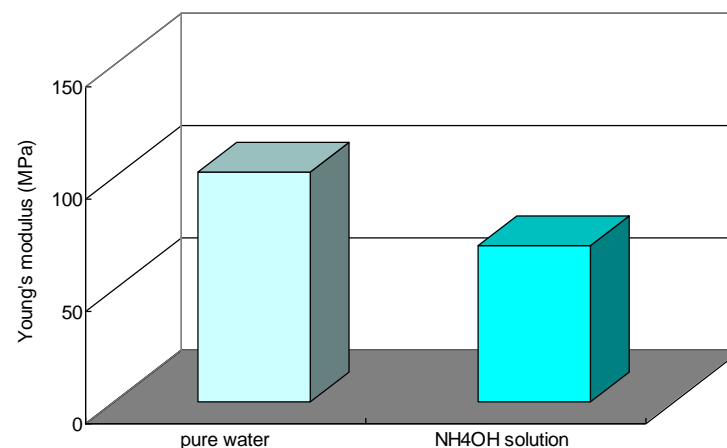
|                  |  |
|------------------|--|
| <b>AFM</b>       | <b>MFP-3D<br/>(Asylum Research)</b>                    |
| <b>stylus</b>    | <b>SiO<sub>2</sub></b>                                 |
| <b>substrate</b> | <b>Si with SiO<sub>2</sub> film</b>                    |
| <b>solution</b>  | <b>H<sub>2</sub>O, H<sub>2</sub>O+NH<sub>4</sub>OH</b> |





Adhesion forces and bonding forces between stylus and substrate

☑ SiO<sub>2</sub> wafer's Young's modulus in air : 50-90 GPa



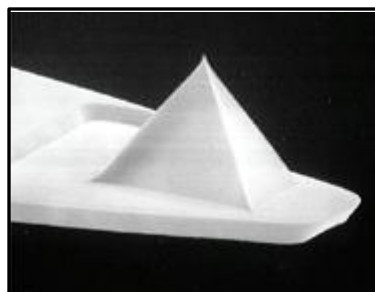
Young's modulus of wafer surface in slurry

Substrate : Si wafer + SiO<sub>2</sub> film  
Stylus : Si + SiO<sub>2</sub> coated

1. Adhesion force and bonding force applies stylus tip
2. Bonding force is about 80% larger than adhesion force , and these are larger in NH<sub>4</sub>OH solution than in water
3. Surface of SiO<sub>2</sub> wafer in NH<sub>4</sub>OH solution becomes much softer than in water

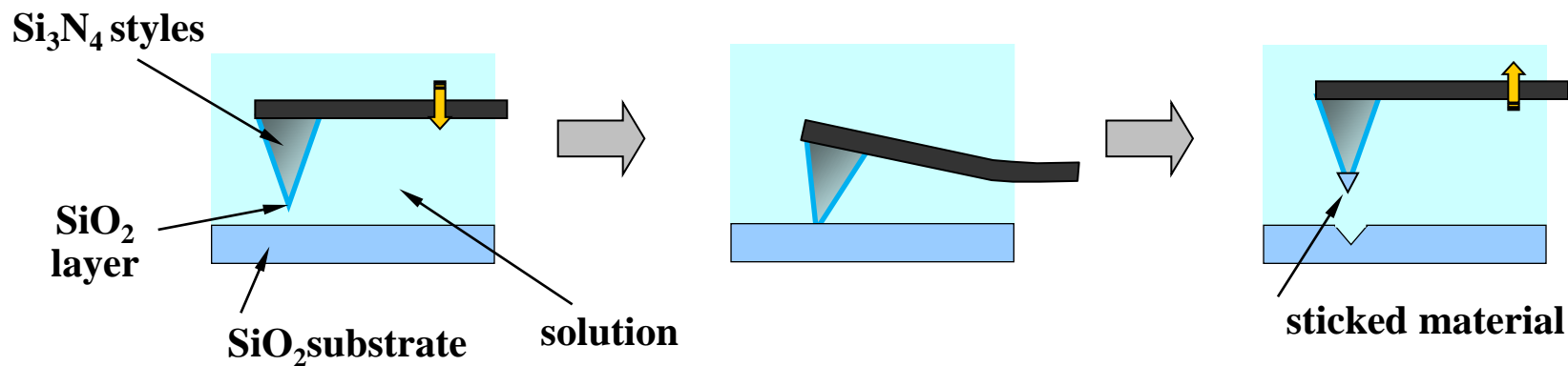


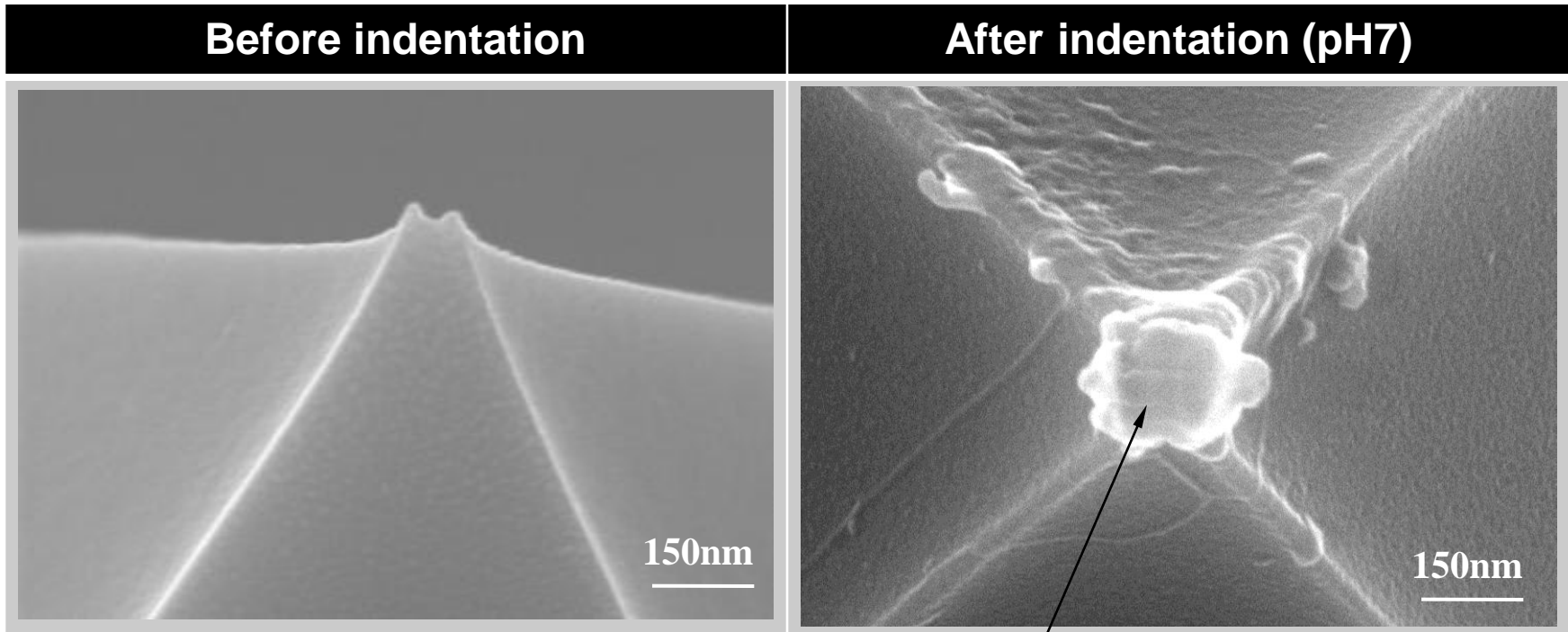
**Nanoscope IIIa**  
**Digital Instruments**



**DNP-S(Veeco)**  
**OMCLTR800PSA(OLYMPUS)**  
**Si<sub>3</sub>N<sub>4</sub> (thermal oxidation on tip)**  
**Radius of tip : 10~40 nm**

| Test piece  | solution   | measurement        |
|---|--|--------------------|
| SiO <sub>2</sub> wafer<br>with thermal<br>oxidation layer | D. I. water<br>pH8.5(KOH,NH <sub>4</sub> OH)<br>pH10(KOH,NH <sub>4</sub> OH) | SEM<br>observation |

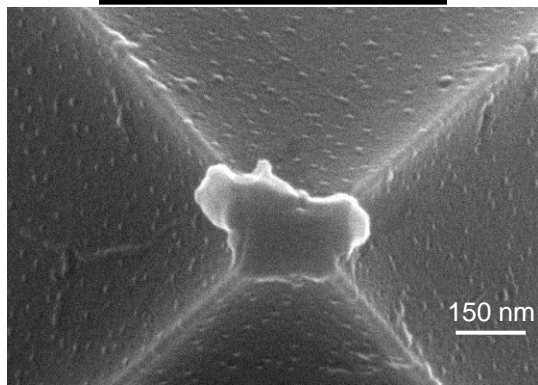




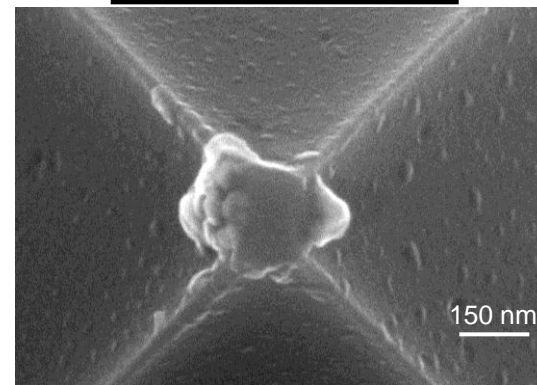
sticking material



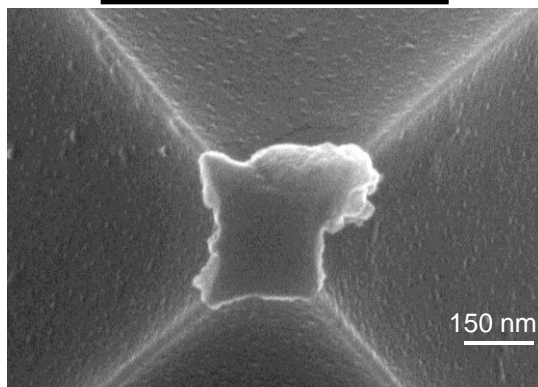
**pH 8.5 / KOH**



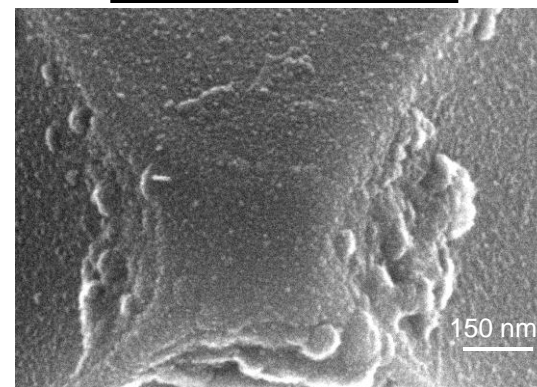
**pH 8.5 / NH<sub>4</sub>OH**

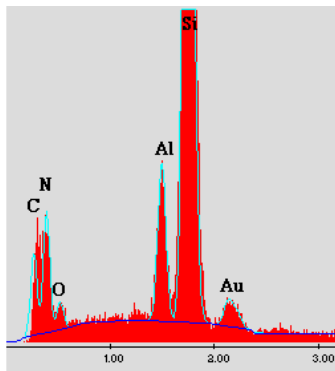


**pH 10 / KOH**

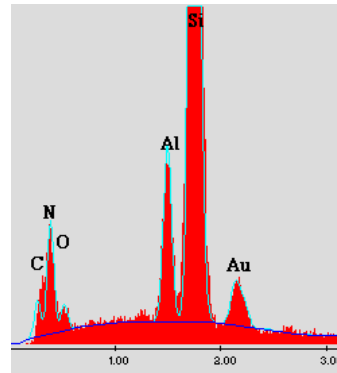


**pH 10 / NH<sub>4</sub>OH**

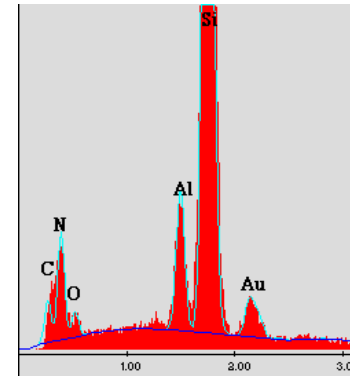




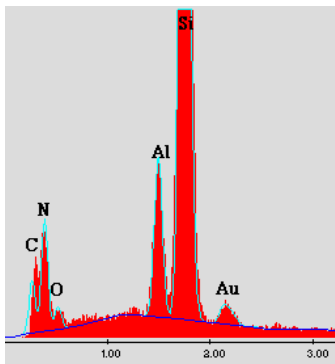
(a) unused



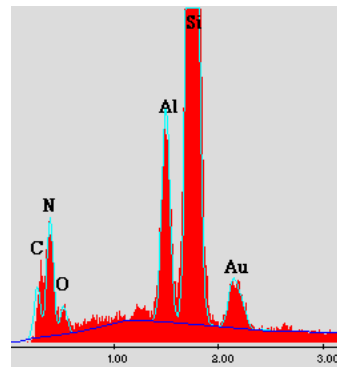
(c) pH8.5 (KOH)



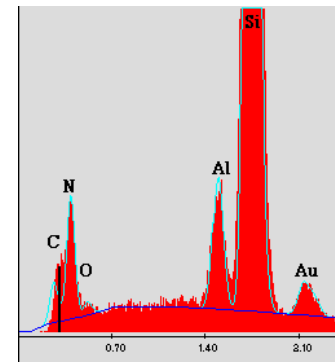
(e) pH10 (KOH)



(b) pH7 (Pure Water)



(d) pH8.5 (NH<sub>4</sub>OH)

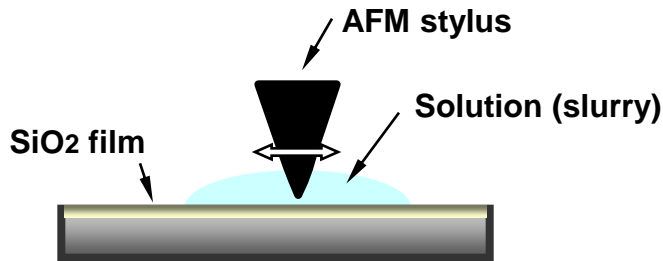


(f) pH10 (NH<sub>4</sub>OH)

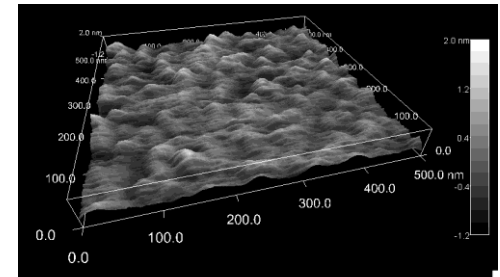
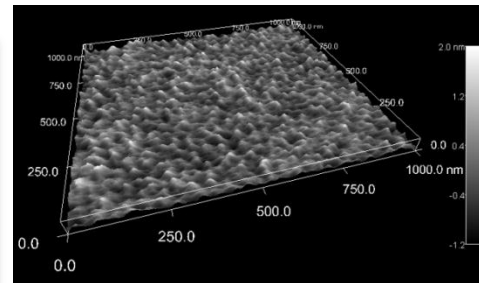
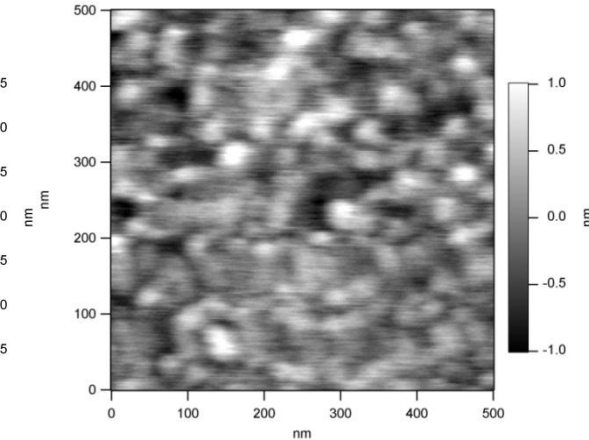
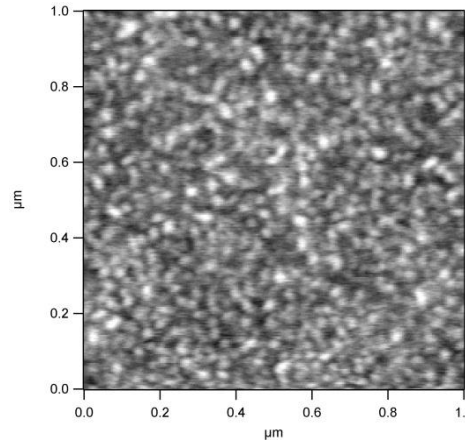
Sticking material is "SiO<sub>x</sub>"

1. Wafer surface [  $\text{SiO}_2$  film ] becomes softer in alkaline solution remarkably  
→ **Soft hydration layer [  $\text{Si(OH)}_4$  ]** is formed on wafer surface
2. Adhesion force applies to the stylus tip  
→ **Fine particles in slurry are drawn to wafer surface**
3. The stylus tip is bonded with the wafer surface after contact on it
4. Sticking material is observed at the tip of stylus after contact wafer surface, and sticking material is considered as **“ $\text{SiO}_x$ ”**

*As a third step, how the mass of particles behave in the space between polishing pad and wafer should be investigated.*



10 min after solution dropped, measured in solution as it is.



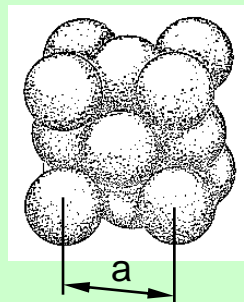
AFM : MFP-3D (Asylum Technology)  
Stylus : Si  
Wafer : SiO<sub>2</sub>  
Solution : **SiO<sub>2</sub> particles** + NH<sub>4</sub>OH (LNA2000)  
Concentration : **12.6 wt%**  
Particle diameter : **approx. 23 nm(#2)**

1. Particles are accumulated on the surface of wafer, and arrayed packed, periodically and uniformly.
2. Distances between two particles are approx. 60 – 80 nm.

## Specifications of slurry

SiO<sub>2</sub> particle :

primary diameter : 14.7 nm,  
secondary diameter : 23.2 nm  
concentration : 12.6 wt%



Lattice constant :  $a$

Proximity distance :  $\frac{\sqrt{2}}{2} a$

Face Centered Cubic structure

## assumption

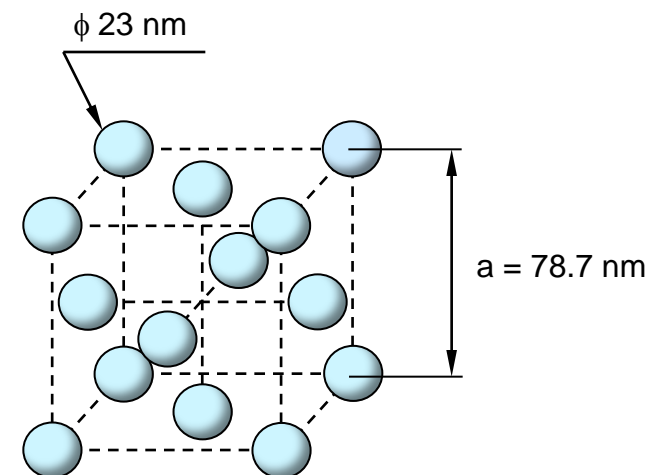
- (1) SiO<sub>2</sub> diameter :  $\phi$  23nm
- (2) Distribution as face centered cubic
- (3) Concentration : 12.6 wt%

**(a) Number of particles in 1cc :  $2.74 \times 10^{18}$**

**(b) distance between particles :**

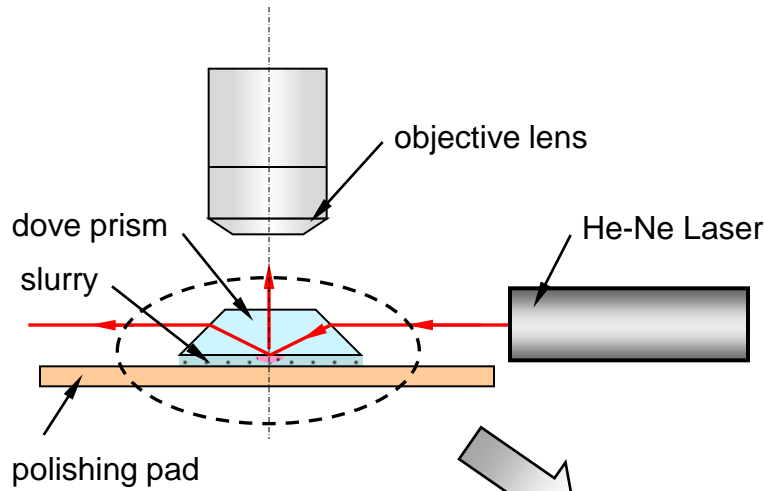
**Lattice constant : 78.7nm**

**Proximity distance : 55.6nm**

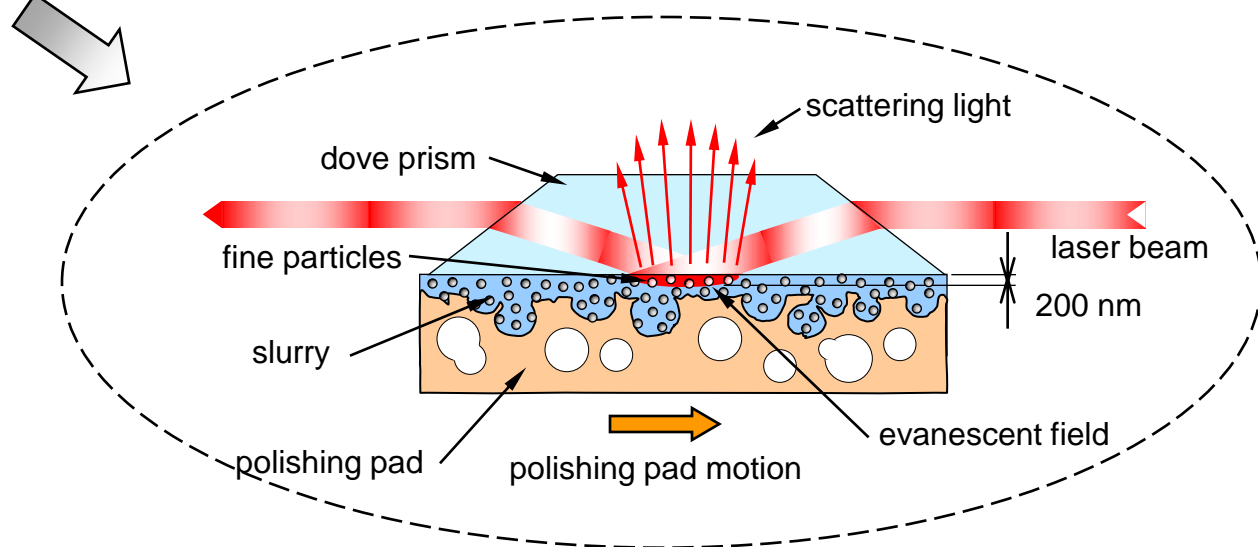


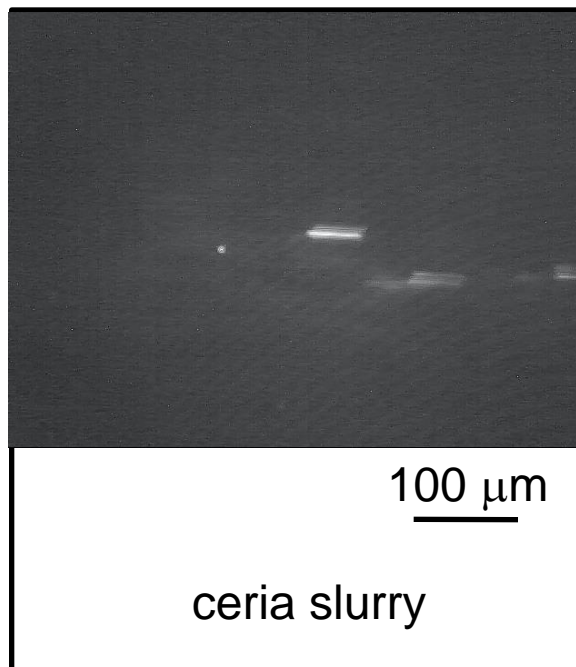
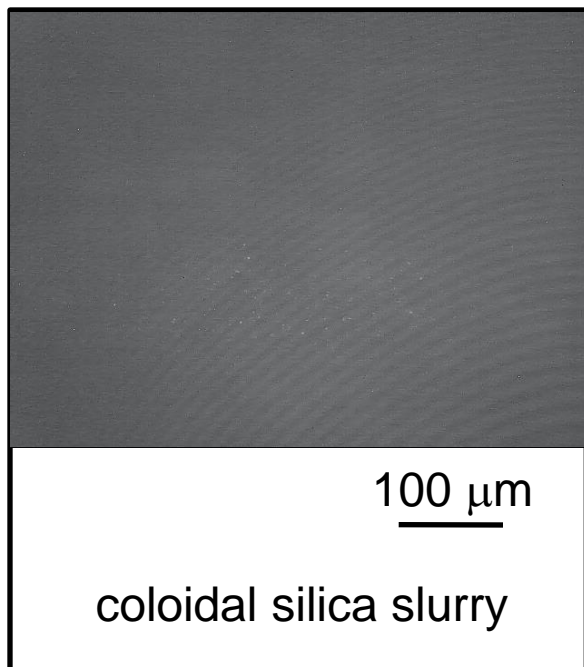
***Fine particles would disperse in slurry as “FCC” structure***

- ☑ Fine particles in slurry are observed with **evanescent light field** in dynamic motion



| slurry             | Colloidal Silica slurry | Ceria slurry |
|--------------------|-------------------------|--------------|
| Particle dia. [nm] | 300                     | 500          |
| Observation area   | 250x330 $\mu\text{m}$   |              |
| camera             | DS-F11 (Nikon)          |              |
| Playback speed     | X 21                    |              |



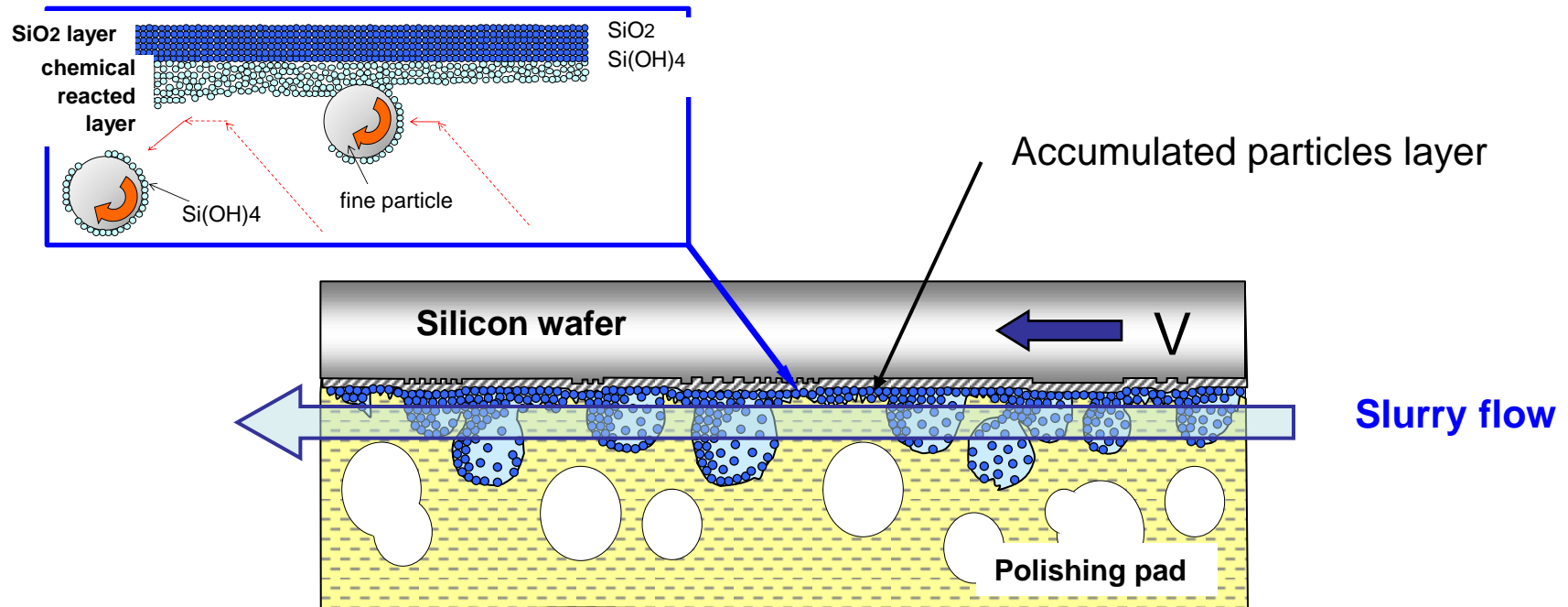


Observation movies (60 fps)

Particles move slowly in the slurry flow on near surface of glass.  
These particles would work as removing materials with adhesion.

## Observation condition

| slurry                | colloidal silica slurry                      | ceria slurry |
|-----------------------|--|--------------|
| particle diameter[nm] | 300  | 500          |
| pressure[kPa]         | 13.8   |              |
| rotation speed[/min]  | 1  |              |
| relative speed[mm/s]  | 2.9  |              |
| polishing pad         | IC1000/Suba400 No-Groove                     |              |
| high speed camera     | VW6000 (KEYENCE)                             |              |
| frame rate[fps]       | 60   |              |
| playback speed        | 4X   |              |
| observation area      | 375 $\mu\text{m}$ $\times$ 500 $\mu\text{m}$ |              |
| laser type            | He-Ne laser (wavelength:632.8nm)             |              |



1. Slurry flows into the thin space between wafer and polishing pad
2. Slurry particles are drawn and stick to the wafer surface
3. Slurry particles contact and roll on the wafer surface, and adhere the atoms from the wafer surface
4. With the motion of the mass of particles, material removal progress continuously



1. Material removal mechanism for SiO<sub>2</sub> CMP was investigated.
2. Contact area between polishing pad and wafer is extremely small, then it is hardly considered that micro cutting by fine particles is carried out.
3. Mechanical property of wafer surface and fine particle's function in slurry were investigated with AFM.
4. Hydration layer is formed on the wafer surface, and bonding strength become lower. Fine particles in slurry flows and contact at the wafer surface, and remove materials from the surface with adhesion.
5. In slurry, a great mass of particles dispersed regularly, and these particles remove material continuously.

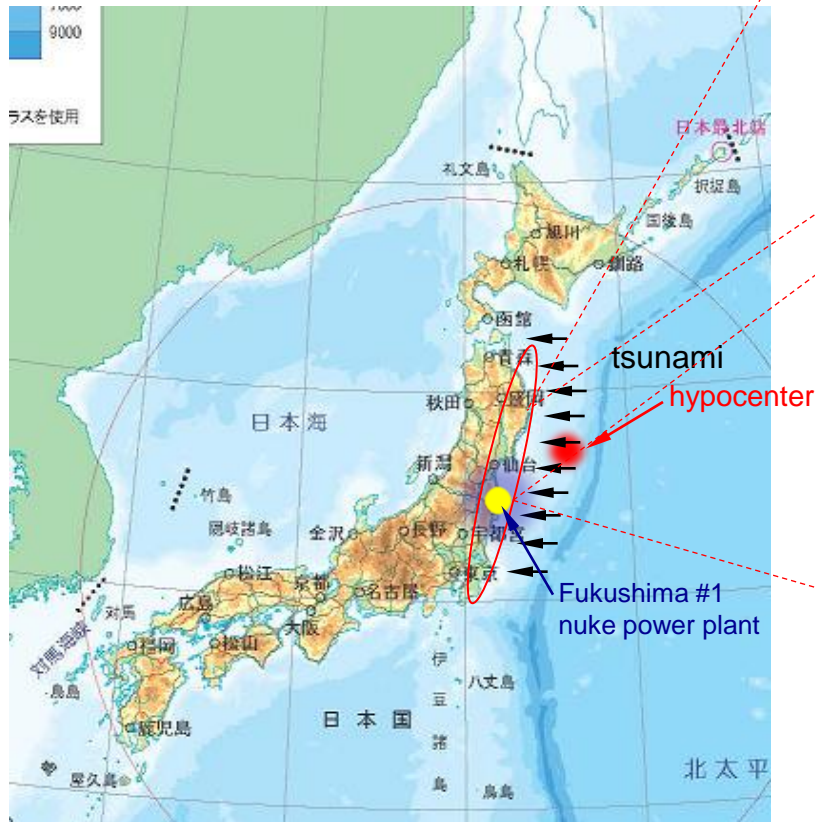
# March 11, 2011 Big Earthquake & Tsunami attacked in Eastern Japan

May 11, 2011



## Three damages hit Japan

1. Earthquake
2. Tsunami
3. Nuclear power plant accidents



Fukushima #1 nuclear power plant

**The dead : 14,949**  
**The missing : 9,880**  
as of May 10, 2011

# Thank you for your warmhearted support to JAPAN! May 11, 2011



*Thank you for your attention !*

Keiichi Kimura

Kyushu Institute of Technology

JAPAN

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# Academic Programs

## Tobata Campus



**School of Engineering**  
Since 1909

Mechanical and Control Engineering

Civil and Architectural Engineering

Electrical Engineering and Electronics

Applied Chemistry

Materials Science and Engineering

Integrated System Engineering

# ~Bachelor~

## Izuka Campus



**School of Computer  
Science and Systems  
Engineering**

Since 1986

Artificial Intelligence

Computer Science and Electronics

Systems Design and Informatics

Mechanical Information Science and Technology

Bioscience and Bioinformatics

# Academic Programs ~M.S. and Ph.D~

## Tobata Campus



### Graduate School of Engineering

Mechanical and Control Engineering

Civil and Architectural Engineering

Electrical Engineering and Electronics

Materials Science

Applied Science for Integrated System Engineering

## Iizuka Campus



### Graduate School of Computer Science and Systems Engineering

Information Science

Information Systems

Creative Informatics

## Wakamatsu Campus



### Graduate School of Life Science and Systems Engineering

Since 2000

Biological Functions and Engineering

Brain Science and Engineering

# Number of Students

The total number of students is 6,063  
(4,363 undergraduate and 1,700 graduates).

| Campus    | Schools (Bachelor and M.S./Ph.D)                            | Number of Students |
|-----------|---|--------------------|
| Tobata    | School of Engineering                                       | 2,446              |
|           | Graduate School of Engineering                              | 779                |
| Iizuka    | School of Computer Science and Systems Engineering          | 1,917              |
|           | Graduate School of Computer Science and Systems Engineering | 539                |
| Wakamatsu | Graduate School of Life Science and Systems Engineering     | 382                |

**Total**                      **6,063**



# Number of Academic Staff

The total number of Academic Staff is 370.

| Campus    | Classification  | Professors | Associate Professors | Lecturers | Assistant Professors | Number of Faculties |
|-----------|---|------------|----------------------|-----------|----------------------|---------------------|
| Tobata    | Faculty of Engineering                                  | 63         | 63                   | 1         | 37                   | 164                 |
| Iizuka    | Faculty of Computer Science and Systems Engineering     | 46         | 50                   | 2         | 28                   | 126                 |
| Wakamatsu | Graduate School of Lice Science and Systems Engineering | 22         | 13                   | 1         | 9                    | 45                  |
|           | Other*  | 14         | 12                   | 1         | 8                    | 35                  |

**Total 370**

\*Other:

- Center for Student Health
- Information Science Center
- Center for Microelectronic Systems
- Collaboration Center
- Center for Instrumental Analysis

- Network Design Research Center
- Advanced Mold and Die Technology Center
- Eco-Town Collaborative R&D Center for the Environment and Recycling
- Science Education Center