



SPCC 2018

**Hanyang University NEMPL**

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# Acidic Cleaning Solutions for Post InGaAs CMP Cleaning

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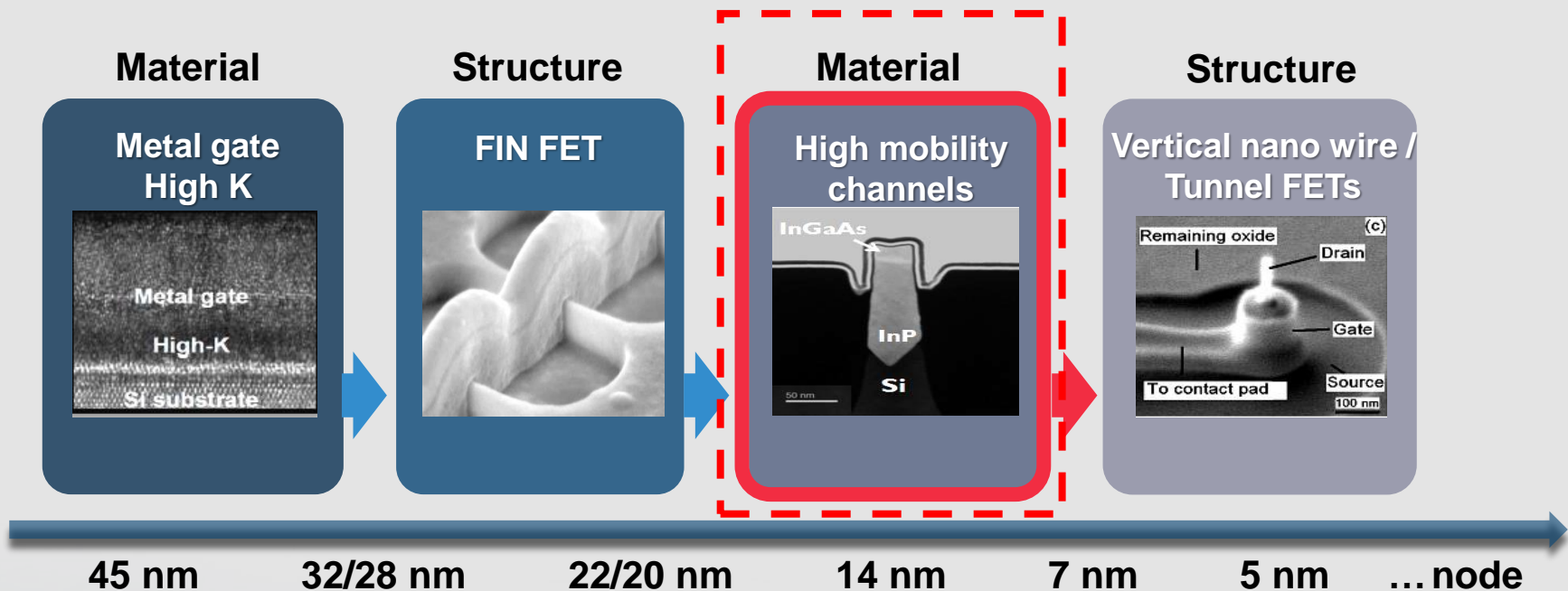
## 05 Summary

# High mobility semiconductor for Logic Device

## Technology development trend of semiconductor industry

- ✓ Demand of high speed
- ✓ Decreasing the power consumption
- ✓ Shrinking device size (high density of integration)

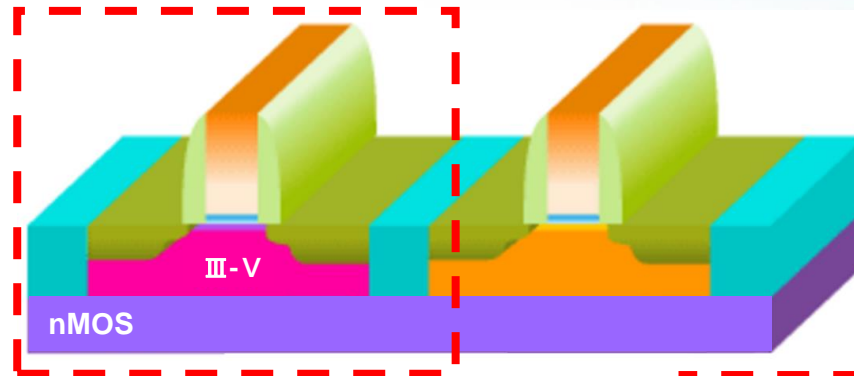
## Logic Devices Roadmap



**Recently new material is required for high mobility channels**

# Properties of semiconductor materials

## CMOS Structure



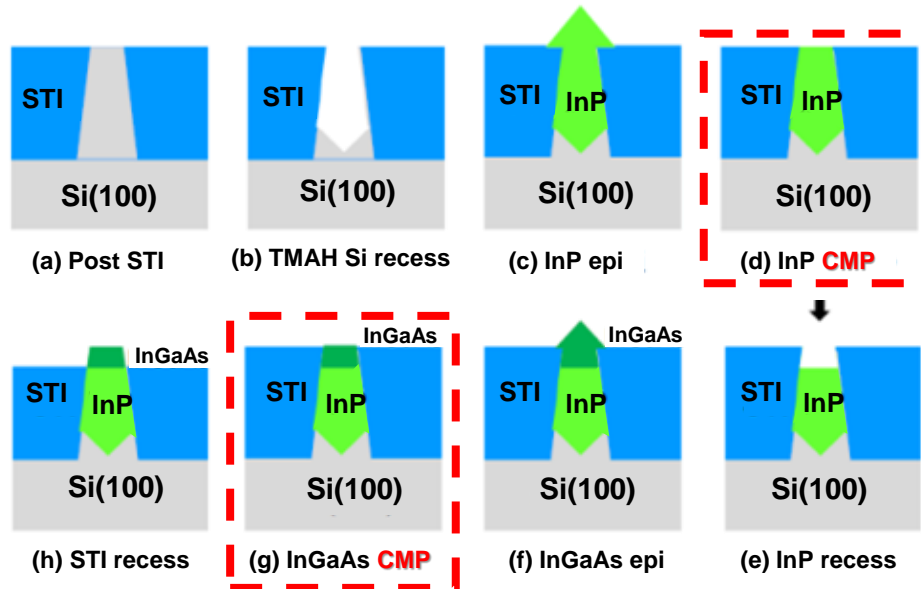
Material	Si	III / V				
		GaAs	InP	InGaAs	InAs	InSb
Electron mobility (cm <sup>2</sup> /Vs)	1,600	9200	5400	<b>11,200</b>	40,000	77000
Hole mobility (cm <sup>2</sup> /Vs)	430	400	200	<b>300</b>	500	850
Lattice constant (Å)	5.43	5.65	5.86	<b>5.87</b>	6.06	6.48
Band gap (eV)	1.11	1.42	1.34	<b>0.74</b>	0.36	0.17

- **Combined elements from group III and V** shows higher electron mobility and it is considered as an **alternative to Si as a channel material**, specifically for nMOS.
- Considering the lattice mismatches and band gap, **InGaAs** may consider as a promising material for nMOS.

<http://www.yokoyama-gnc.jp/english/research/cmos.html>

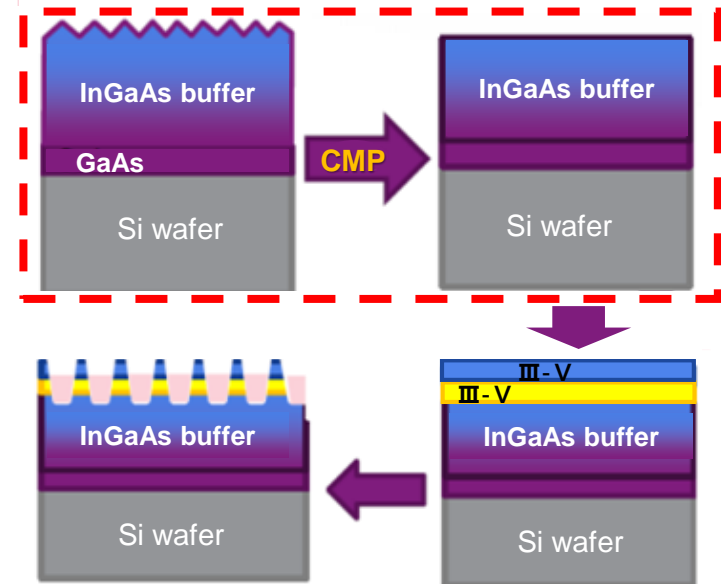
# CMP process for III-V Fins

## ❖ STI first



- ✓ ART (Aspect-Ratio Trapping) : Trapped defects in the III-V layer
- ✓ **CMP is needed to reduce the overburden post epitaxial growth**

## ❖ STI last



- ✓ SRB (Strain Relaxed Buffer): Reduced lattice mismatch with Si
- ✓ **CMP is needed before epi growth of new layer**

In both approaches, InGaAs requires **at least 1 CMP step** using **silica slurry**

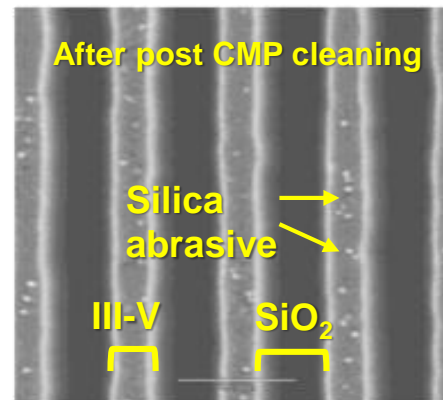
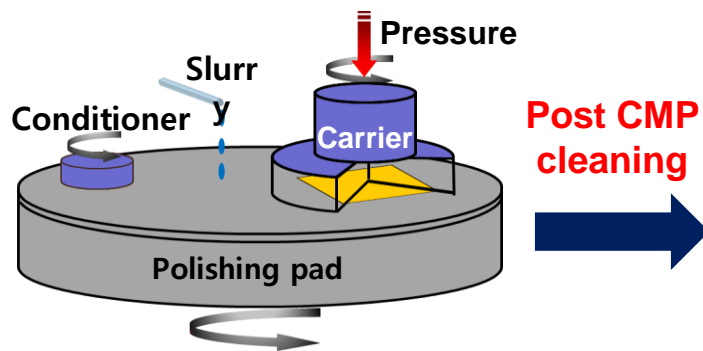


# Motivation

- After CMP process, **post InGaAs CMP cleaning** process was performed using megasonic, brush and **diluted ammonia solutions** to remove slurry contaminants.

## ❖ Issues of post InGaAs CMP cleaning

- The **conventional post InGaAs CMP cleaning process is not effective** for removing nano silica slurry particles from InGaAs surface.



- **Development of new cleaning chemical is required for post InGaAs CMP process**
  - ✓ High cleaning efficiency
  - ✓ Minimal material loss with less oxidized surface

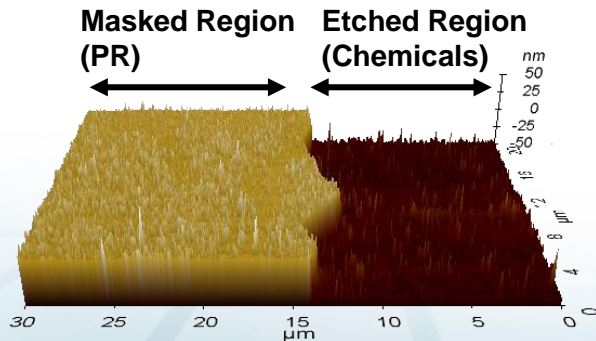
# Experiments for **etch rate** and **oxidation**

## ❖ Materials & Experimental condition

- Substrate : Polished InGaAs ( $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ ; ~75nm thickness)
- Cleaning Chemicals: **HCl** (0.001 M, 0.005 M, 0.01 M, 0.05 M)  
**H<sub>2</sub>O<sub>2</sub>** (0.001 M, 0.005 M, 0.01 M)

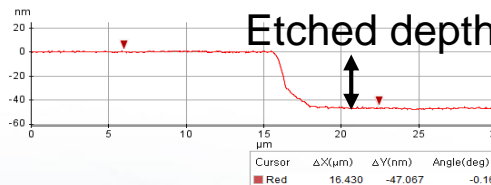
### Etch Rate (Material Loss)

- Photoresist (PR) line patterns of 110  $\mu\text{m}$ 
  - Selective Etching
- AFM (XE 100, Park System, Korea)
  - Etched step height



### Composition Analysis (Oxidation)

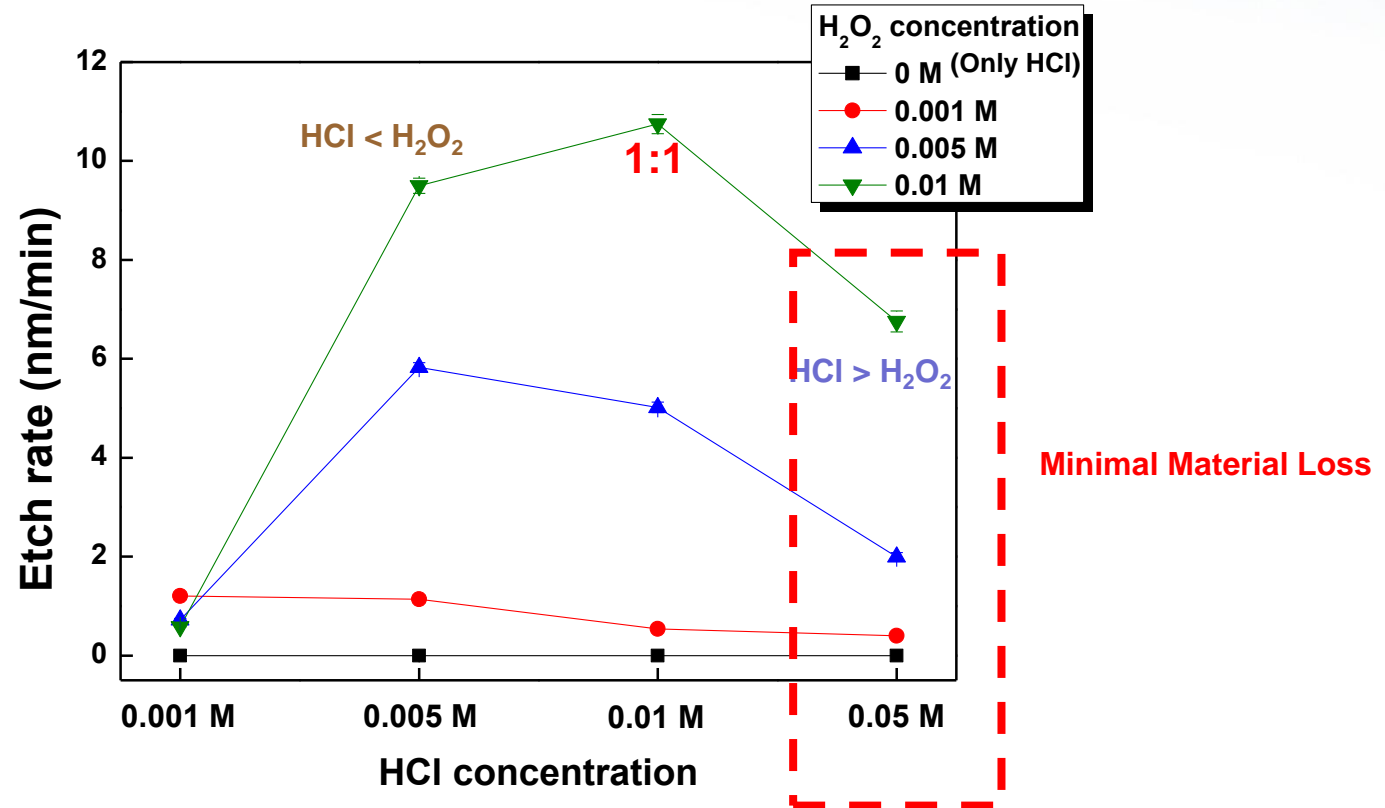
- XPS (Sigma Probe, Thermo, UK)
  - Oxide percentage



$$\text{Etch rate } \left( \frac{\text{\AA}}{\text{min}} \right) = \frac{\text{Etched step height}}{\text{Etching time}}$$

# Etch rate analysis

## ❖ Etch rate vs. various concentration of chemical



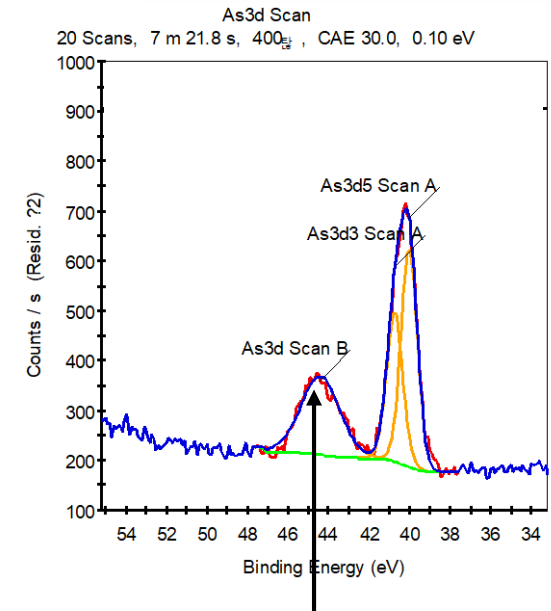
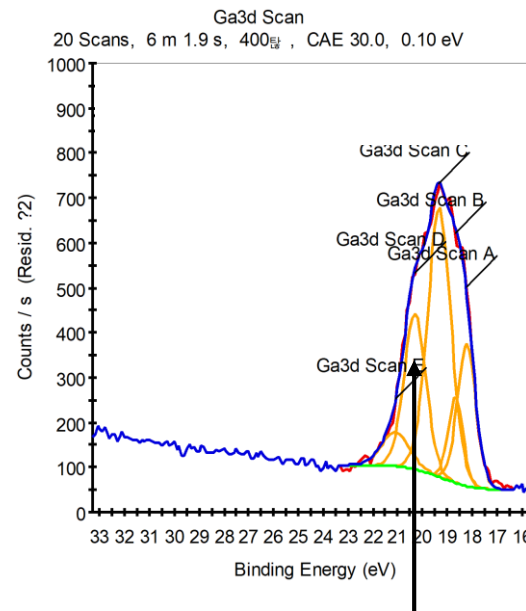
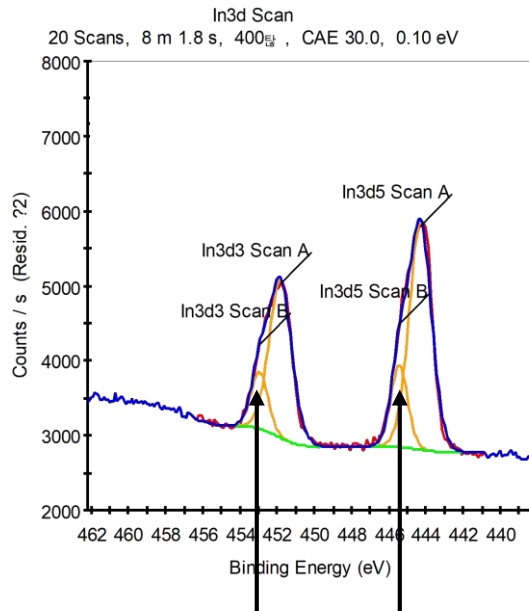
- Only HCl doesn't etch the InGaAs surface without H<sub>2</sub>O<sub>2</sub>
- Highest etch rate was achieved at **HCl : H<sub>2</sub>O<sub>2</sub> = 1 : 1**
- **Lower concentration of HCl than H<sub>2</sub>O<sub>2</sub> (ER ↑)**
- **Higher concentration of HCl than H<sub>2</sub>O<sub>2</sub> (ER ↓)**



# XPS analysis for oxide content

❖ XPS analysis was carried out for analyzing the oxide percentage of InGaAs

✓ Ref. (As-received wafer)



Chemical  
state

$\text{In}_2\text{O}_3$

$\text{Ga}_2\text{O}_3$

$\text{As}_2\text{O}_3$

O Atomic %

21.28 %

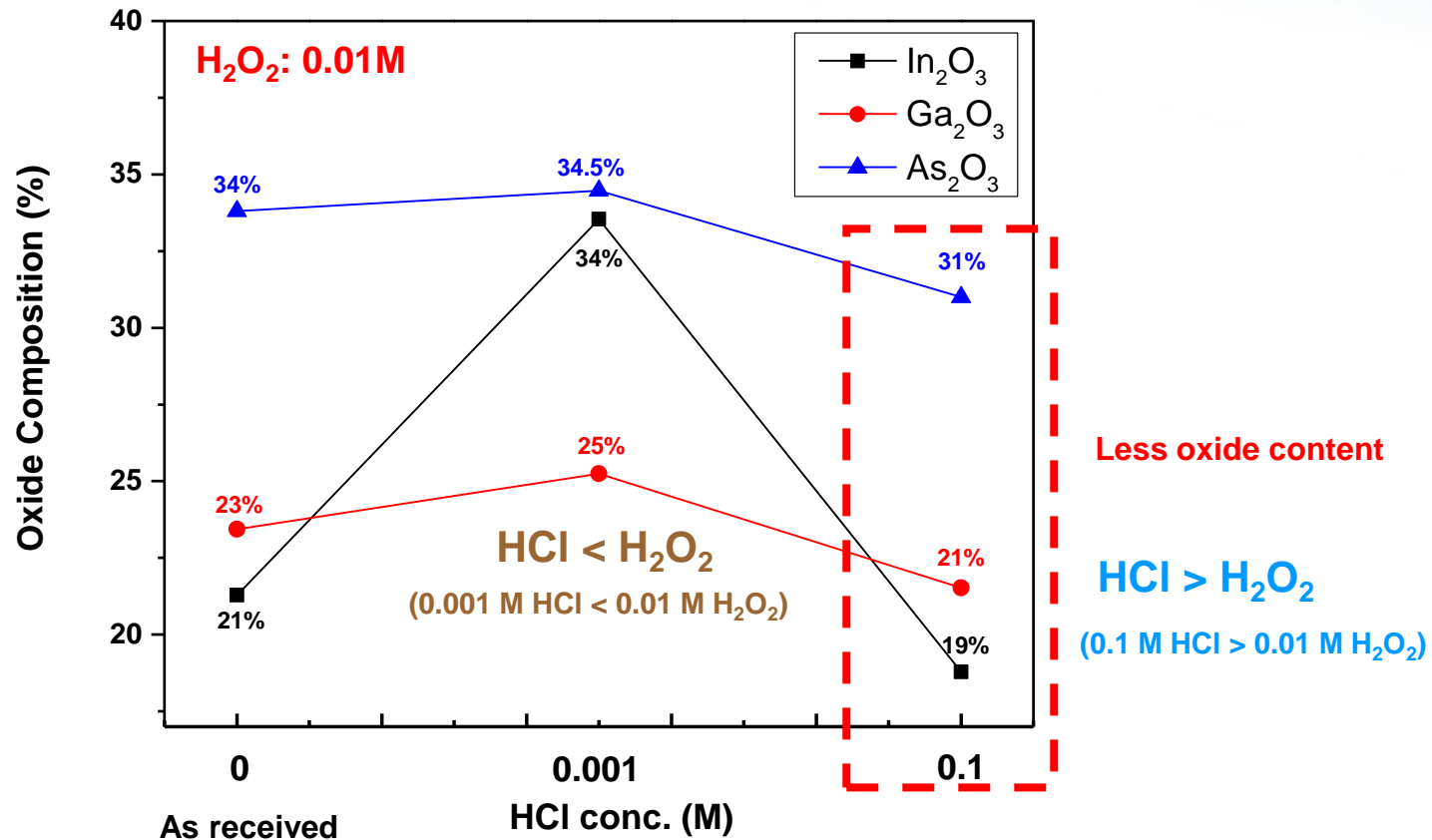
23.43 %

33.8 %

➤ XPS was used to evaluate the cleaning chemical effect on InGaAs oxidation

# XPS analysis for **oxide content**

## ❖ Oxide content vs. HCl Conc.

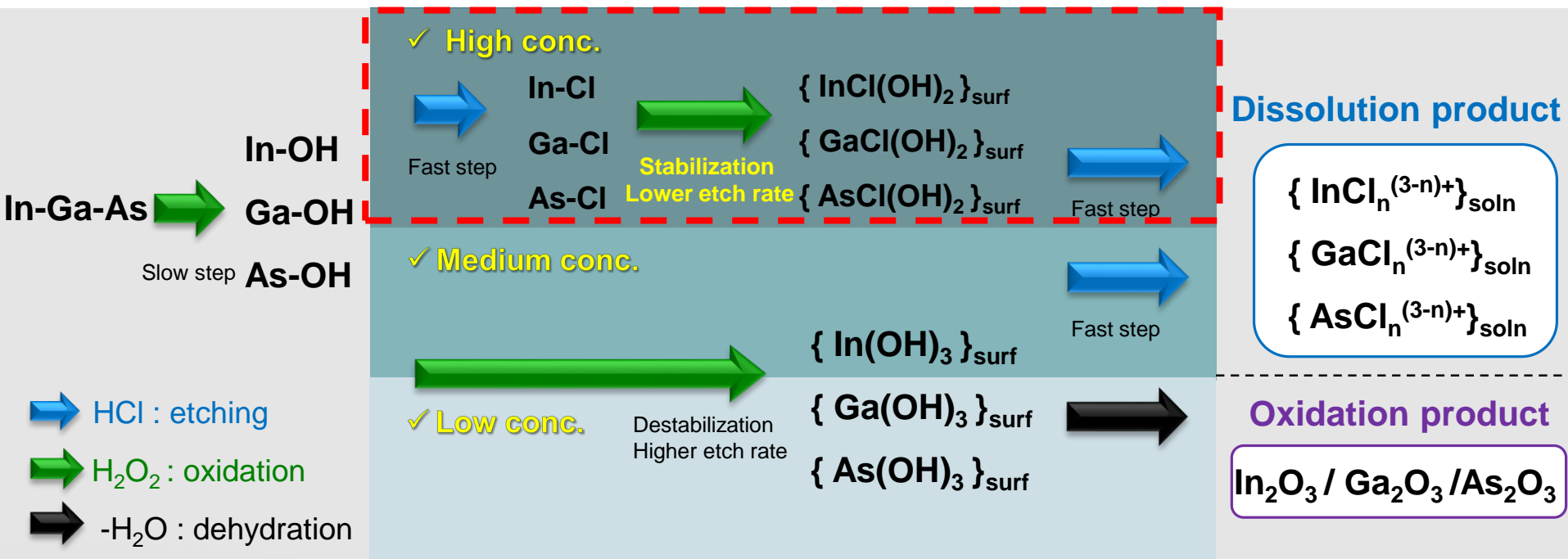


- Lower conc. of HCl increases the oxide composition
- Higher conc. of HCl decreases the oxide composition
- High conc. of HCl than H<sub>2</sub>O<sub>2</sub> can minimize the oxide layer formation

# Etching mechanism

## ❖ Etching mechanism according to the HCl concentration

- ✓ High HCl concentration: oxidation rate  $\leq$  dissolution rate (**ER** ↓) / (**Oxidation** ↓)
- ✓ Low HCl concentration : oxidation rate  $\geq$  dissolution rate (**ER** ↑) / (**Oxidation** ↑)

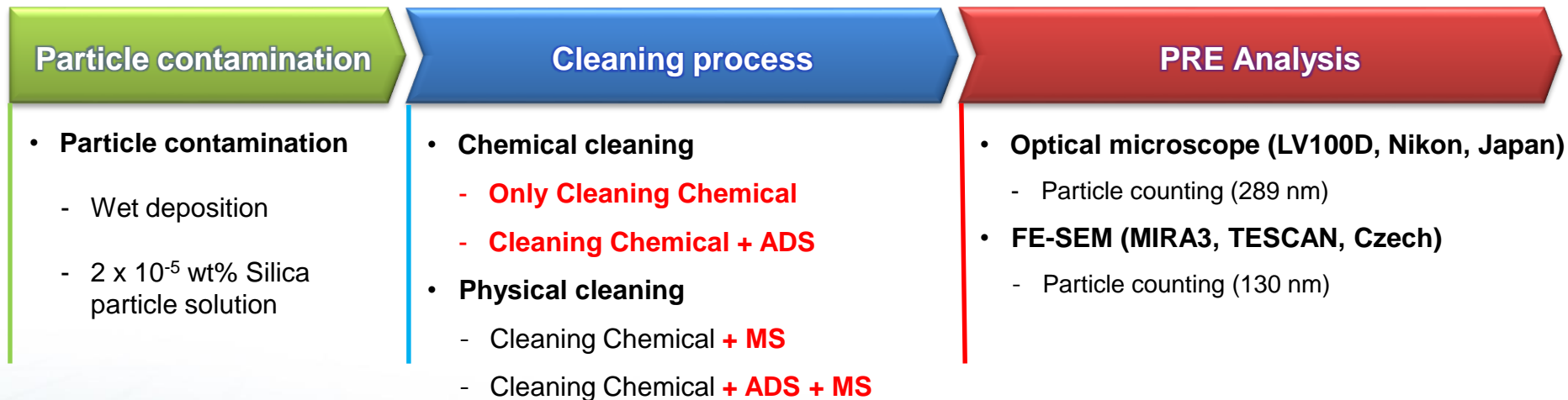


➤ Based on the material loss and surface oxidation results, **higher conc. of HCl (0.05 M)** and **lower conc. of  $\text{H}_2\text{O}_2$  (0.001M)** is considered as a optimized cleaning solution

# Experiments for the evaluation of PRE

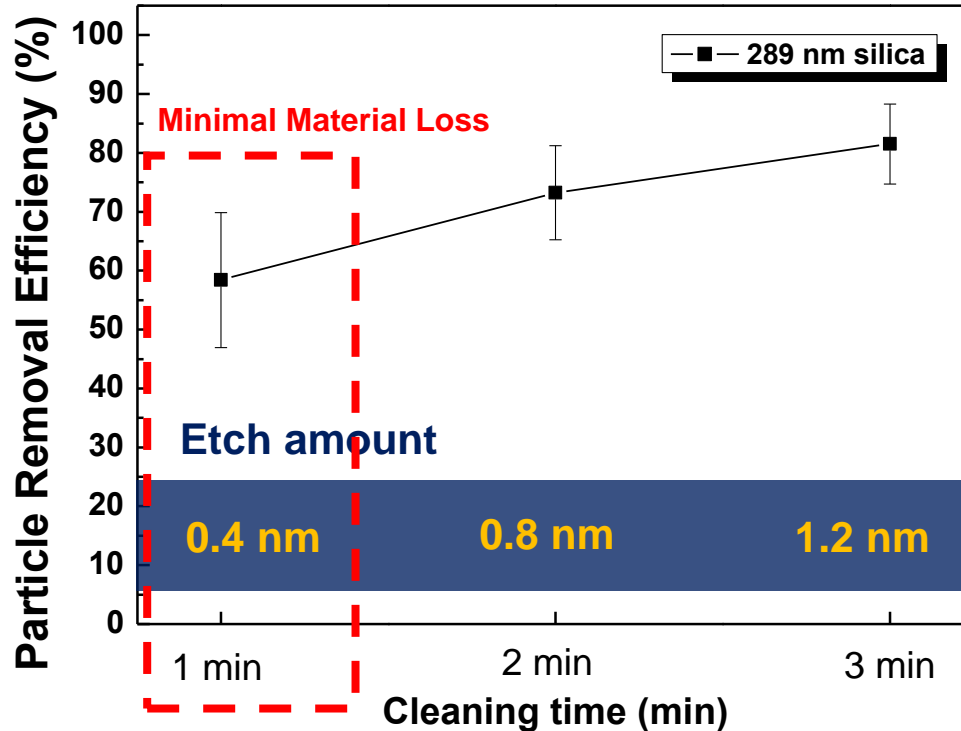
## ❖ Materials & Experimental condition

- Substrate : InGaAs ( $\text{In}_{0.47}\text{Ga}_{0.53}\text{As}$ )
- Particle : Silica (130 nm, 289 nm)
- Optimized Conc. of cleaning solution: **HCl (0.05M) +  $\text{H}_2\text{O}_2$  (0.001M)**
- **Surfactant : ADS** (Ammonium Dodecyl Sulfate) (0 – 0.5mM)
- **Megasonic: batch type, 1MHz, 600W**



# PRE vs. Etch amount

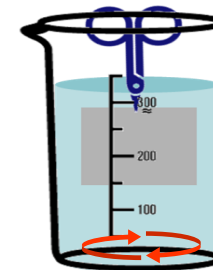
## ❖ PRE and Etch amount vs. Cleaning Time



## ❖ Cleaning condition

- Cleaning solution :  
HCl : 0.05M, H<sub>2</sub>O<sub>2</sub> : 0.001M  
pH : 1.36

- Dipping process



- Dipping time

- 1 min
- 2 min
- 3 min

- Cleaning time  $\propto$  Etch amount
- PRE is increased with increase in the etch amount
- **Modification of zeta-potential** is required to increase the **PRE with minimal material loss.**

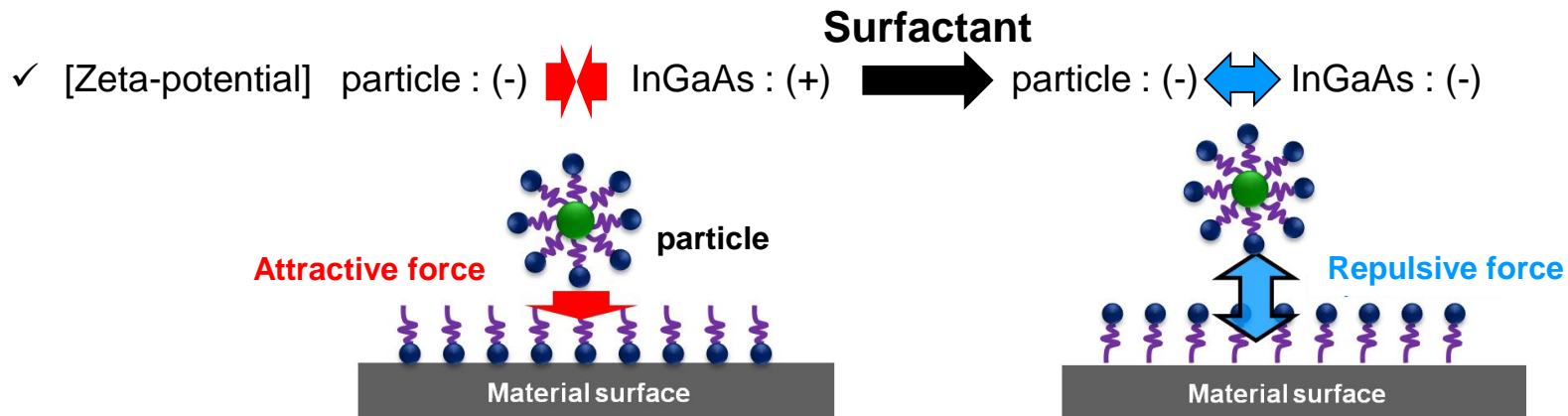
✓ [Zeta-potential] particle : (-) , InGaAs : (+) ➡ particle : (-) , InGaAs : (-)



# Mechanism of surfactant cleaning

## ❖ Modification of electrostatic force using surfactant

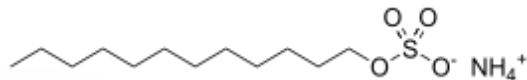
- Introduction of **strong electrostatic repulsion** between particle and substrate can enhance the PRE during the post CMP cleaning process, which was achieved by using surfactant



## ❖ Ammonium Dodecyl Sulfate (ADS)

- ADS is one of an anionic surfactant widely used for cleaning process

< ADS Properties >



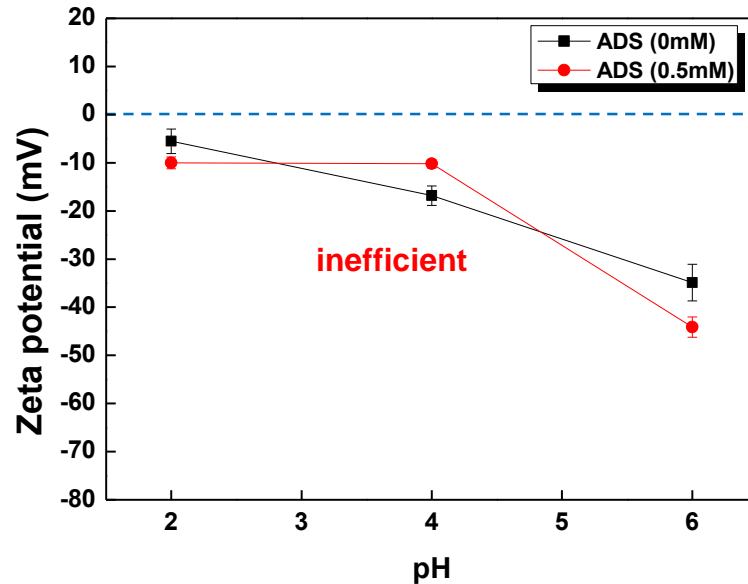
Chemical Formula :  $\text{C}_{12}\text{H}_{29}\text{NO}_4$

CMC value: 6.6 mM

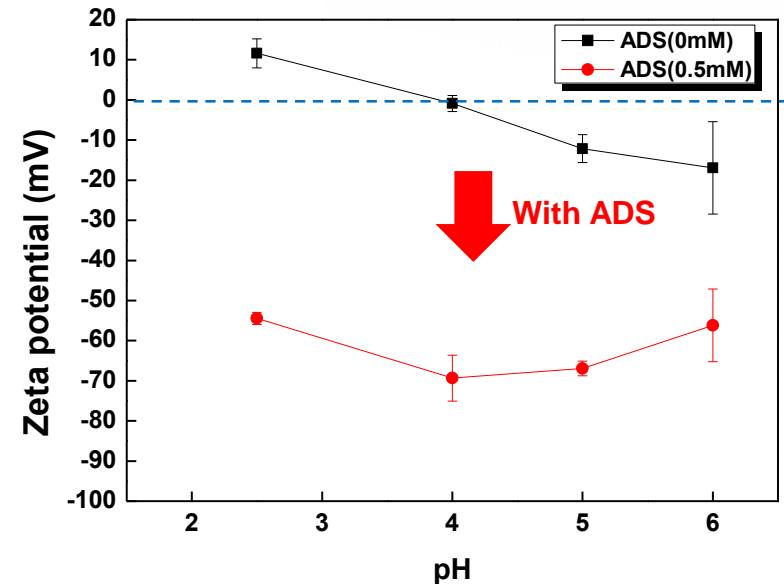
R. Vos, M. Lux et al., Journal of The Electrochemical Society, 148 (12) G683-G691 (2001)

# Zeta potential Measurement

## ❖ Particle zeta-potential (silica)<sup>1</sup>



## ❖ Surface zeta-potential (InGaAs)<sup>2</sup>



### ▪ Analysis equipment

ELS-Z (Electrophoretic Light Scattering Spectrophotometer) Otsuka, Japan <sup>1</sup>  
 SurPASS Electrokinetic Analyzer Anton Paar, Austria <sup>2</sup>

- Zeta potential of InGaAs surface changed significantly after addition of ADS

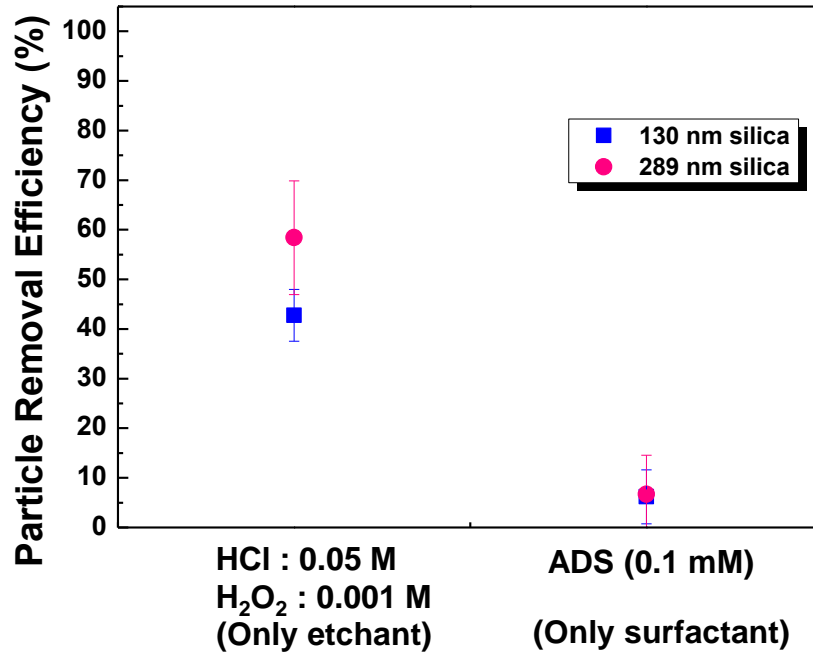
✓ (+) Z.P. ➡ (-) Z.P. in acidic region

- Addition of ADS introduces strong repulsive force between silica particle and InGaAs substrate

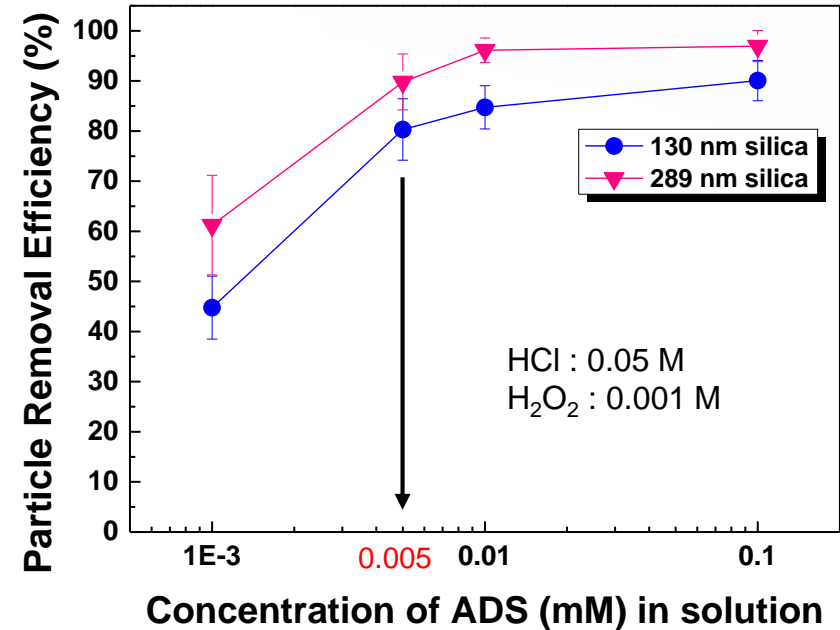
✓ **Silica particles can be easily removed from the InGaAs surface**

# PRE vs. ADS conc.

## ❖ (HCl + H<sub>2</sub>O<sub>2</sub>) vs. ADS

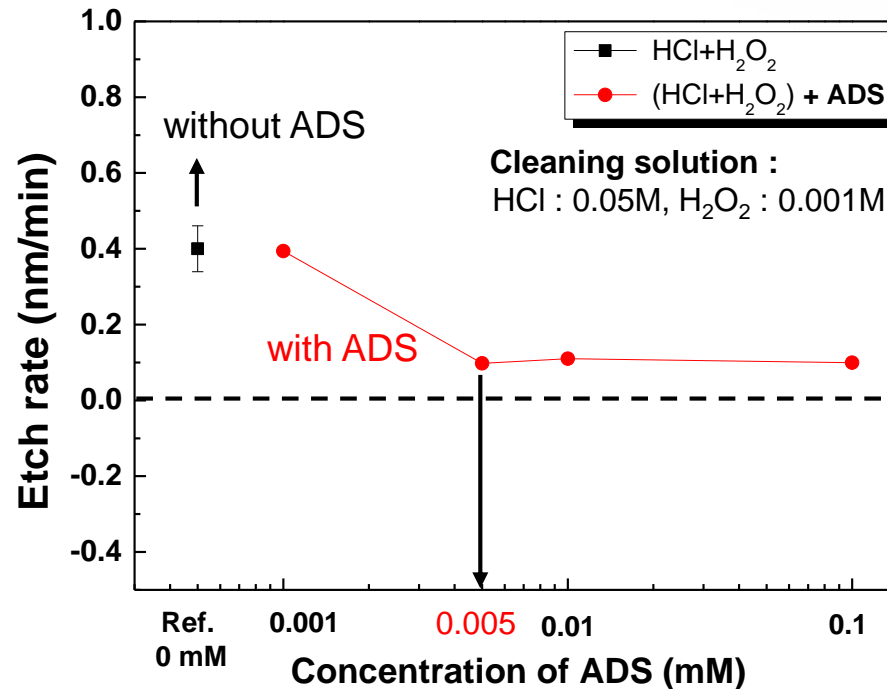


## ❖ (HCl + H<sub>2</sub>O<sub>2</sub>) + ADS



- Using ADS alone is ineffective to remove particles without etchant
  - ✓ Etching process is necessary to remove the particles
- PRE increases dramatically with the addition of ADS
  - ✓ Combined effect of surface etching and electrostatic repulsive force increases the PRE
  - ✓ PRE was increased drastically at the addition of 0.005 mM conc. of ADS

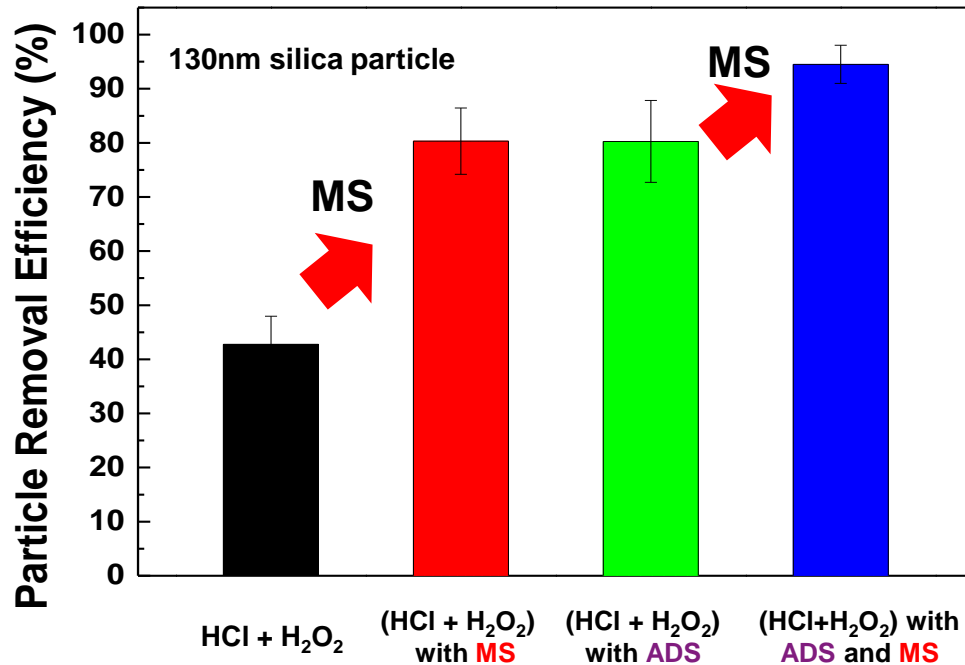
# Etch rate vs. ADS conc.



## ➤ Addition of ADS decreases the etch rate of InGaAs

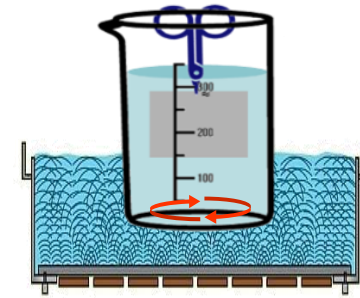
- ✓ Etch rate was decreased drastically at 0.005 mM and above conc. of ADS
- ✓ Introduction of ADS not only increasing the PRE but also decreasing the material loss

# PRE vs. Megasonic



## ❖ Cleaning condition

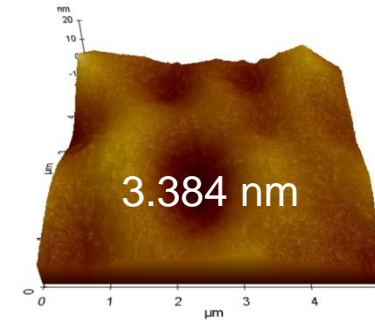
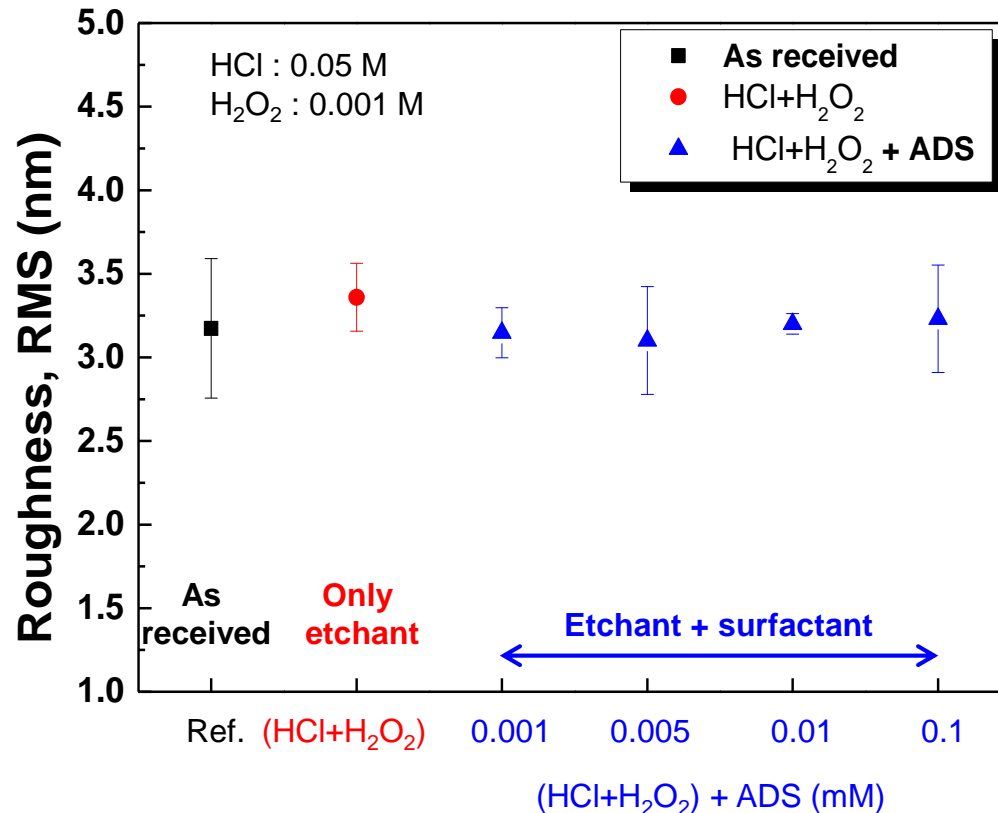
- ✓ Cleaning solution :  
HCl : 0.05M, H<sub>2</sub>O<sub>2</sub> : 0.001M  
ADS (0.005 mM)
- ✓ Megasonic (1 MHz, 600 W)



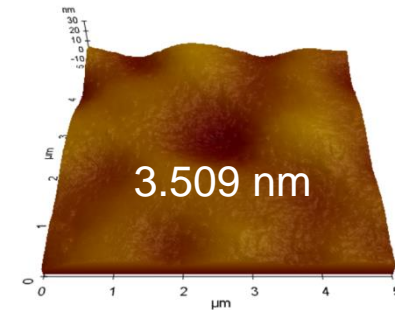
- Megasonic increases the PRE of various cleaning solutions
- HCl and H<sub>2</sub>O<sub>2</sub> cleaning solution with ADS and MS achieved above 95% PRE
  - ✓ Mechanism of megasonic cleaning : acoustic streaming, bubble collapse
  - ✓ Combined effects of **physical force, surface etching and electrostatic repulsive force**



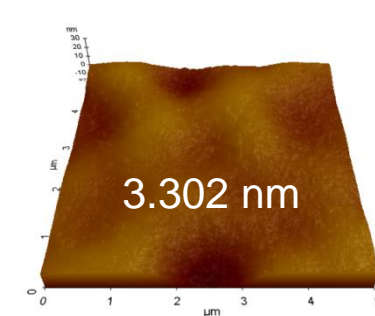
# Surface roughness



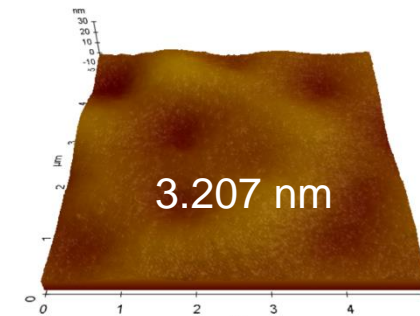
As received



(HCl+H<sub>2</sub>O<sub>2</sub>)



(HCl+H<sub>2</sub>O<sub>2</sub>) +  
ADS (0.001 mM)



(HCl+H<sub>2</sub>O<sub>2</sub>)  
+ ADS (0.005 mM)

- After cleaning process, there is **no significant change in roughness** compared to the initial state
- ✓ Etch amount was negligible to affect the roughness (**minimized surface damage**)

- ❖ **Acid cleaning solution** was used for **post InGaAs CMP cleaning** process.
- ❖ **Higher HCl conc. than  $H_2O_2$**  is considered as a **optimized cleaning solution** based on the results of material loss and surface oxidation.
- ❖ **ADS** was introduced for **increasing repulsive force** between silica particle and InGaAs substrate to **enhance the PRE**.
- ❖ Introduction of **ADS** not only **increasing the PRE** but also **decreasing the material loss**.
- ❖ There was **no noticeable roughness change** was observed after cleaning process compared to the initial state.
- ❖ **Acid cleaning with surfactant and megasonic achieved above 95% PRE.**



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Your Attention !!**

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