

MEGASONIC CLEANING OF WAFERS IN ELECTROLYTE SOLUTIONS: POSSIBLE ROLE OF ELECTRO-ACOUSTIC AND CAVITATION EFFECTS

Manish Keswani¹, Sriniraghavan¹, Pierre Deymier¹ and Steven Verhaverbeke²

¹The University of Arizona, Tucson

²Applied Materials



Outline

- INTRODUCTION
- PROPOSED WORK
- EXPERIMENTAL MATERIALS AND METHODS
- RESULTS AND DISCUSSION
- SUMMARY
- FUTURE WORK



INTRODUCTION



Growth and Challenges Integrated Circuit Industry

Year of Production	2005	2006	2007	2008	2009	2010	2011	2012	2013	Driver
DRAM ½ Pitch (nm) (contacted)	80	70	65	57	50	45	40	36	32	D ½
MPU/ASIC Metal 1 (M1) ½ Pitch (nm)(contacted)	90	78	68	59	52	45	40	36	32	M
MPU Physical Gate Length (nm)	32	28	25	23	20	18	16	14	13	M
Wafer diameter (mm)	300	300	300	300	300	300	300	450	450	D ½, M
Wafer edge exclusion (mm)	2	2	1.5	1.5	1.5	1.5	1.5	1.5	1.5	D ½, M
Front surface particles										
Killer defect density, $D_p R_p$ (#/cm ²) [A]	0.027	0.017	0.022	0.027	0.017	0.022	0.027	0.017	0.022	D ½
Critical particle diameter, d_c (nm) [B]	40.1	35.7	31.8	28.4	25.3	22.5	20.1	17.9	15.9	D ½
Critical particle count, D_{pw} (#/wafer) [C]	94.2	59.3	75.2	94.8	59.7	75.2	94.8	135.3	170.4	D ½
Back surface particle diameter: lithography and measurement tools (µm) [D][E]	0.16	0.12	0.12	0.12	0.1	0.1	0.1	0.1	NA	D ½
Back surface particles: lithography and measurement tools (#/wafer) [D][E]	400	400	200	200	200	200	200	200	NA	D ½
Back surface particle diameter: all other tools (µm) [D][E]	0.2	0.16	0.16	0.16	0.14	0.14	0.14	0.14	NA	D ½
Back surface particles: all other tools (#/wafer) [D][E]	400	400	200	200	200	200	200	200	NA	D ½
Critical GOI surface metals (10 ¹⁰ atoms/cm ²) [F]	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	MPU
Critical other surface metals (10 ¹⁰ atoms/cm ²) [F]	1	1	1	1	1	1	1	1	1	MPU
Mobile ions (10 ¹⁰ atoms/cm ²) [G]	1.9	1.9	2	2.2	2.4	2.5	2.3	2.5	2.4	MPU
Surface carbon (10 ¹³ atoms/cm ²) [H]	1.4	1.3	1.2	1	0.9	0.9	0.9	0.9	0.9	
Surface oxygen (10 ¹³ atoms/cm ²) [I]	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	D ½, M
Surface roughness LVGX, RMS (Å) [J]	4	4	4	4	4	2	2	2	2	
WAS Silicon loss (Å) per cleaning step [K]	0.8	0.7	0.5	0.4	0.4	0.3	0.3	0.3	0.2	M
IS Silicon loss (Å) per cleaning step [K]	0.8	0.7	0.5	0.4	0.4	0.3	0.3	0.3	0.2	
WAS Oxide loss (Å) per cleaning step [L]	0.8	0.7	0.5	0.4	0.4	0.3	0.3	0.3	0.2	M
IS Oxide loss (Å) per cleaning step [L]	0.8	0.7	0.5	0.4	0.4	0.3	0.3	0.3	0.2	
Allowable watermarks # [M]	0	0	0	0	0	0	0	0	0	M

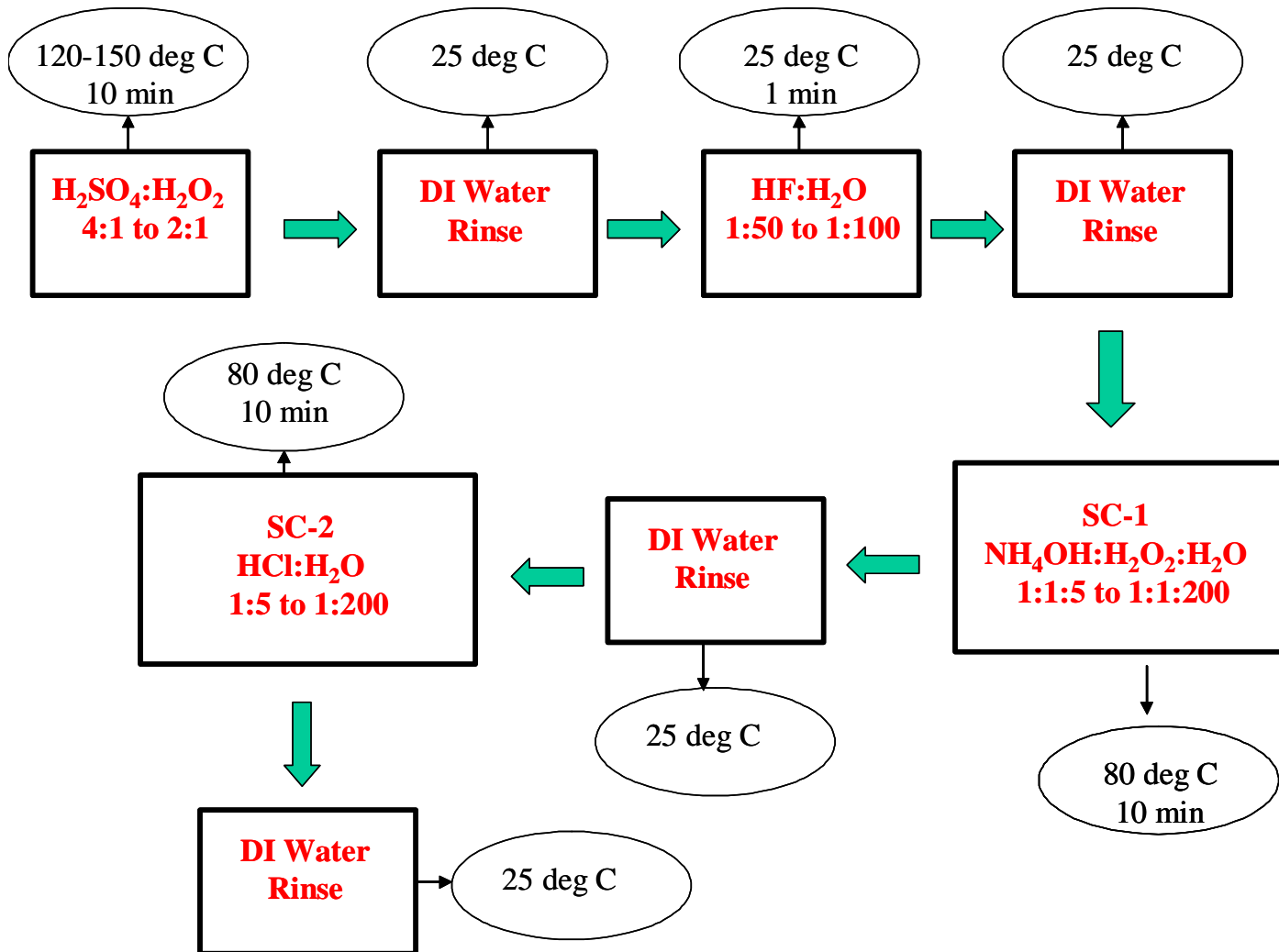
- Fifty Percent of yield losses due to particle contamination
- Over 200 cleaning steps
- Critical particle diameter and count to be 25 nm and 59 #/wafer respectively for a 300 mm wafer
- Particulate impurities on the wafer critically affects the device performance, reliability, and product yield of integrated circuits

ITRS Requirements

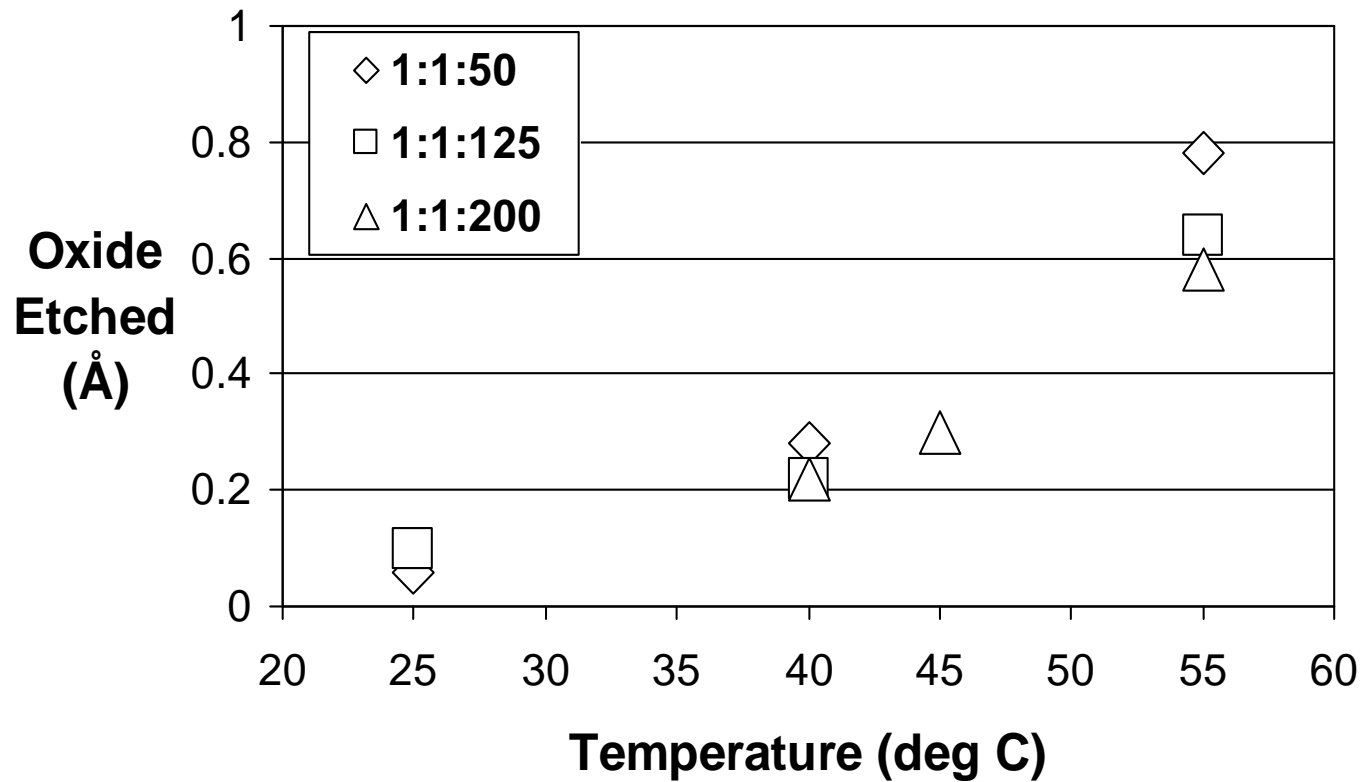


SRC/Sematech Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Typical Wafer Cleaning Sequence



Etching of Thermal Oxide in SC1 (time = 1 min)



Strong Etch Dependence on Temp

Courtesy: Dr. J. Butterbaugh, FSI

Wafer Cleaning Techniques

- **High Pressure Water Jet Cleaning**
- **Immersion Cleaning and Spray Cleaning without Megasonics**
- **Immersion Cleaning and Spray Cleaning with Megasonics**
 - Very simple and easy to use.
 - Great potential in removing sub – micron size particles in combination with dilute chemistries



Megasonic Cleaning

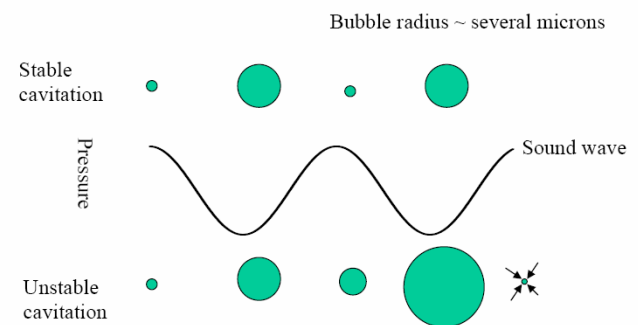
MHz frequency sound waves with different cleaning chemistries

Particle on wafer surface

- Hydrodynamic boundary layer thickness 400 - 1000 μm in DI water
(viscosity = 10^{-3} Pa-s, density = 10^3 kg/m³, velocity = 0-10 m/s)
- Acoustic boundary layer at 1 MHz in DI water ~ 0.5 μm

Particle Removal Mechanisms:

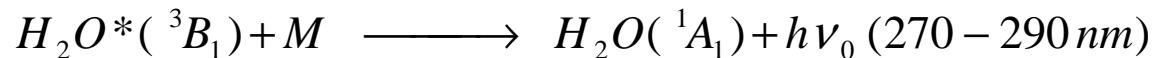
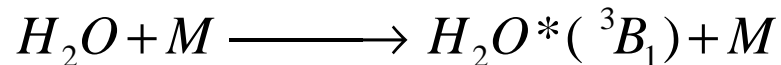
- **Acoustic Streaming:** Drag forces and rolling moments @S/L interface to help particle removal.
- **Acoustic Cavitation:**
High energy shock waves or fluid jet dislodging the particle.
- **Chemistry + Sonic field**



Physical Effect of Cavitation

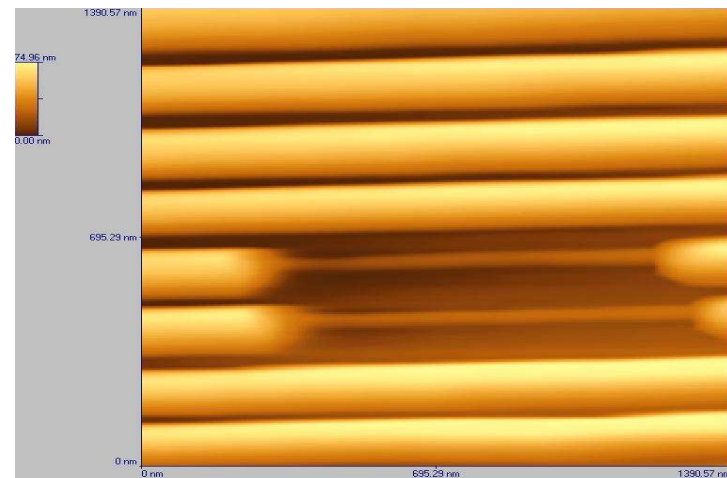
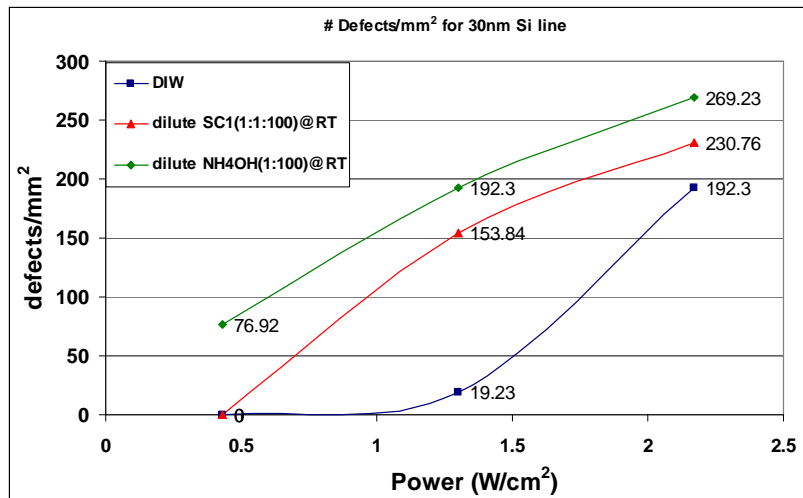
Sonoluminescence (Photon emission)

- At collapse, the gas inside the cavity reaches extremely high temperatures (4300 K) and pressures (few hundred bars).
- Results in production of free radical species (excited hydroxyl radicals).
- Recombination of free radicals gives rise to photon emission.



Challenges Associated with Megasonic Cleaning

- Megasonic cleaning is typically done in alkaline pH condition (using SC1-1 solution) which causes loss of wafer surface due to etching
- Use of DI water instead of SC-1 requires much higher power density. A significant level of damage of features (in the case of patterned wafers) occurs at these power densities



Images from -H. Shende, MS Thesis, The University of Arizona, 2006



PROPOSED WORK

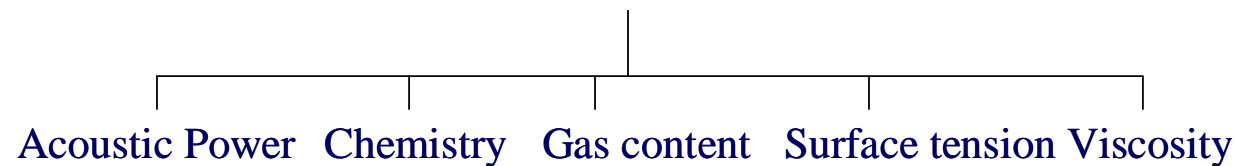


Need for Present Work

1) Identify a non-etching chemistry with advanced non-damaging megasonics

2)

Focus of Past Work



- No literature on effect of Ionic Strength on Particle Removal Studies in Megasonics
- Industry uses cleaning solutions (eg SC1, SC2, TMAH, Piranha etc) which may have varying ionic strengths

Use of Electrolytes can achieve both objectives

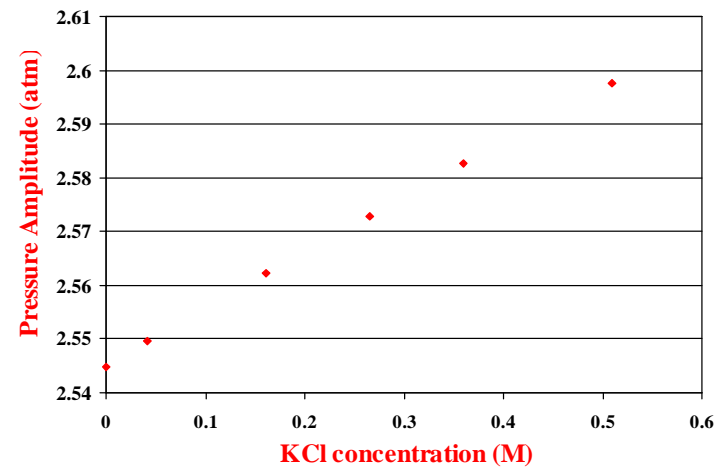
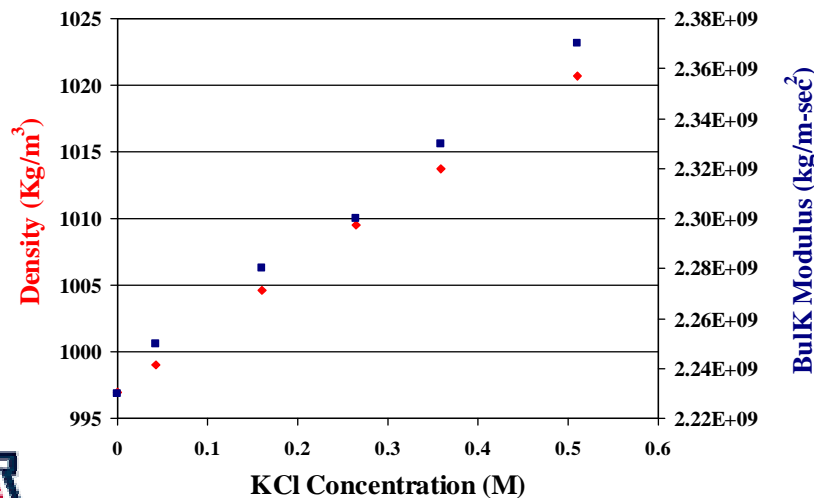


Why Study KCl Solutions?

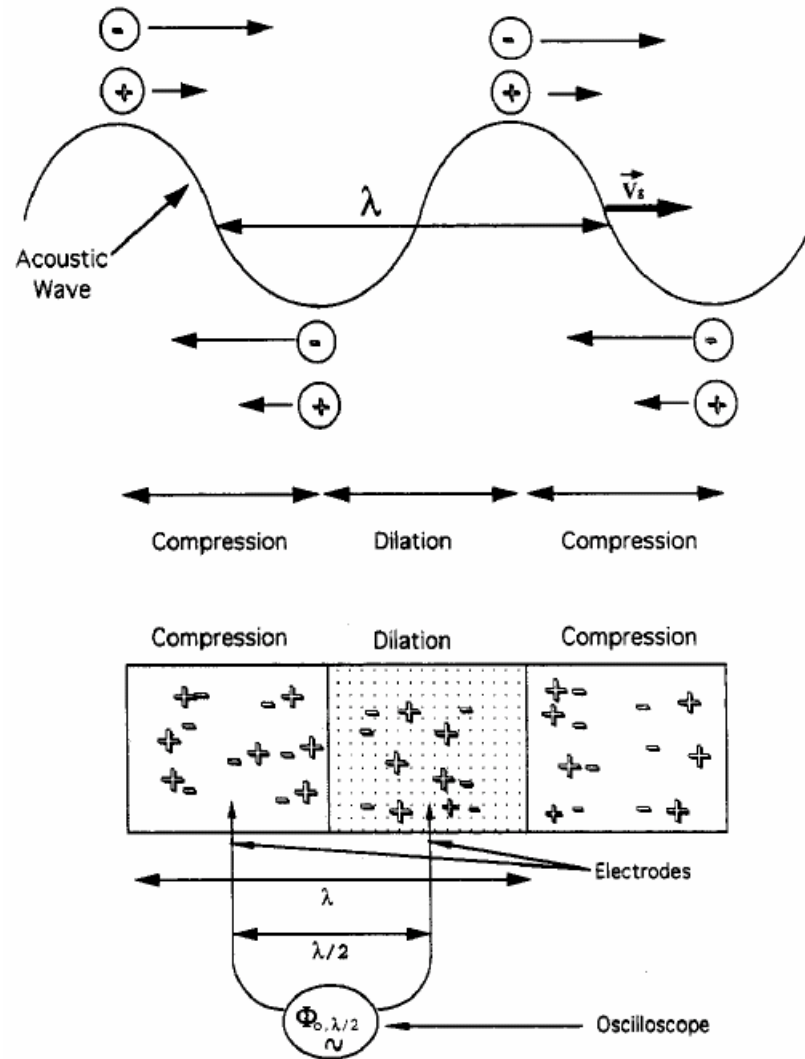
- Addition of KCl to water increases the bulk modulus (κ) and density (ρ_0) of solution and hence may modulate the pressure amplitude (a) of the propagating sound wave and affect Cavitation
- The propagation of sound waves through an electrolyte solution containing particles is known to result in the generation of two types of oscillating electric potentials, namely, Ionic Vibration Potential (IVP) and Colloid Vibration Potential (CVP).
- These potentials and their associated electric fields can exert forces on particles adhered to a surface, resulting in their removal.

$$a = (2I)^{0.5} (\rho_0 \kappa)^{0.25}$$

$I = \text{Intensity of transducer} = 2.17 \text{ W/cm}^2$



Debye Effect



S. D. Vidal et al., J. Phy. Chem., 99, pp.6733-6738 (1995)

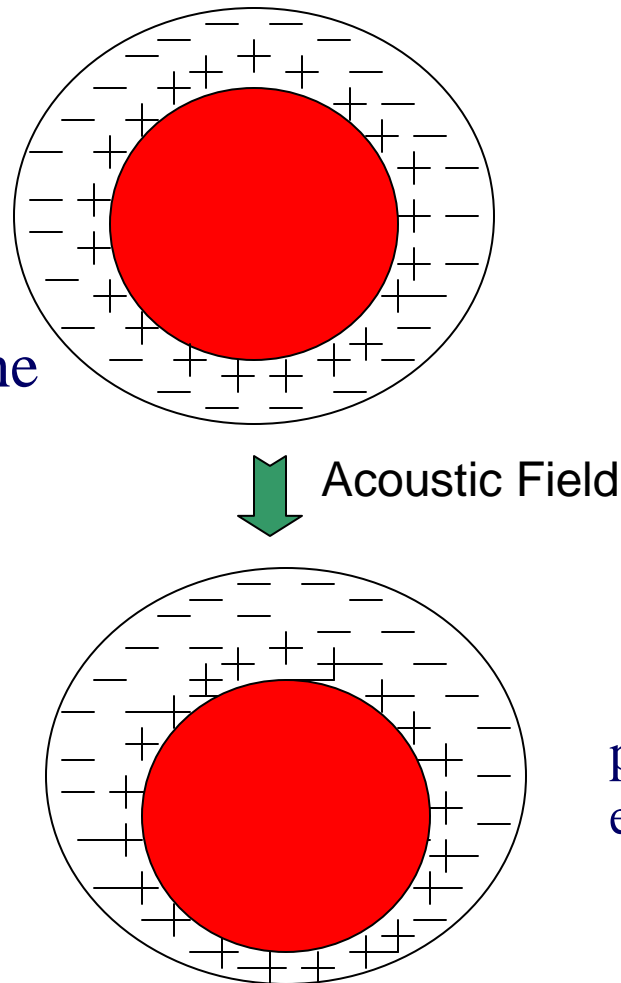


Interaction of Sonic Field with Colloidal Particles: Colloid Vibration Potential (CVP)

periodic polarization of the ionic atmosphere surrounding the particles.

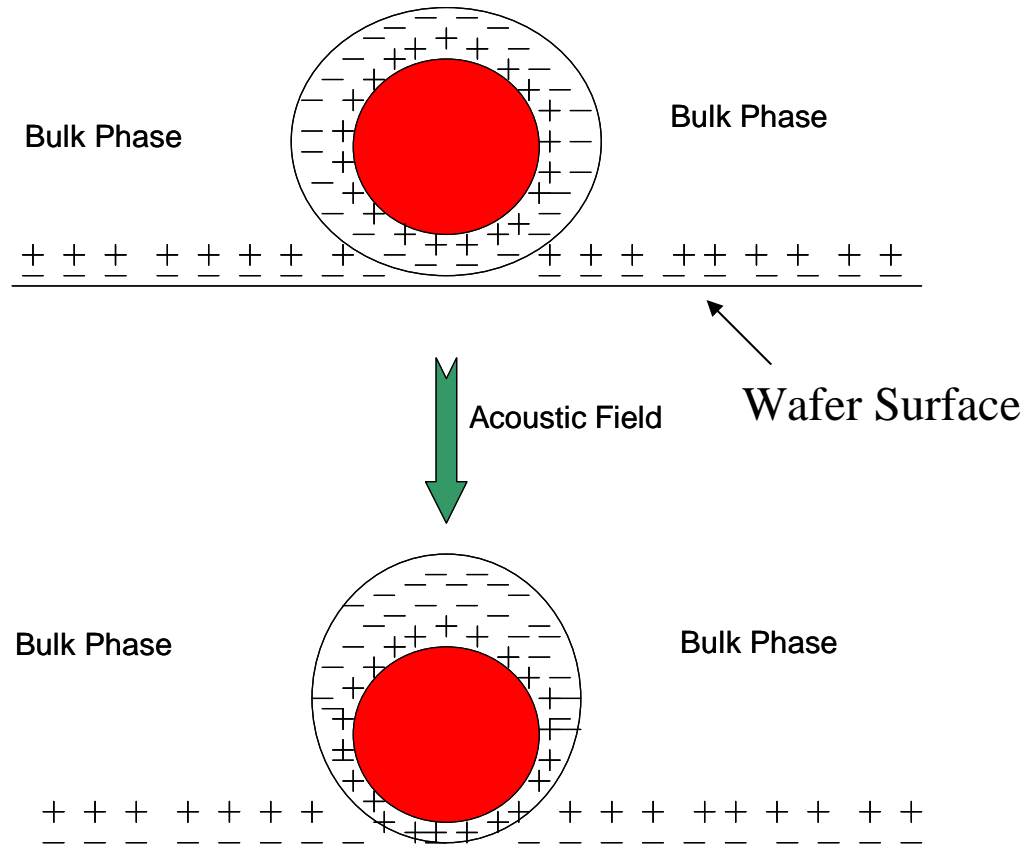
CVP ~ 5 to 10 mV for silica suspensions subjected to sound waves of 0.2 MHz at 0.22 W/cm² of power density

E. Yeager et al., *The Journal of the Acoustical Society of America*, 25, 3, 456-460 (1953)



periodic polarization causes each particle to act as a vibrating dipole

Dipole Moment Formation Due to Asymmetry of Double Layer Around a Charged Particle Adhered on a Wafer Surface



Can the electric field associated with CVP exert a force on charged particles adhered to a surface, resulting in their removal?



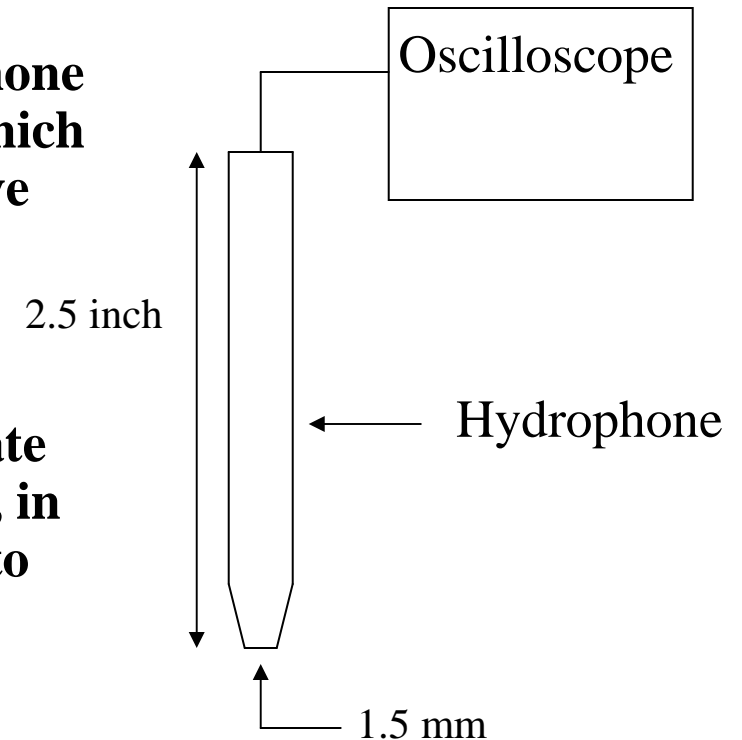
EXPERIMENTAL MATERIALS AND METHODS



Hydrophone-Oscilloscope Experiments

Method and Analysis

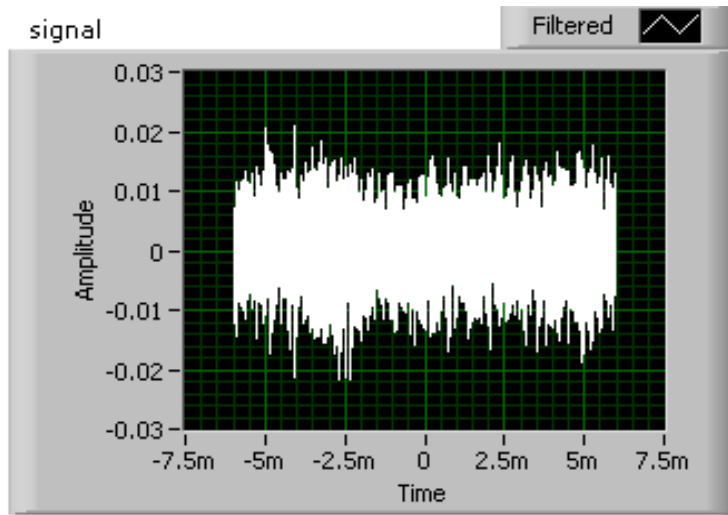
- Experiments were performed in a Prosys immersion megasonic system using a hydrophone (Onda Corporation/Specialty Engineering) which was positioned in the far field region to achieve consistency in results.
- The set up has a capacity to acquire 65000 continuous samples for each run. Sampling rate was 20 million samples/sec. The output signal, in volts, from the oscilloscope is then converted to the Root Mean Square value. Fast Fourier Transform of the raw output signal was performed to convert the data from time to frequency domain.



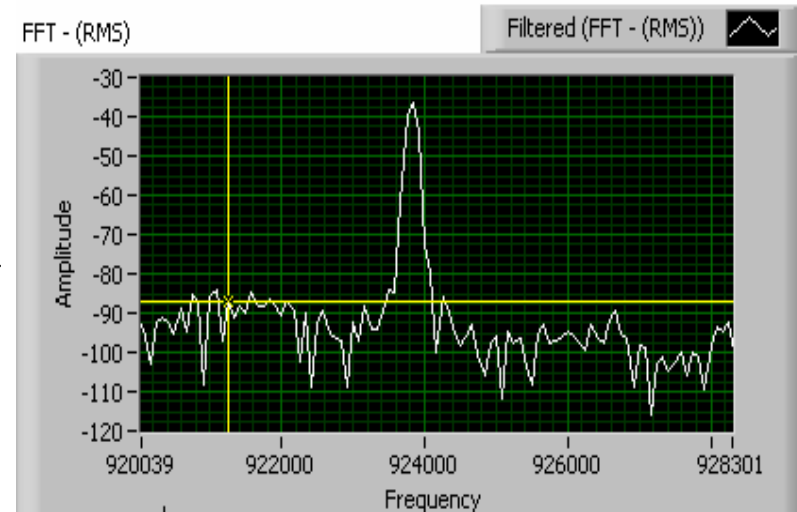
Hydrophone donated by Dr. Steven Verhaverbeke, Applied Materials

SRC/Sematech Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Spectral Analysis using Fast Fourier Transform



FFT



Linear Theory

$$\rho \omega_r^2 R_r^2 = 3 \gamma (P_0 + 2\sigma/R_r) - 2 \sigma/R_r$$

ρ is the density of the liquid

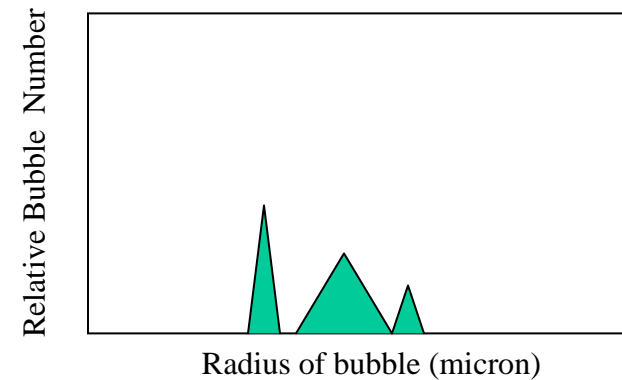
ω_r is the resonance frequency of the bubble

R_r is the radius of the resonating bubble

γ is the ratio of specific heats of the gas

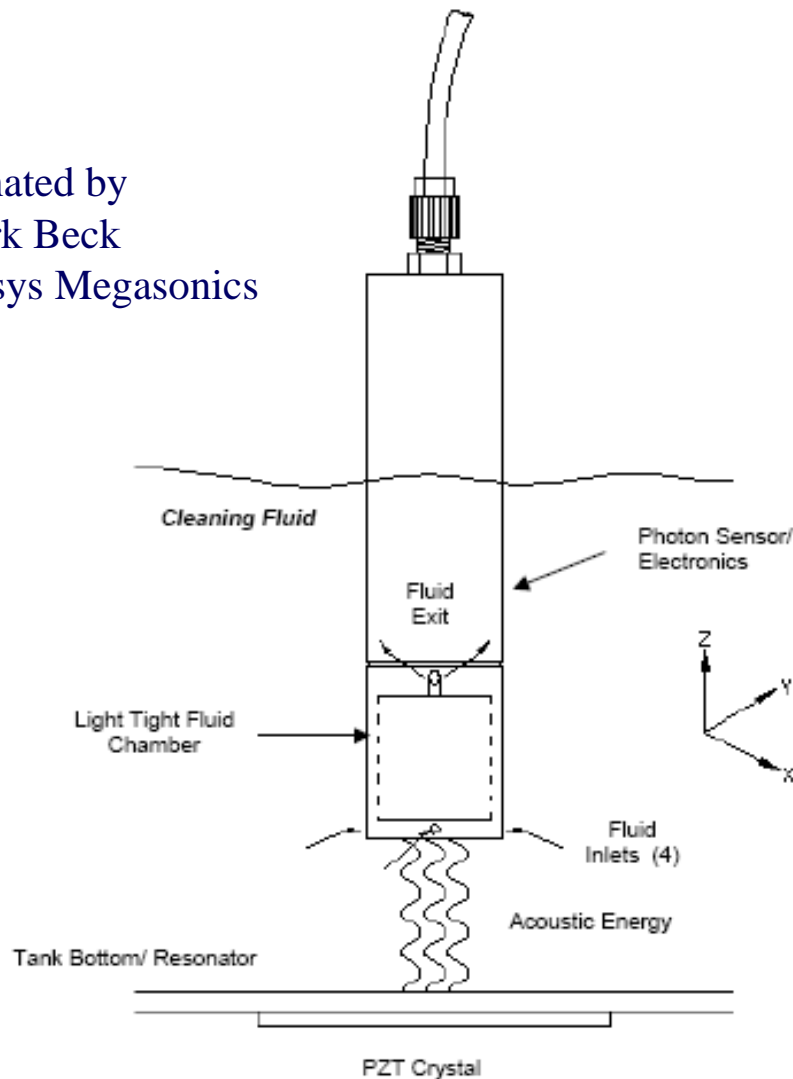
P_0 is the steady pressure in the absence of the sound field

σ is the surface tension of liquid



How to measure cavitation???

Donated by
Mark Beck
Prosys Megasonics



- Detects Sonoluminescence produced by Cavitation (Spectral Range 270 -650 nm)
- Real Time Monitoring of Cavitation Density
- Indicator of Feature Damage



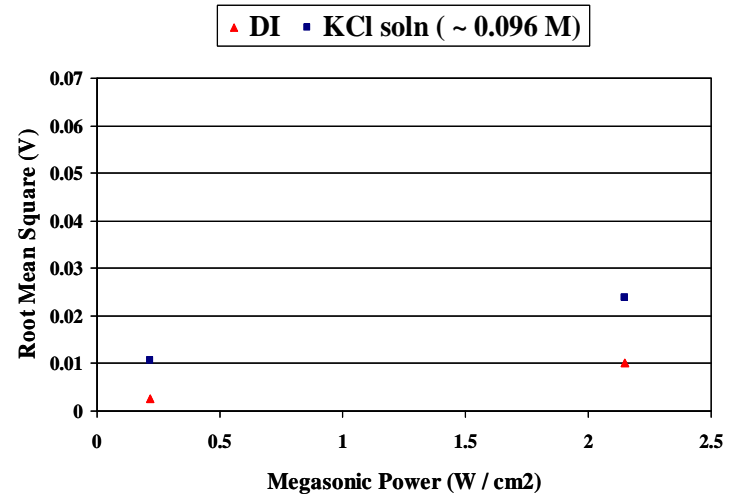
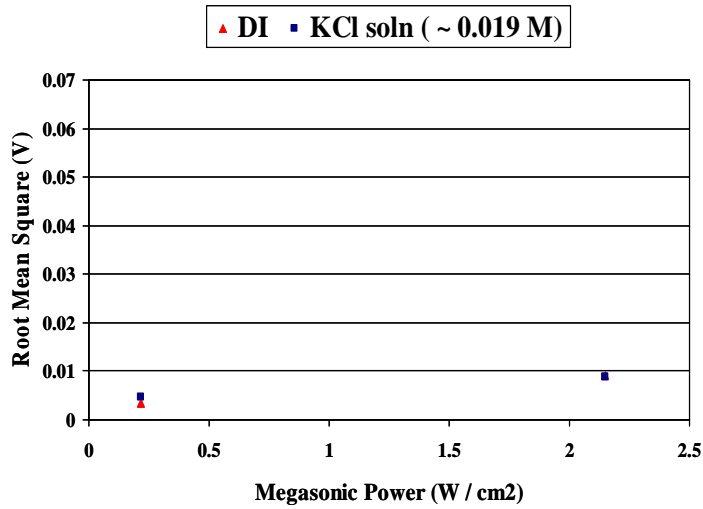
Wafer Pre-clean, Contamination and Cleaning Protocol

- **Pre-cleaning of 150-mm silicon wafers in 1:1:50 SC1 solution**
- **Aminated and Plain silica particles (Mean Size ~ 370 ± 20 nm purchased from Corpuscular Inc.) used for contamination. Typical particle count on wafers after deposition : ~ 2500**
- **Cleaning done in a Prosys immersion megasonic tank for one minute at 30 °C**
- **Surfscan 5500 used for particle counts**

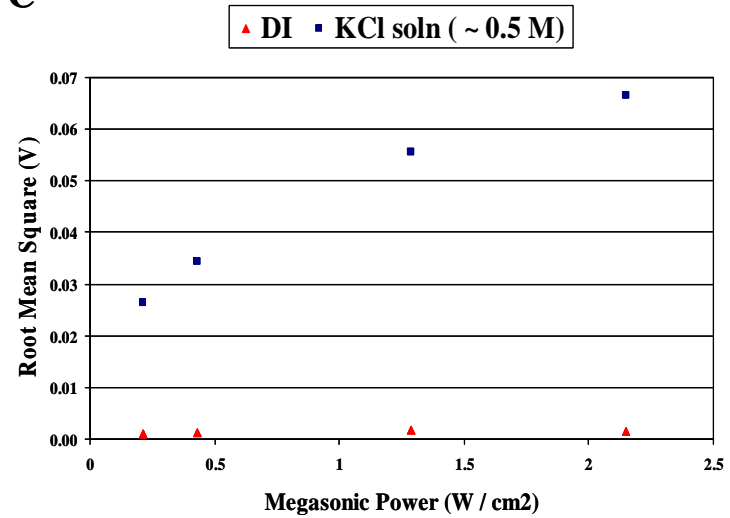
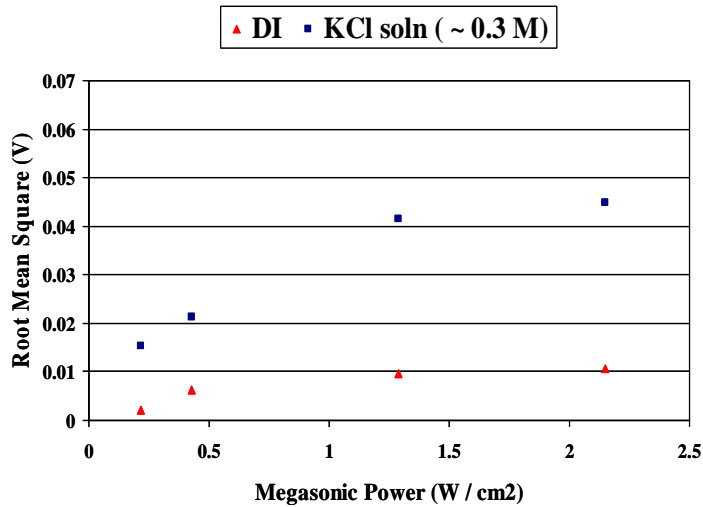


RESULTS AND DISCUSSION

Role of KCl on Sound Wave Amplitude

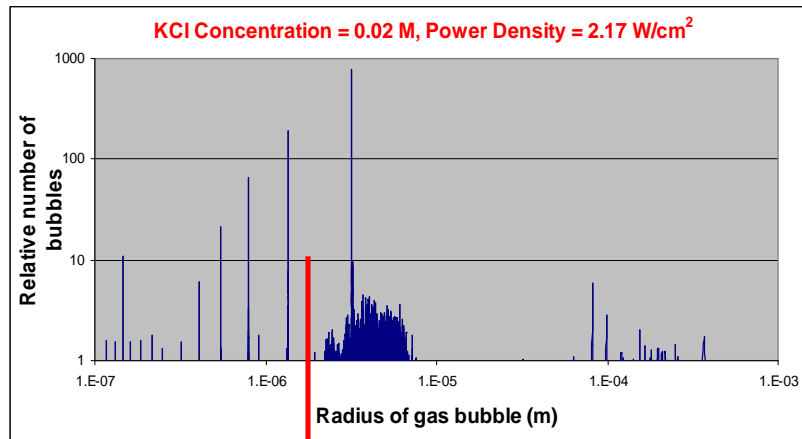


T = 33 C



Cavity Size Distribution from FFT and Energy Balance

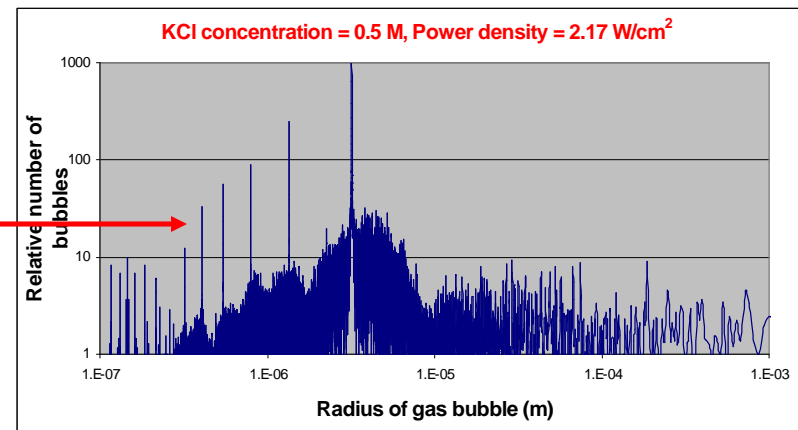
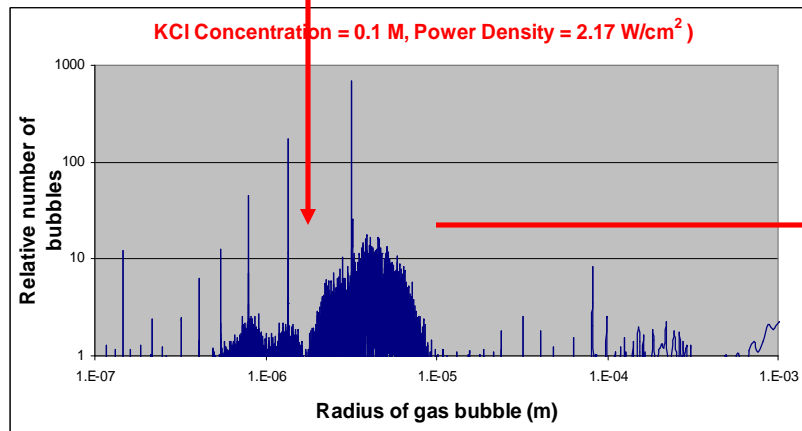
Correlating frequencies of oscillating bubbles with their sizes



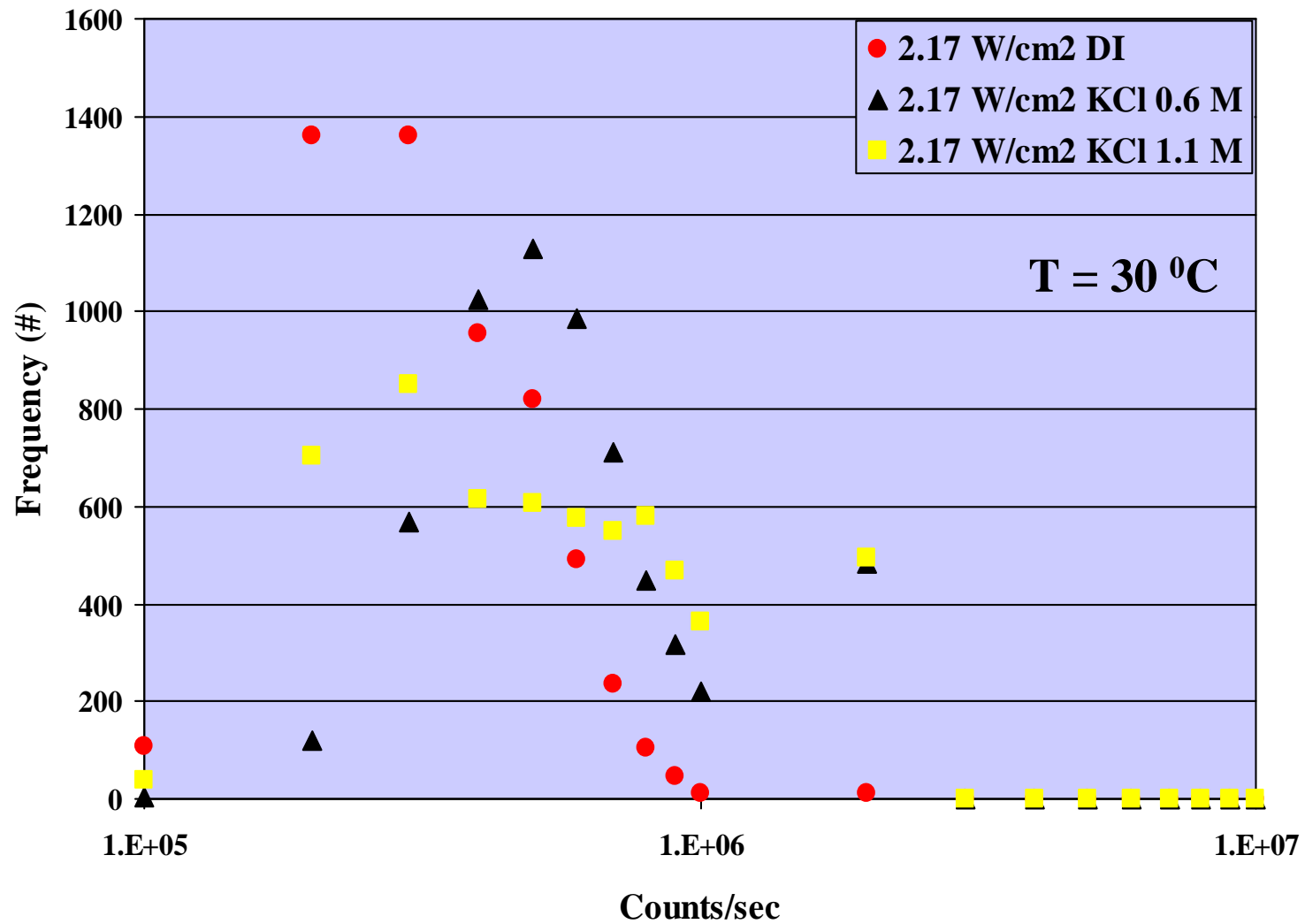
Increasing appearance of bubbles with increase in KCl concentration

Distribution of bubbles between 1-1000 micron

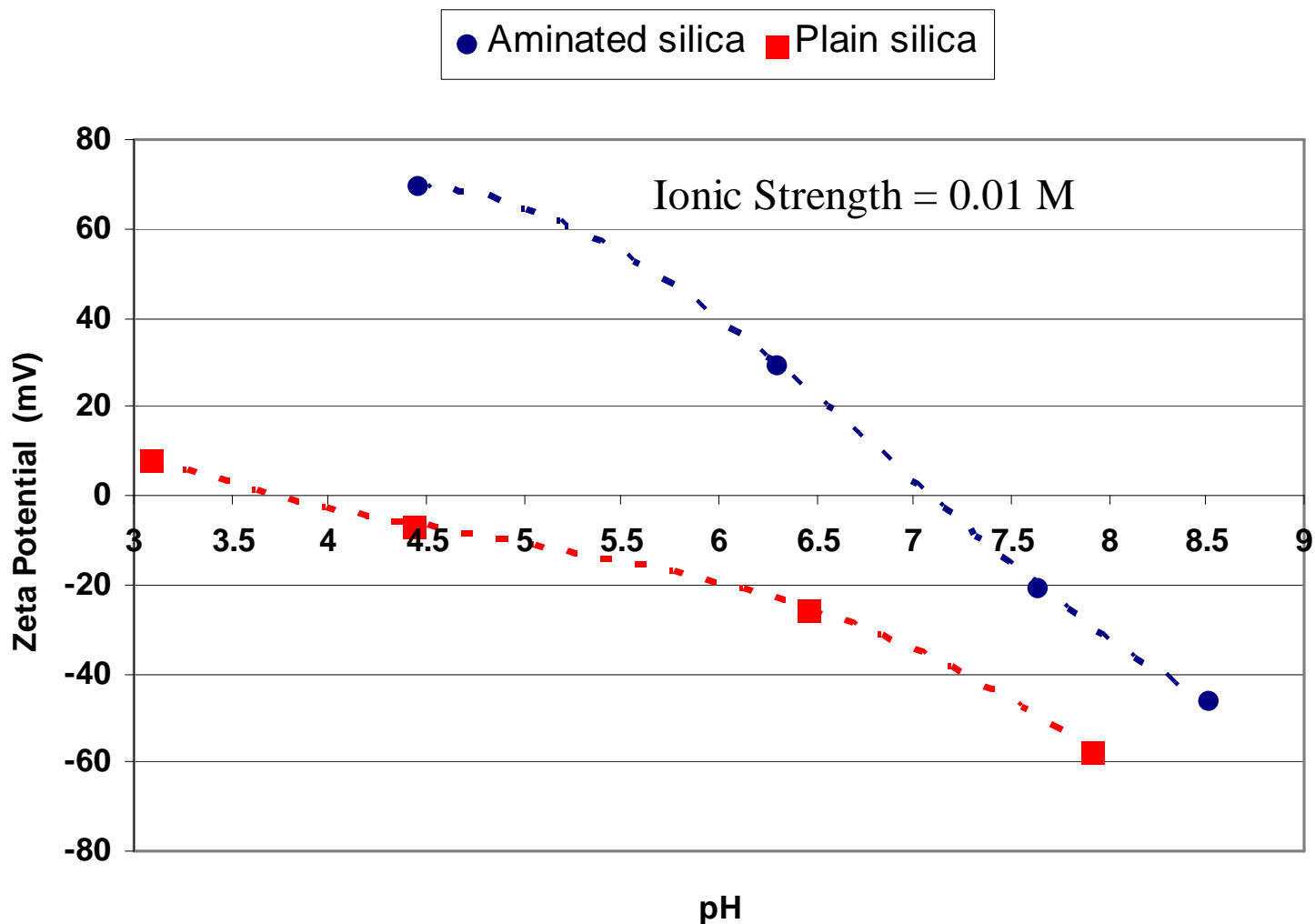
Bubble size larger than about 1 mm can be ignored as such bubbles will be removed from the system due to buoyancy.



Sonoluminescence in KCl Solution



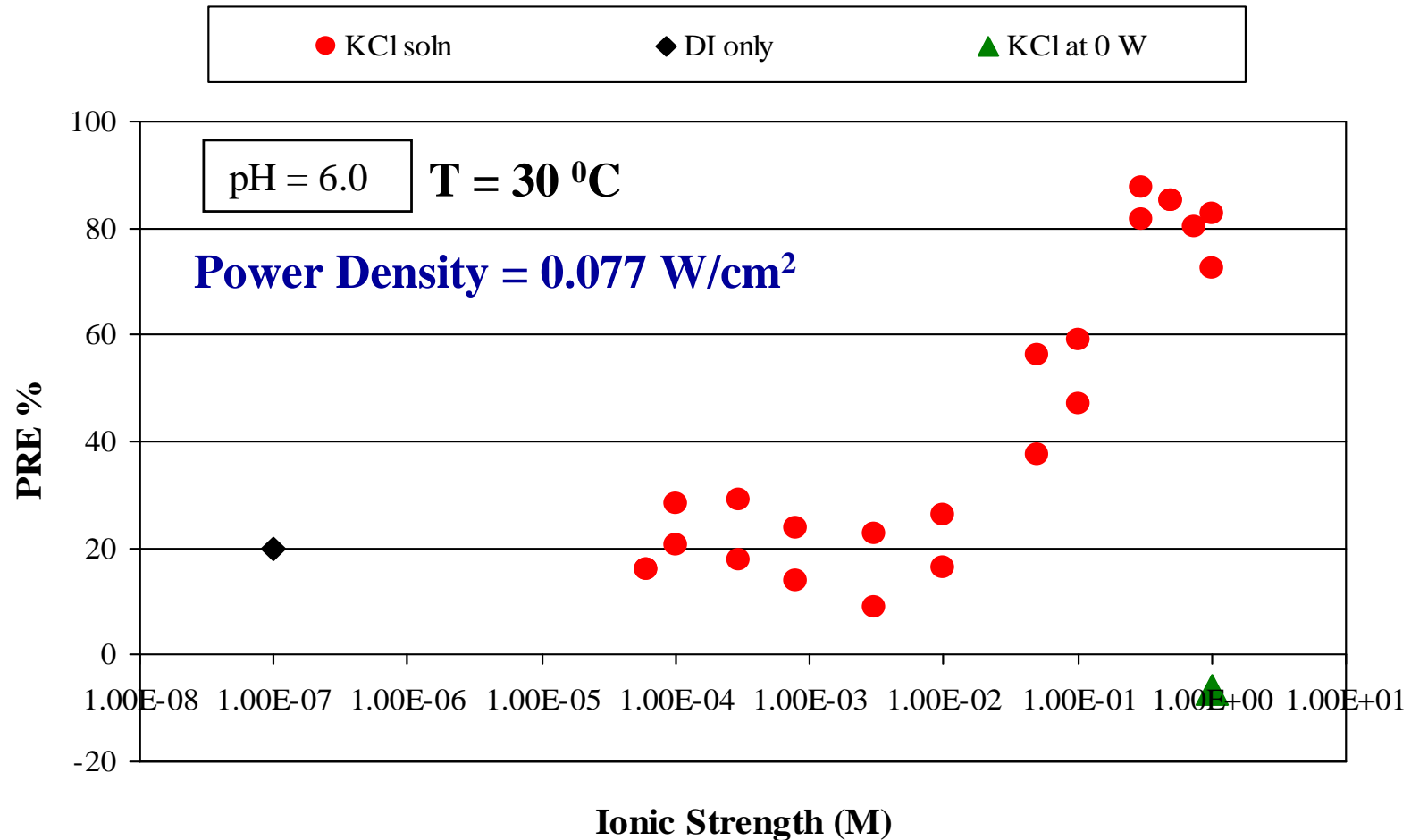
Zeta Potential of Aminated and Plain Silica Particles



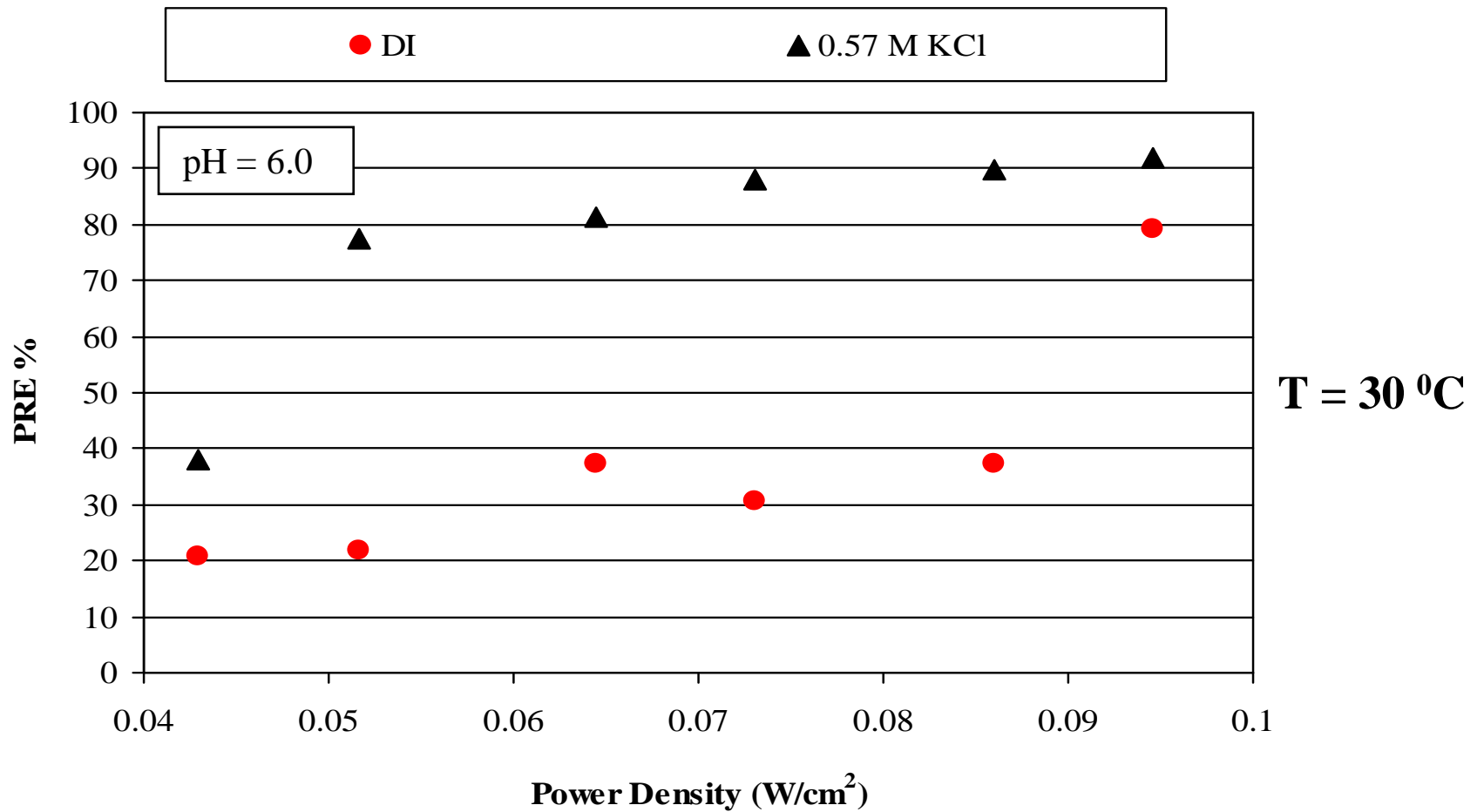
The isoelectric point (IEP) of aminated silica and plain silica particles was found to occur at pH values of 7.1 and 3.9 respectively.



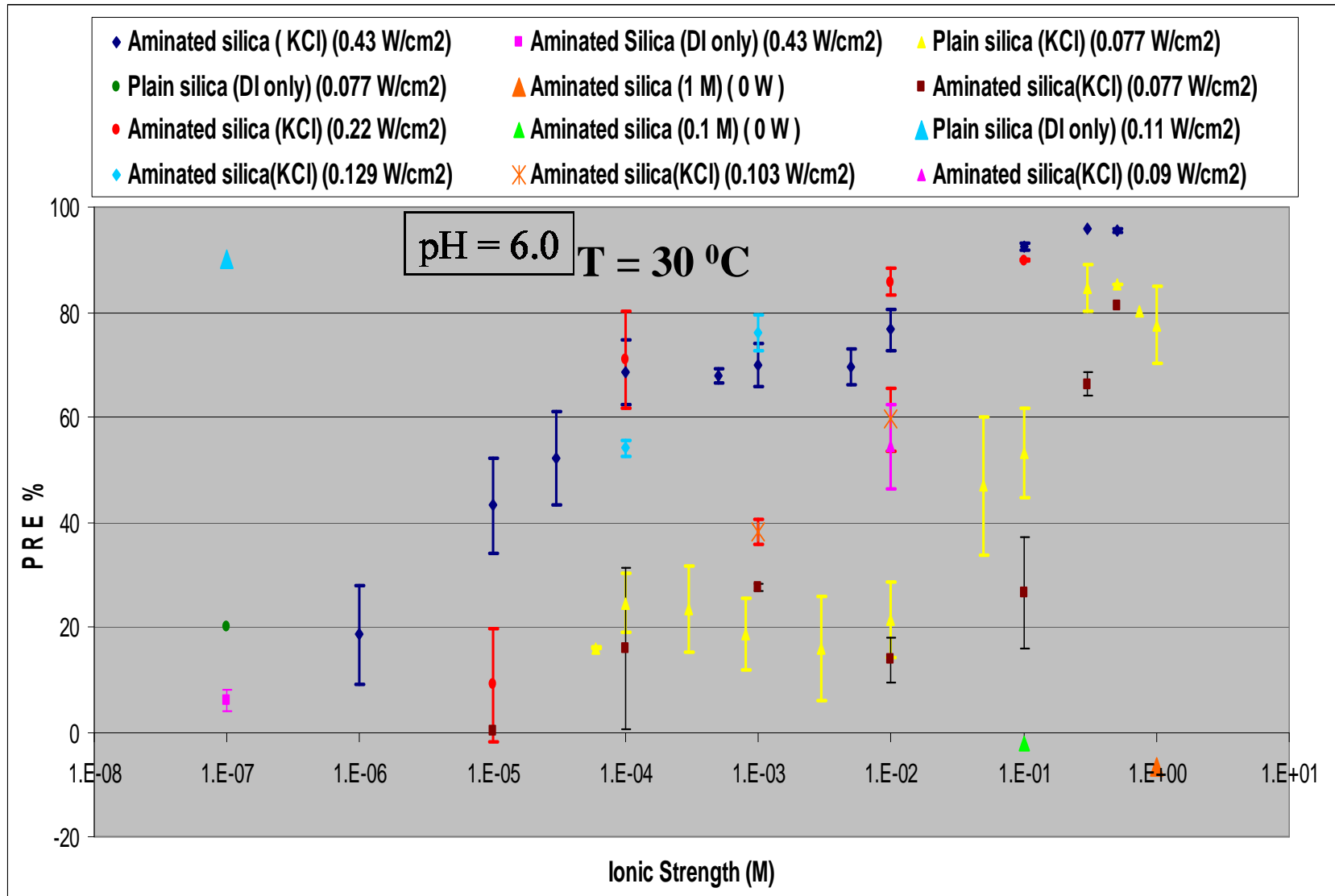
Megasonic Removal of Plain Silica Particles in KCl Solutions of Different Concentration



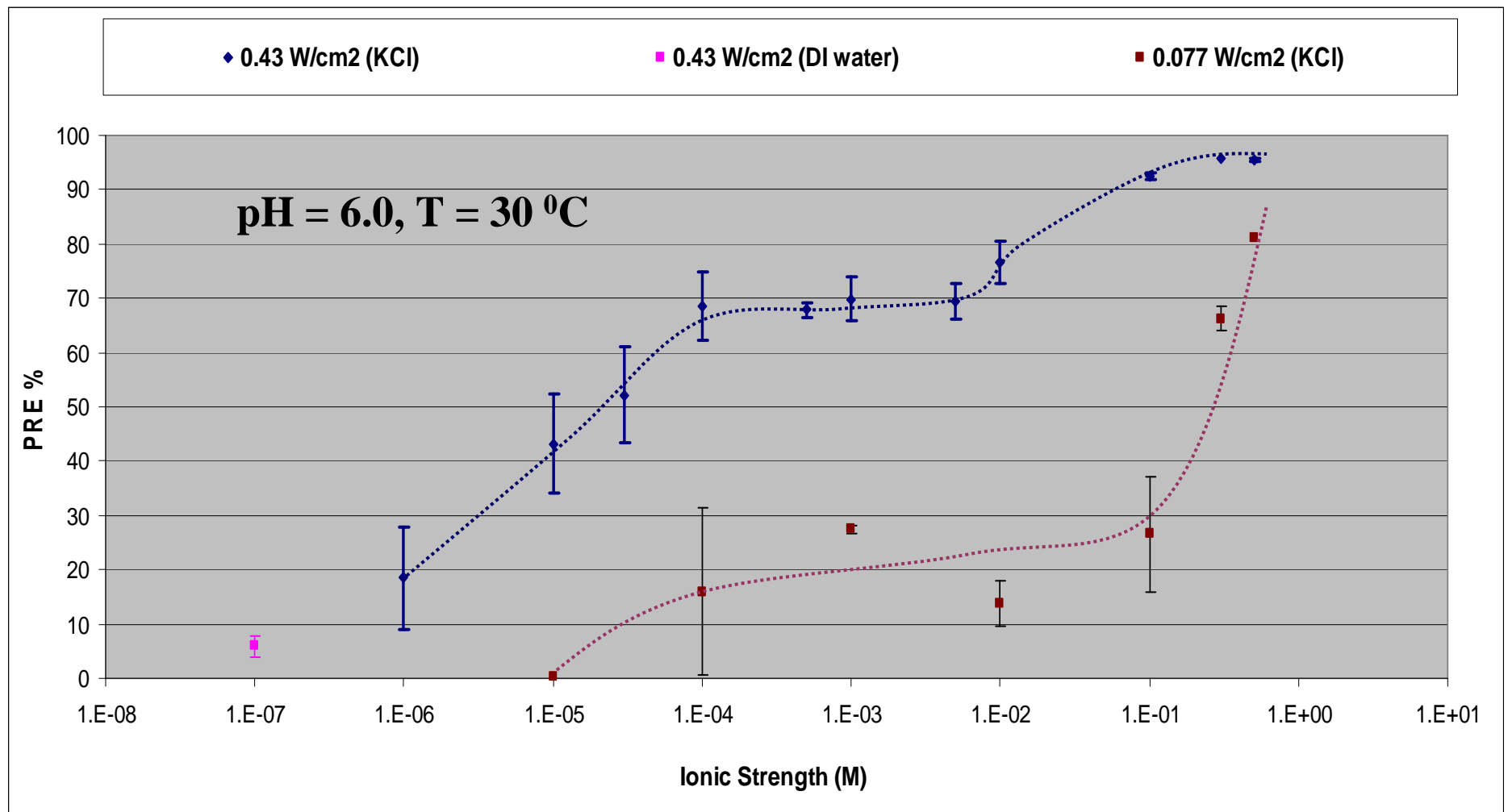
Megasonic Removal of Plain Silica Particles in 0.57 M KCl Solution at Different Power Densities



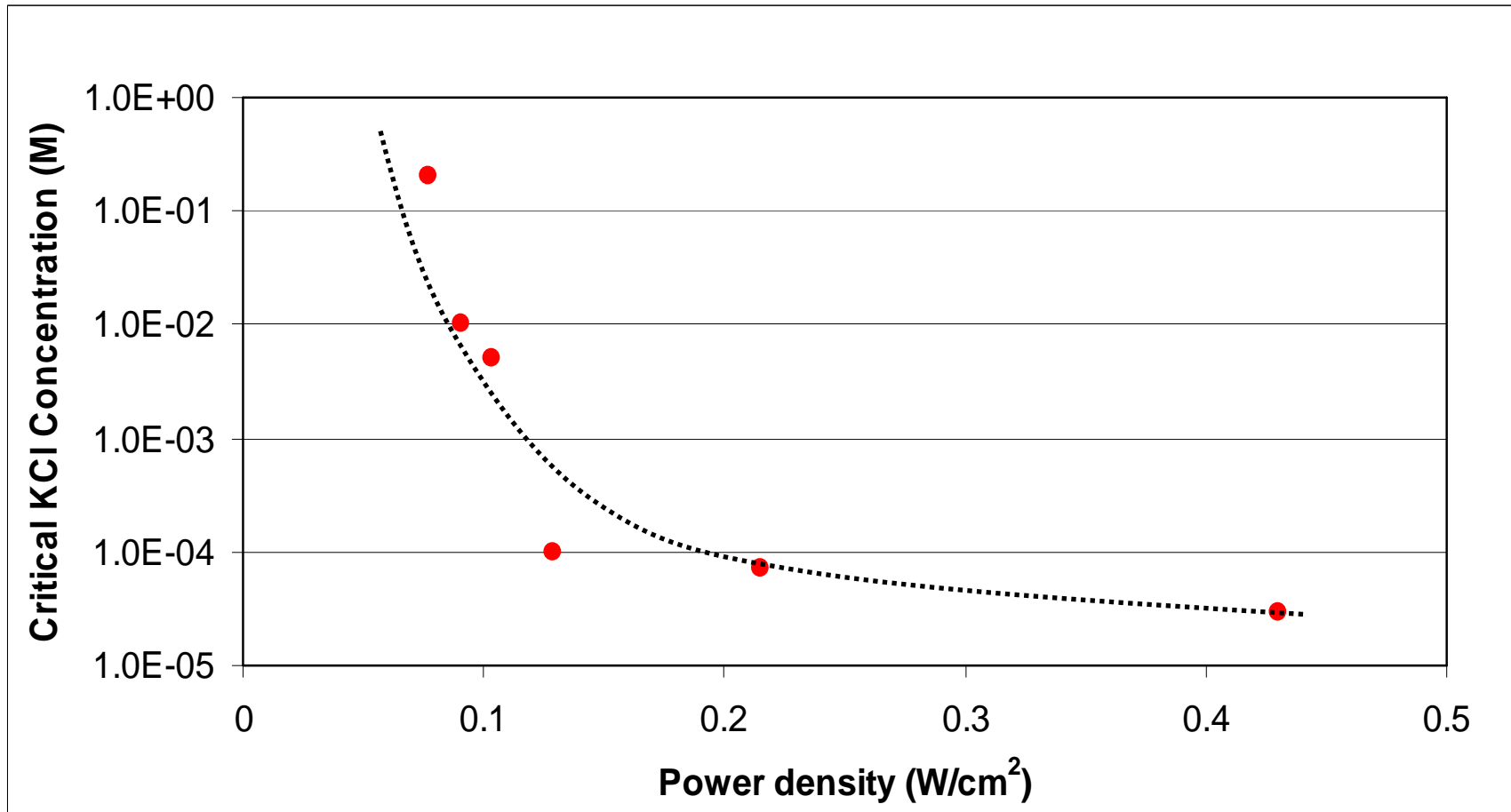
Megasonic Removal of Aminated Silica Particles in KCl Solutions of Different Concentration at Different Power Densities



Megasonic Removal of Aminated Silica Particles in KCl Solutions of Different Concentration at Different Power Densities



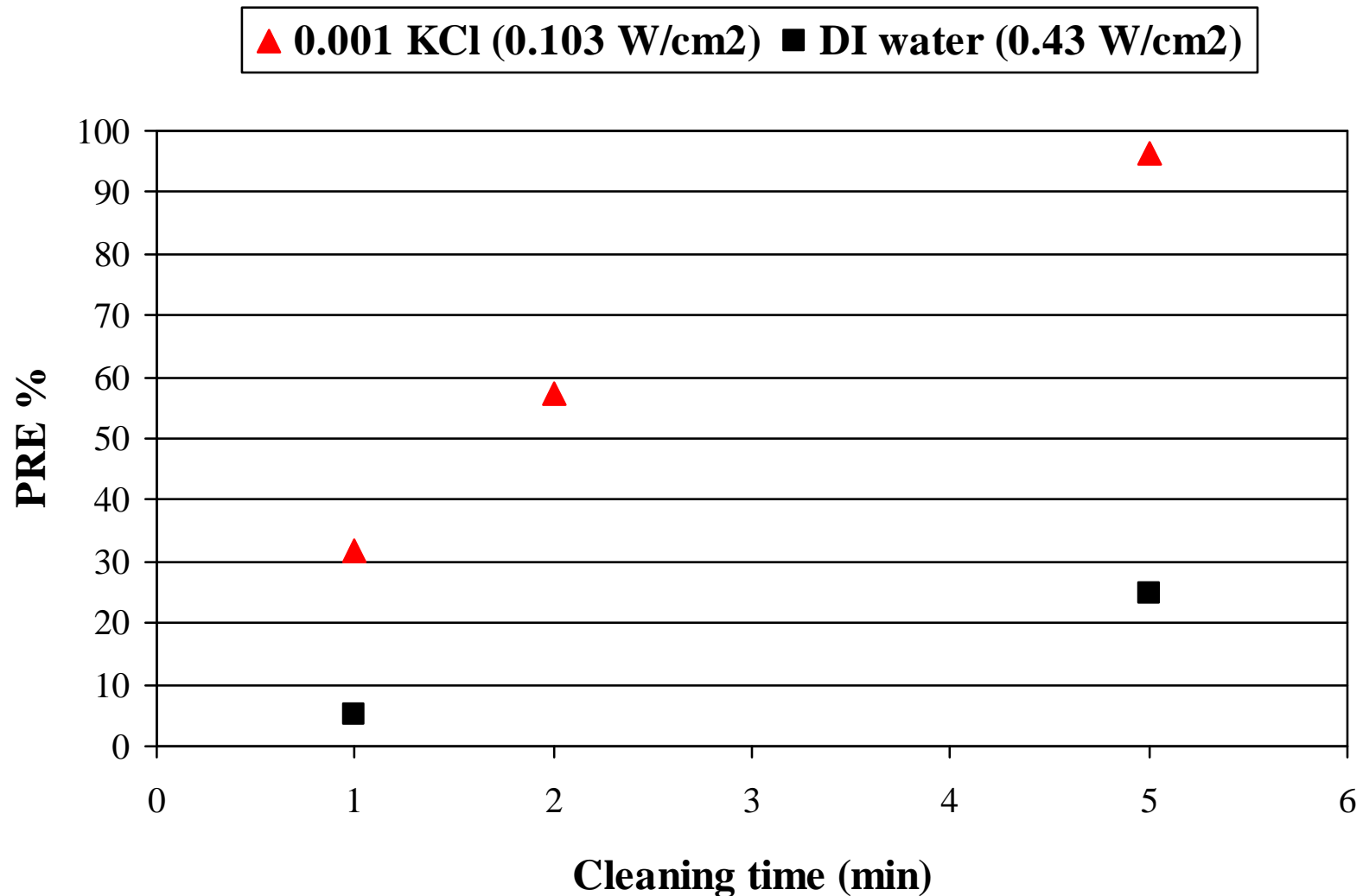
Critical Concentration of KCl for Removal of Aminated Silica at Different Power Densities



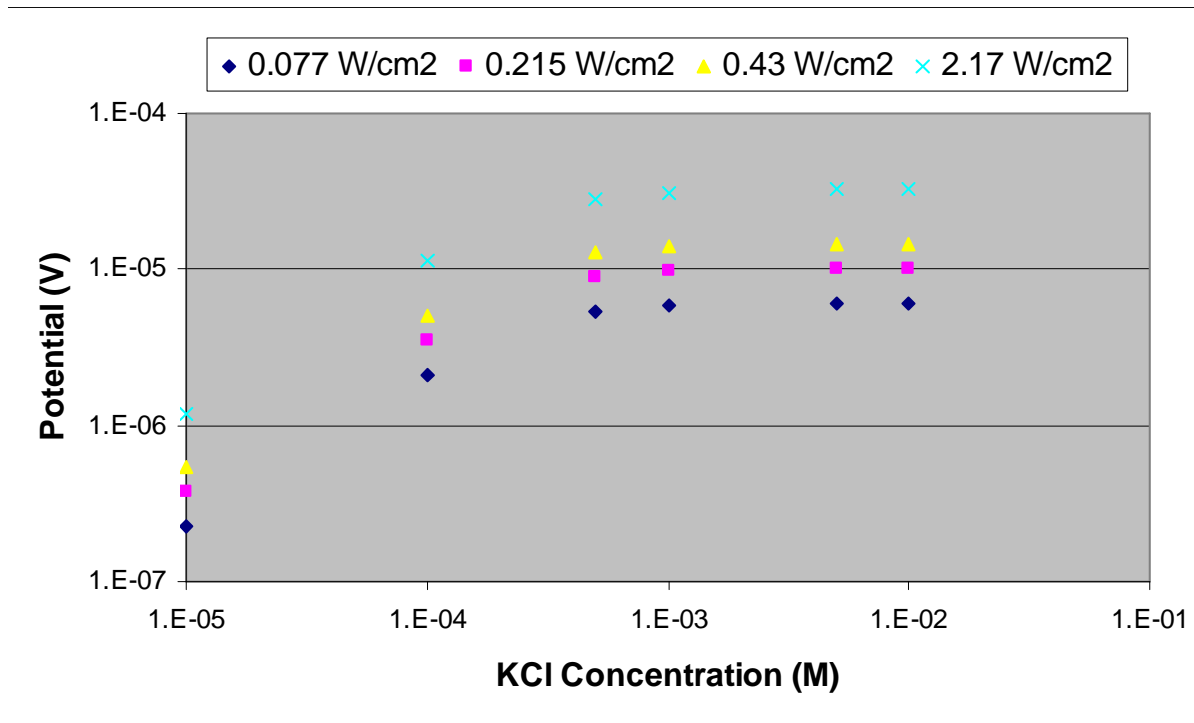
Critical concentration (Cc) is defined as the concentration at which 50 % particle removal efficiency (PRE) is achieved



Effect of Cleaning Time on Removal of Aminated Silica Particles



Effect of KCl Concentration on Debye Potential



$$\phi_0 = c u_0 \frac{(4\Pi / D w) \sum (\bar{n}_j m_j e_j / f_j)}{[1 + ((4\Pi / D w) \sum (\bar{n}_j e_j^2 / f_j))^2]^{0.5}}$$

Removal Force (10⁻³ M KCl and 2.17 W/cm²) = 10⁻¹⁵ N

Van der Waals Force = 3 * 10⁻¹⁰ N

$$e_i X - f_i (v_i - v_0) = m_i (dv_i/dt)$$

Force Balance (1)

$$(\delta n_i / \delta t) + \delta(n_i v_i) / \delta t = 0$$

Continuity Equation (2)

$$D (\delta X / \delta x) = 4\Pi \Sigma (n_j e_j)$$

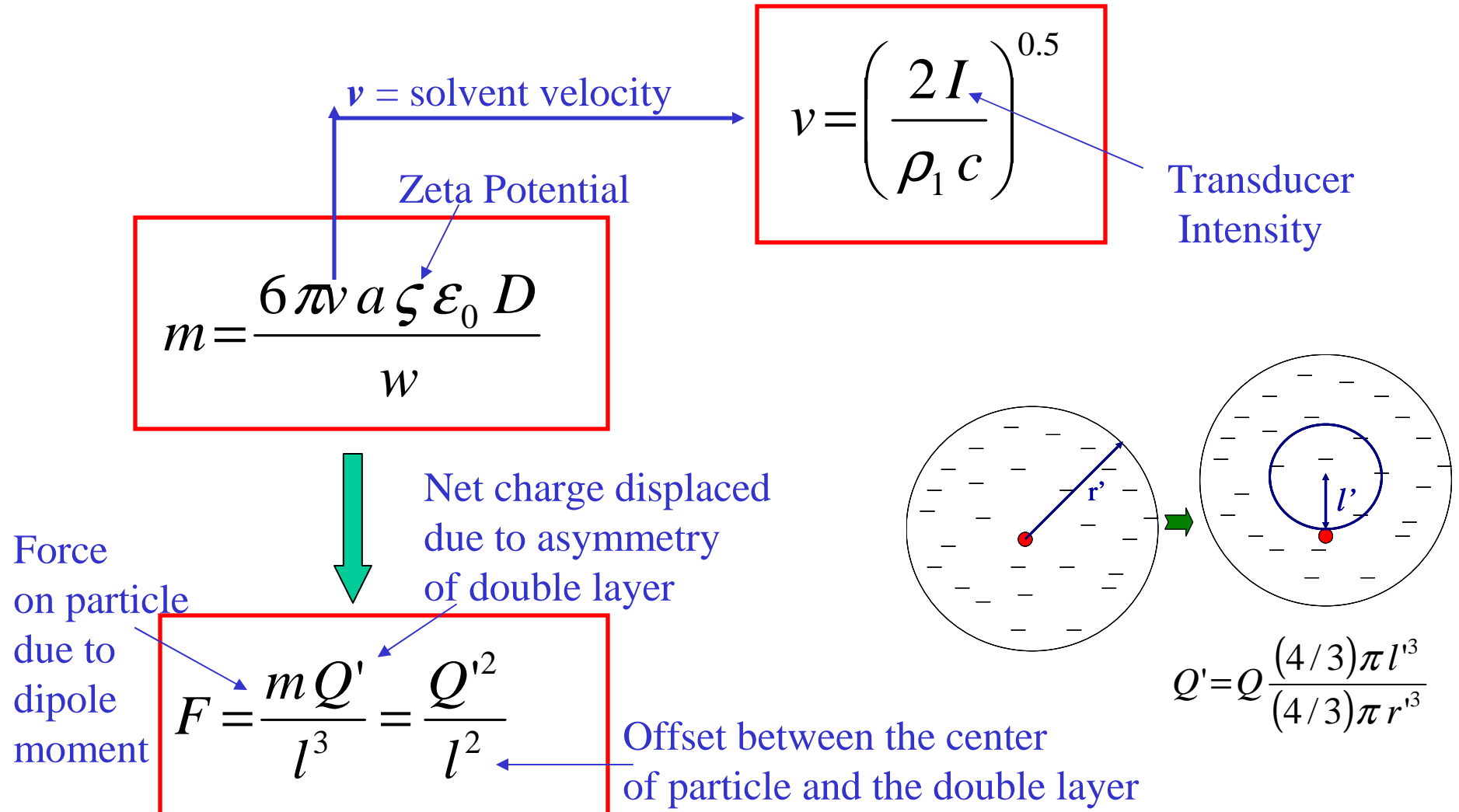
Poisson's Equation (3)

e_i is the charge on the ion, m_i is the mass of the ion, f_i is the friction coefficient, X is the electric field strength
 v_i and v_0 are velocities of ion and solvent molecules, n_i is the number of ions in a given volume
 D is the dielectric constant of the solvent

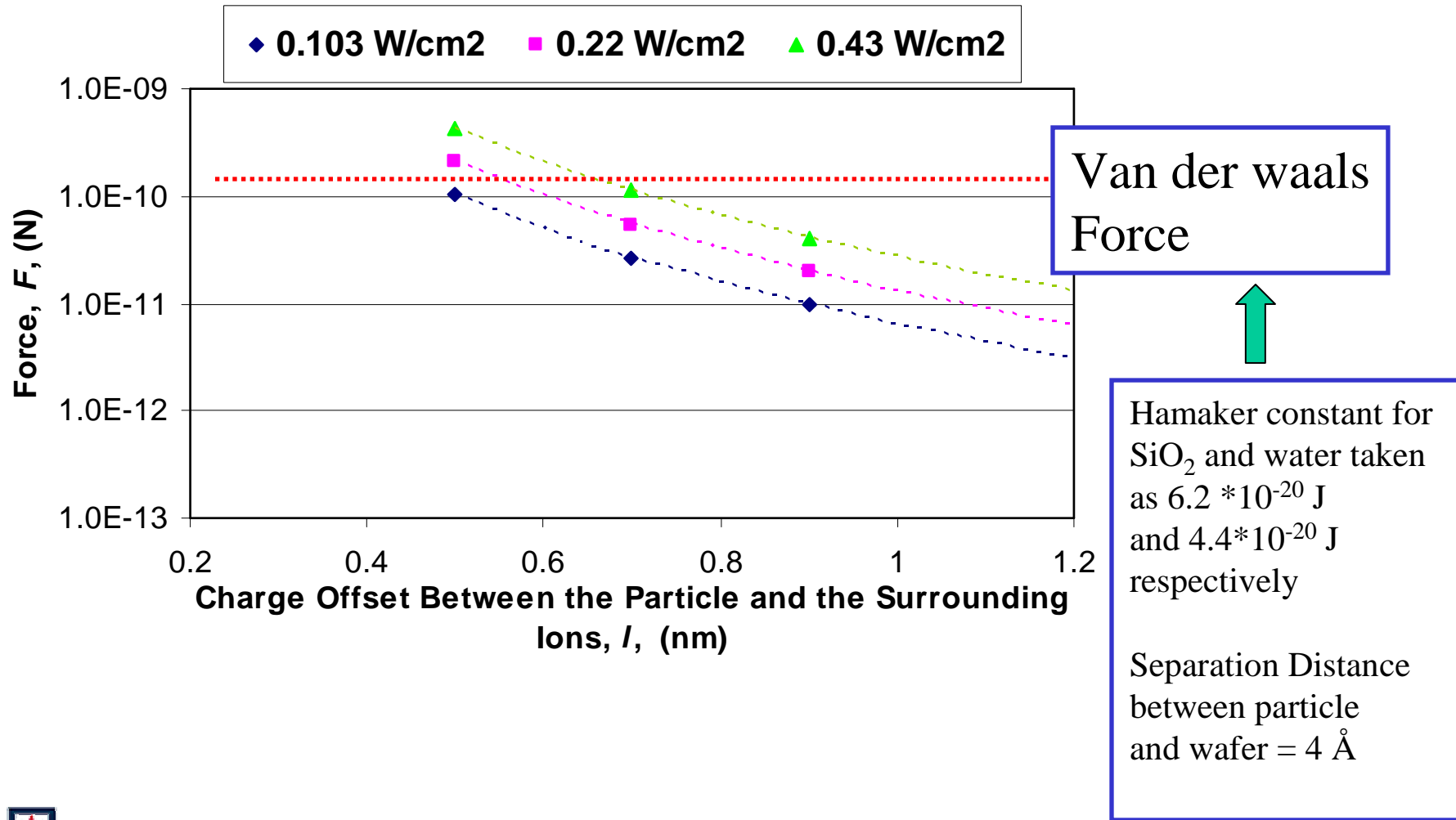


Computation of Force Due to Dipole Moment

CVP Effect

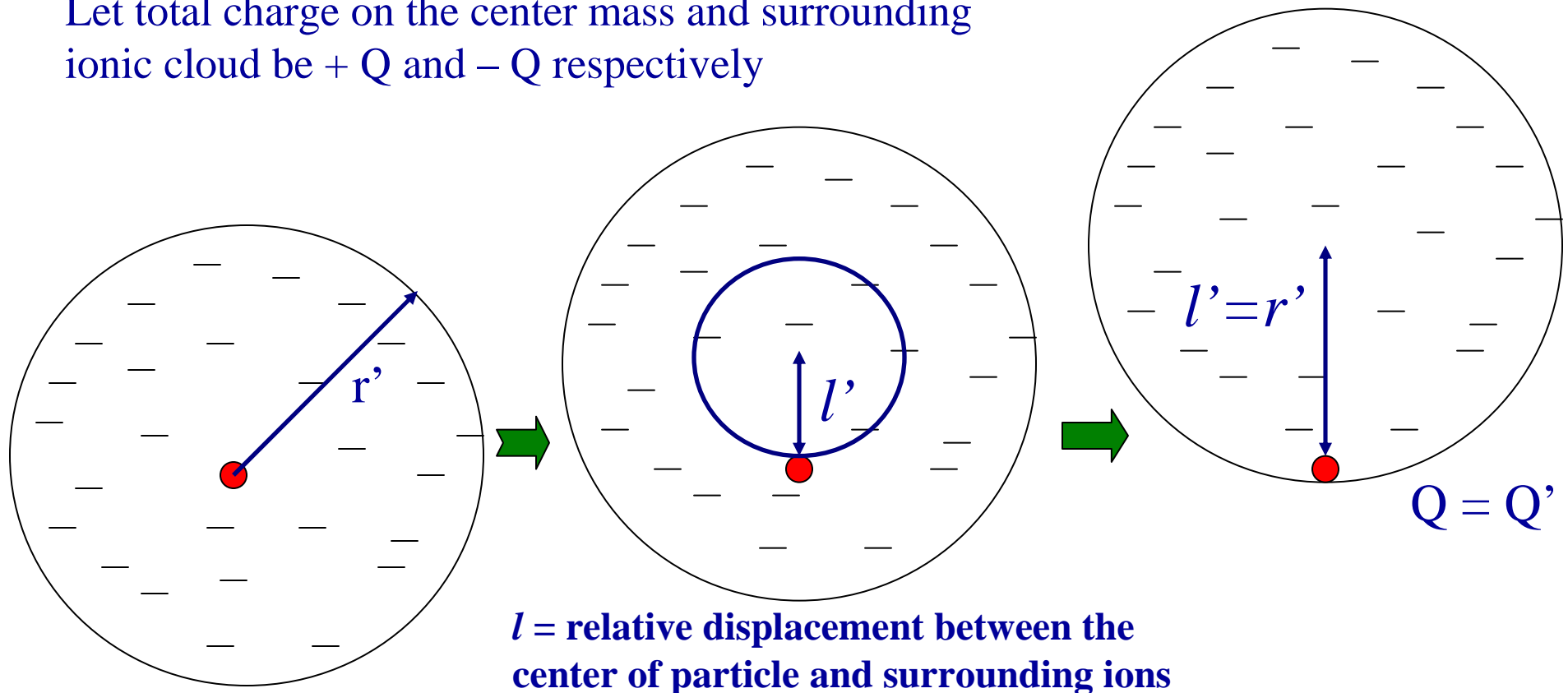


Magnitude of removal force, F , as a function of transducer power density for solution ionic strength of 10^{-4} M



HOW TO EXPLAIN THE EFFECT OF IONIC STRENGTH?

Let total charge on the center mass and surrounding ionic cloud be + Q and - Q respectively



l = relative displacement between the center of particle and surrounding ions

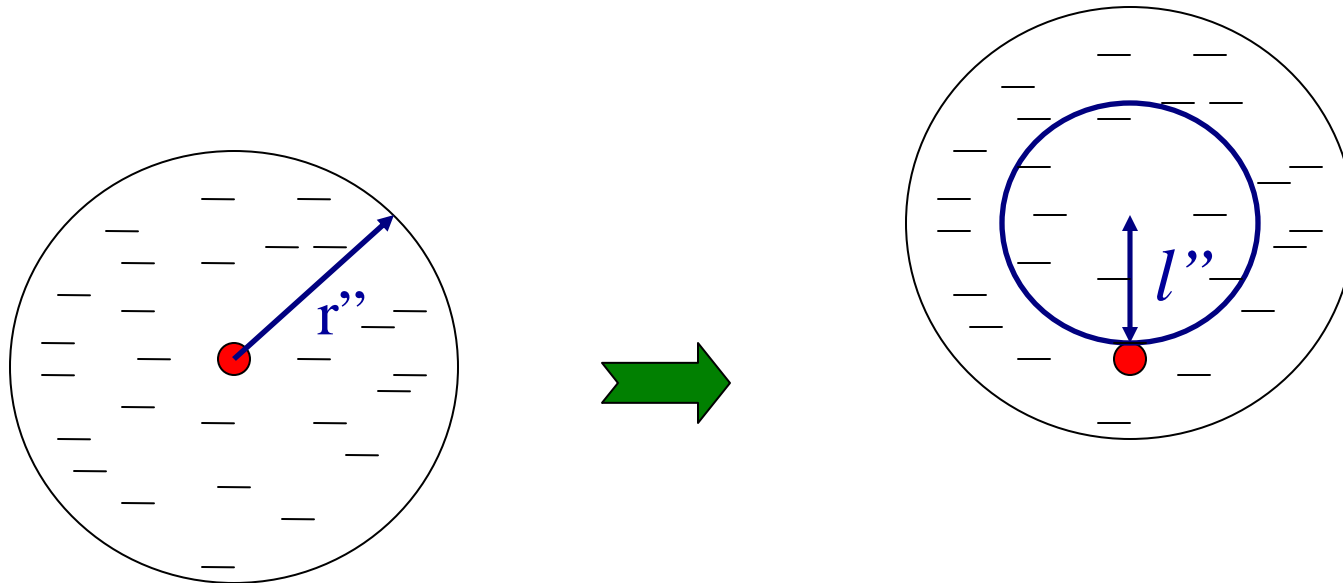
Force, $F' = Q'^2 / l'^2$ where

$$Q' = Q \frac{(4/3)\pi l'^3}{(4/3)\pi r'^3}$$

$$F' = \frac{Q'^2}{l'^2} = \frac{Q^2 l'^4}{r'^6}$$



Now, if the outer ionic cloud is compressed due to increase in solution ionic strength



Force, $F'' = Q''^2 / l''^2$

where

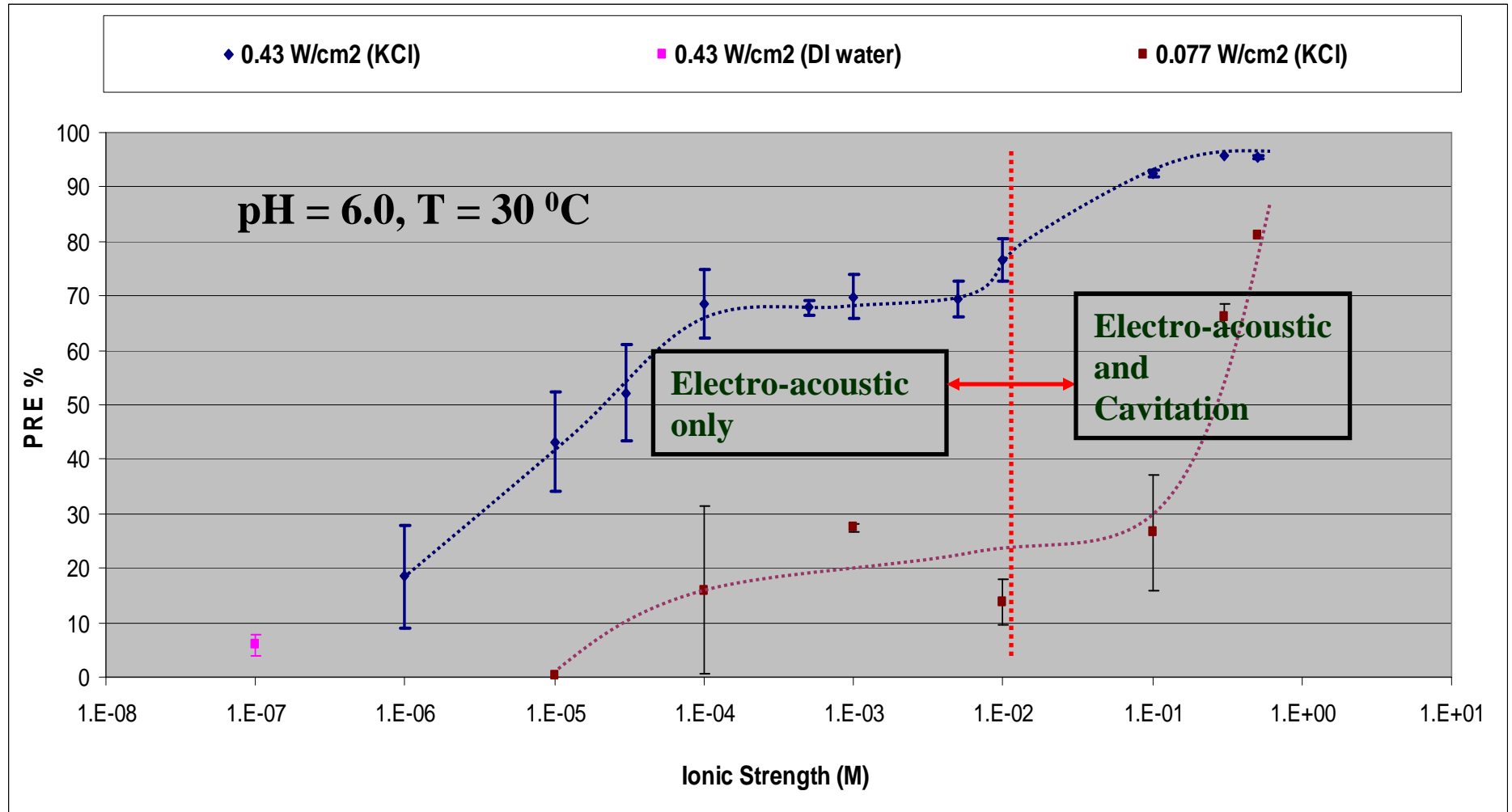
$$Q'' = Q \frac{(4/3)\pi l''^3}{(4/3)\pi r''^3}$$

$$F'' = \frac{Q''^2}{l''^2} = \frac{Q^2 l''^4}{r''^6}$$

Removal force increases with increase of ionic strength



Separation of Electro-acoustic and Cavitation Effects



Summary

- Using simple electrolyte solutions, removal of positively charged silica particles from native oxide wafers in a megasonic field can be significantly improved by increasing solution ionic strength.
- Simple calculations have shown that depending on ionic strength and power density, CVP can exert a removal force that is equal to adhesive force on the particles.
- Hydrophone and sonoluminescence studies revealed that pressure amplitude of the sound wave can be increased in KCl solutions of concentration greater than 0.01 M. This higher pressure amplitude leads to more intense cavitation which could provide additional force for removal of particles.



Future Work

- Attempt to deconvolute the effect of cavitation from electro-acoustic effect. This can be done using CO₂ that is known to suppress cavitation. The ionic strength of the solution can be modulated by controlling the amount of CO₂ dissolved.
- Carry out particle removal studies on patterned test structures. Relate particle removal to damage of structures.
- Study the effect of electrolytes for particles with varying degree of adhesion to surface. Investigate the degree of adhesion by atomic force microscopy measurements.



Acknowledgement

- Dr. Steven Verhaverbeke, Applied Materials (donation of hydrophone and wafers)
- Mark Beck, Prosys Megasonics (donation of Megasonic tank)
- Group: Nandini, Jovanny, Ashok and Raj

