Nanoparticle Gas-Phase Electrophoresis and Ambient Pressure Mass Measurement

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The 5th NIST Polymer MS Workshop

March 6, 2008

Overview

- Discuss the theory of differential electrical mobility analysis (DMA) for classifying and sizing spherical nanoparticles as an aerosol.

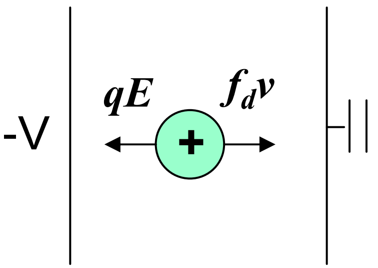
- Present a model for measuring the length distribution of nanowires using a DMA and compare with measurements for multiwalled carbon nanotubes (MWCNT).

- Discuss the theory and application of the Aerosol Particle Mass Analyzer to characterization of MWCNT.

Electrical Mobility

Equation of motion for singly charged particle:

$$m\dot{v} = f_d v - qE$$



Electrical mobility determined from steady state behavior.

$$Z_p \equiv \frac{v}{E} = \frac{q}{f_d}$$

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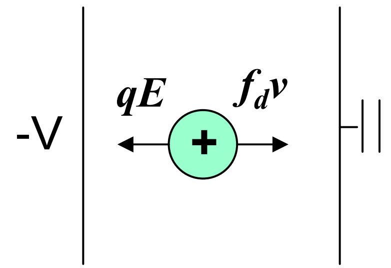


Electrical mobility determined from steady state behavior.

$$Z_p \equiv \frac{v}{E} = \frac{q}{f_d}$$

Friction coefficient for a sphere:

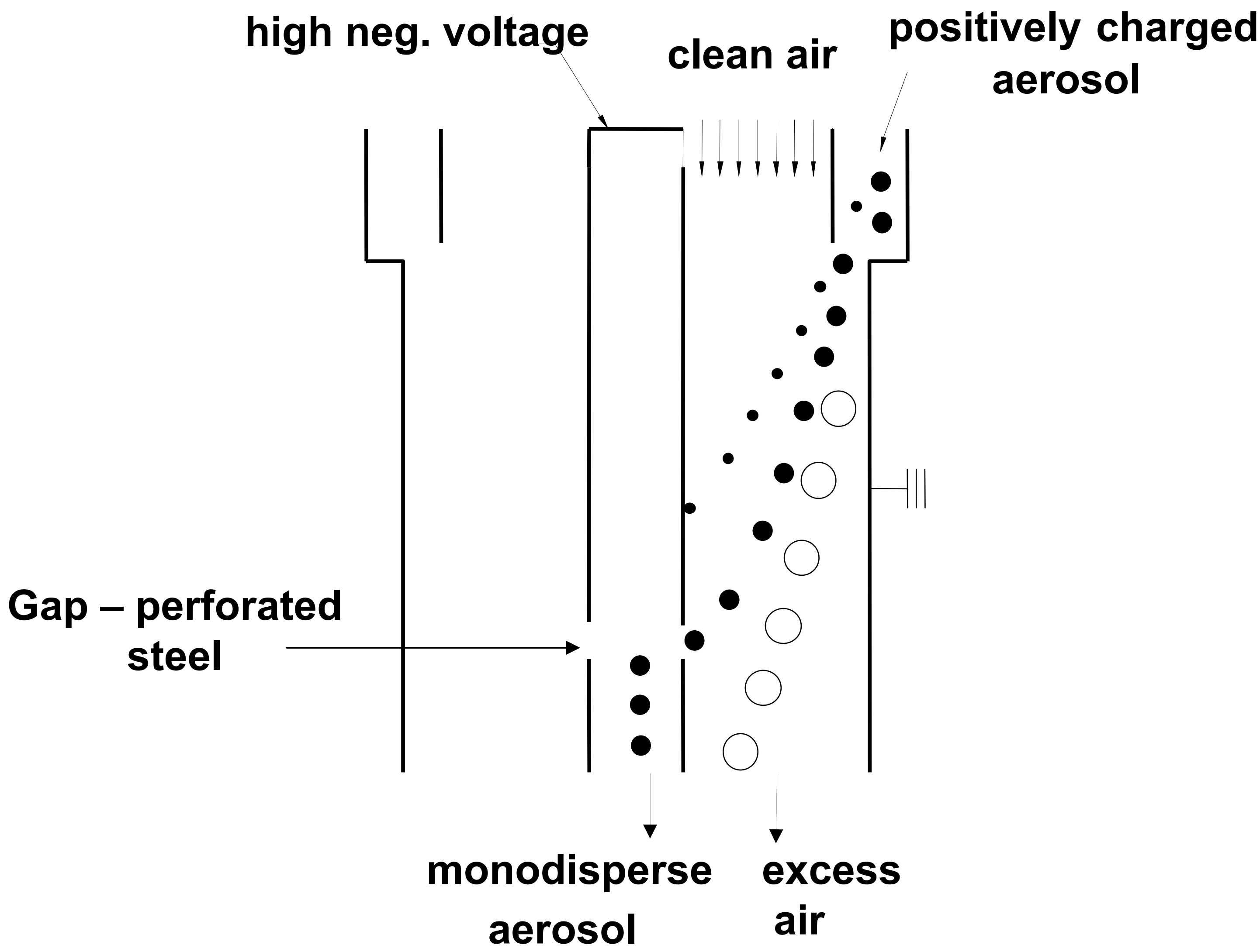
$$f_d = \frac{3\pi\eta D_m}{C(D_m)}$$

Cunningham slip correction as function of K_n=2λ/D_m

$$C(D_m) = 1 + K_n\left[A_1 + A_2\, exp(-A_3 / K_n\right]$$

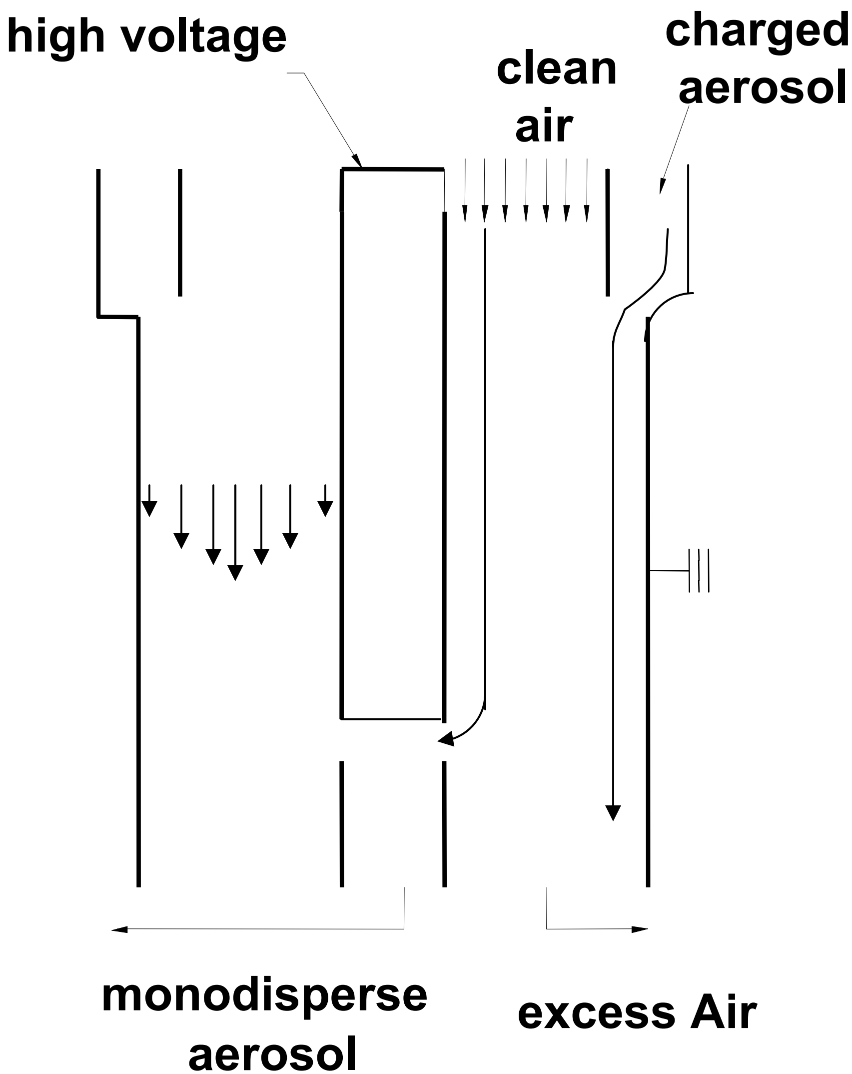
A_s from. Res. Natl. Inst. Stand. Technol., 110, 31-54 (2005).

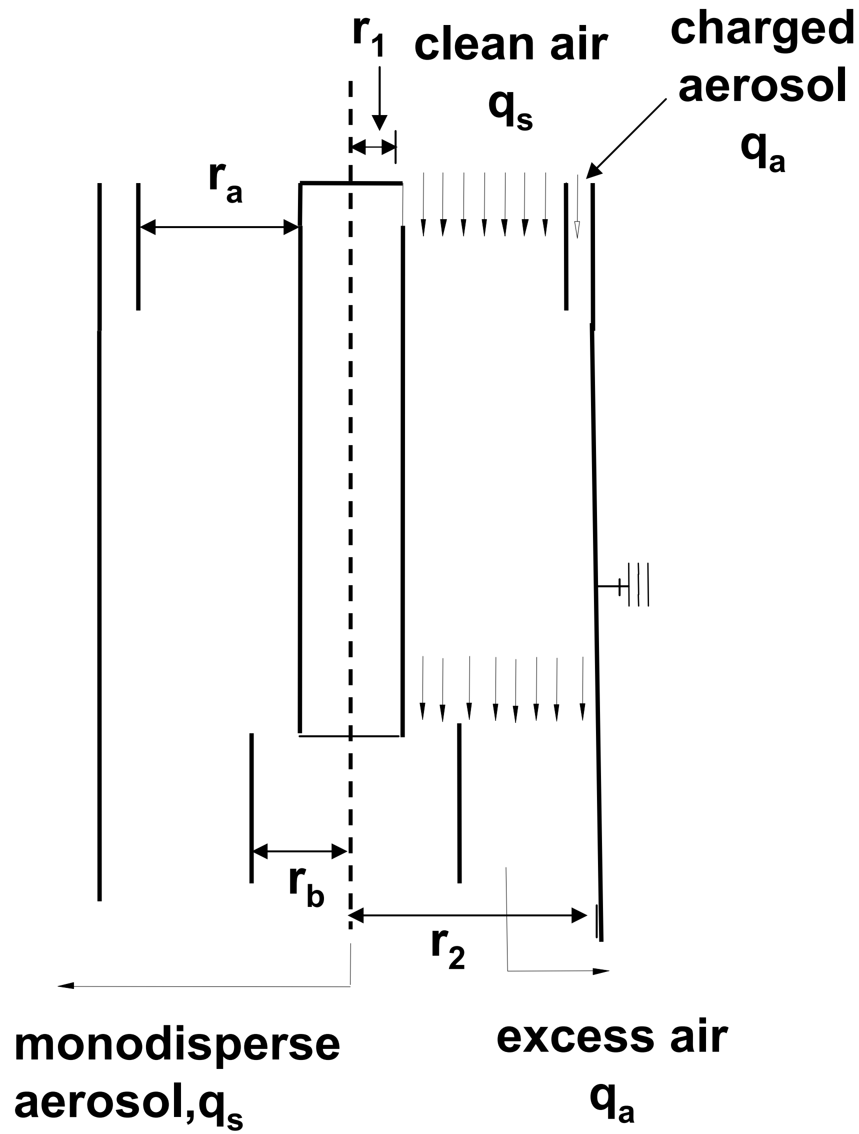
Motion of Positively Charged Aerosol Through a DMA



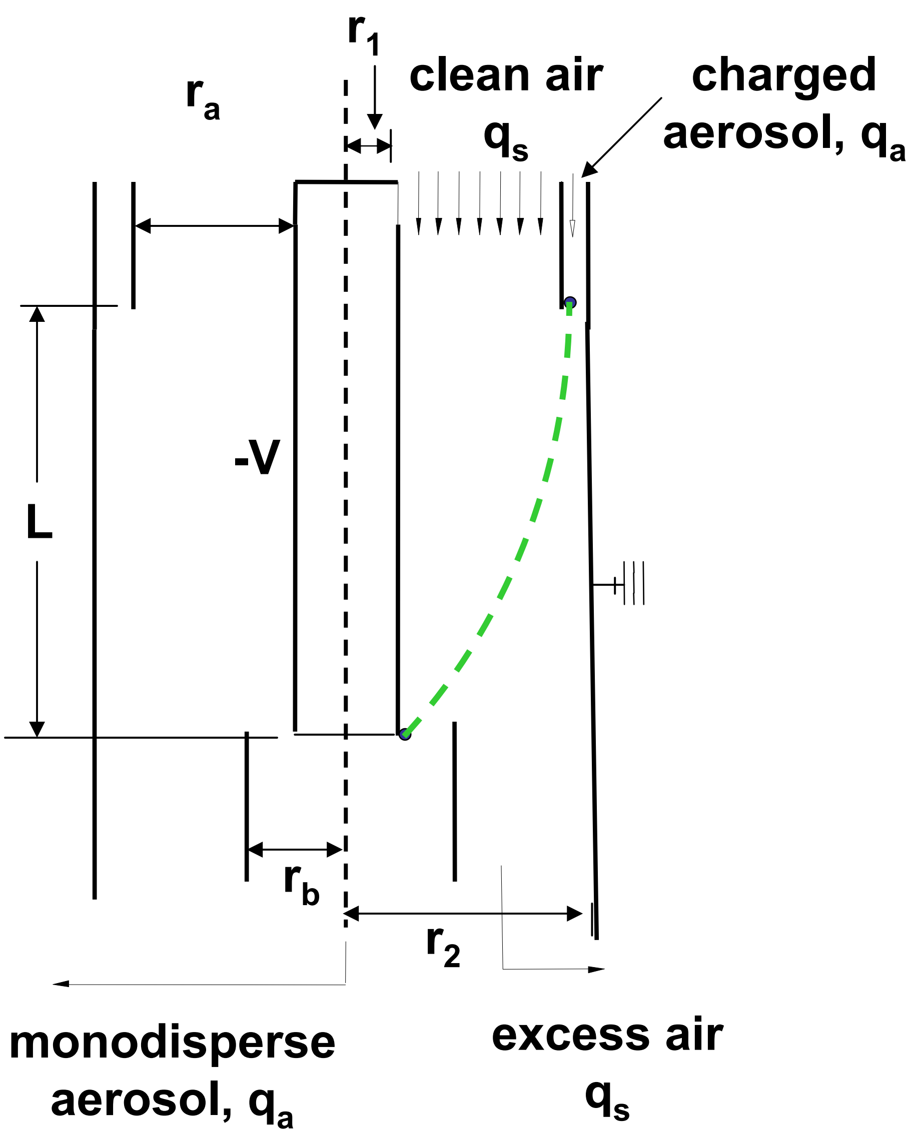
Actual DMA Flow

Idealized Plug Flow DMA





Particle Trajectory through DMA based on Plug Flow



$$\frac{dz}{dt} = U_0 = \frac{L}{\tau}$$

$$\frac{dr}{dt} = Z_p E = \frac{-Z_p V}{r \ln(r_2 / r_1)}$$

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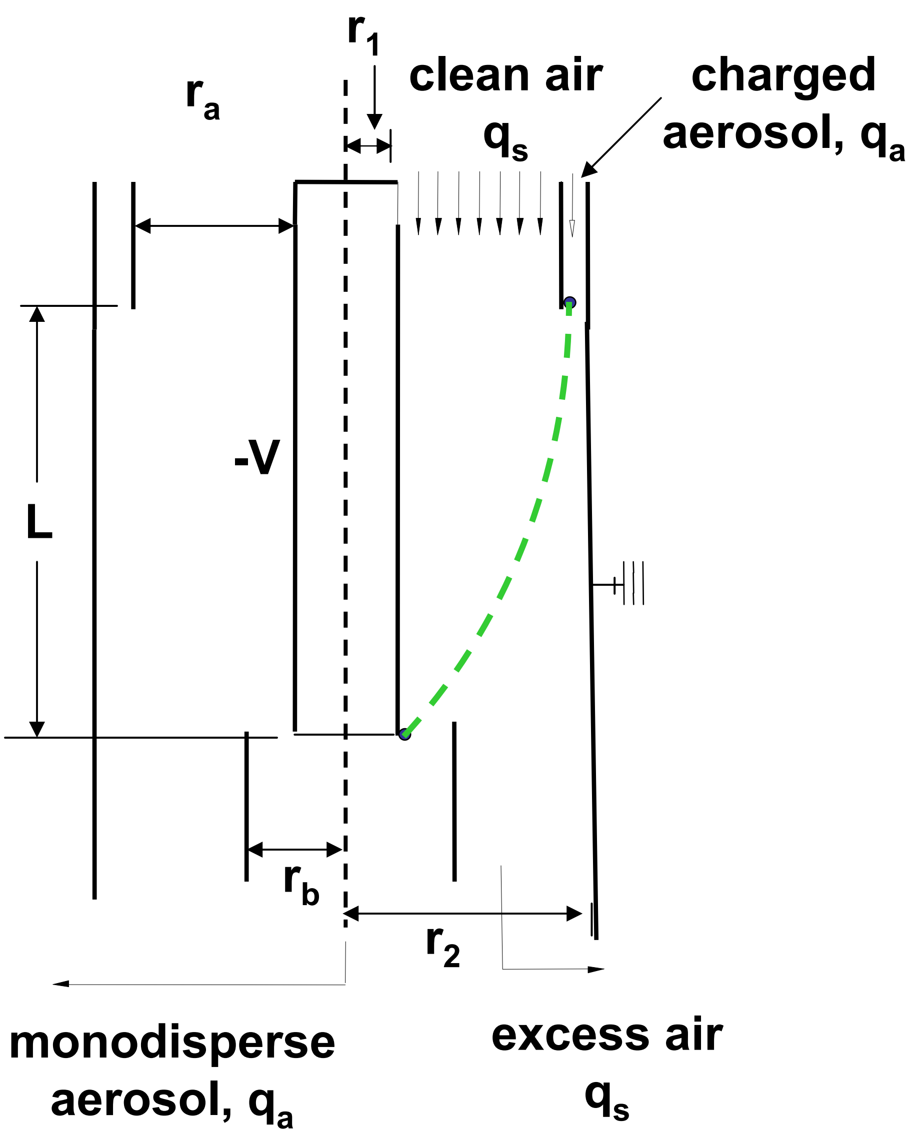


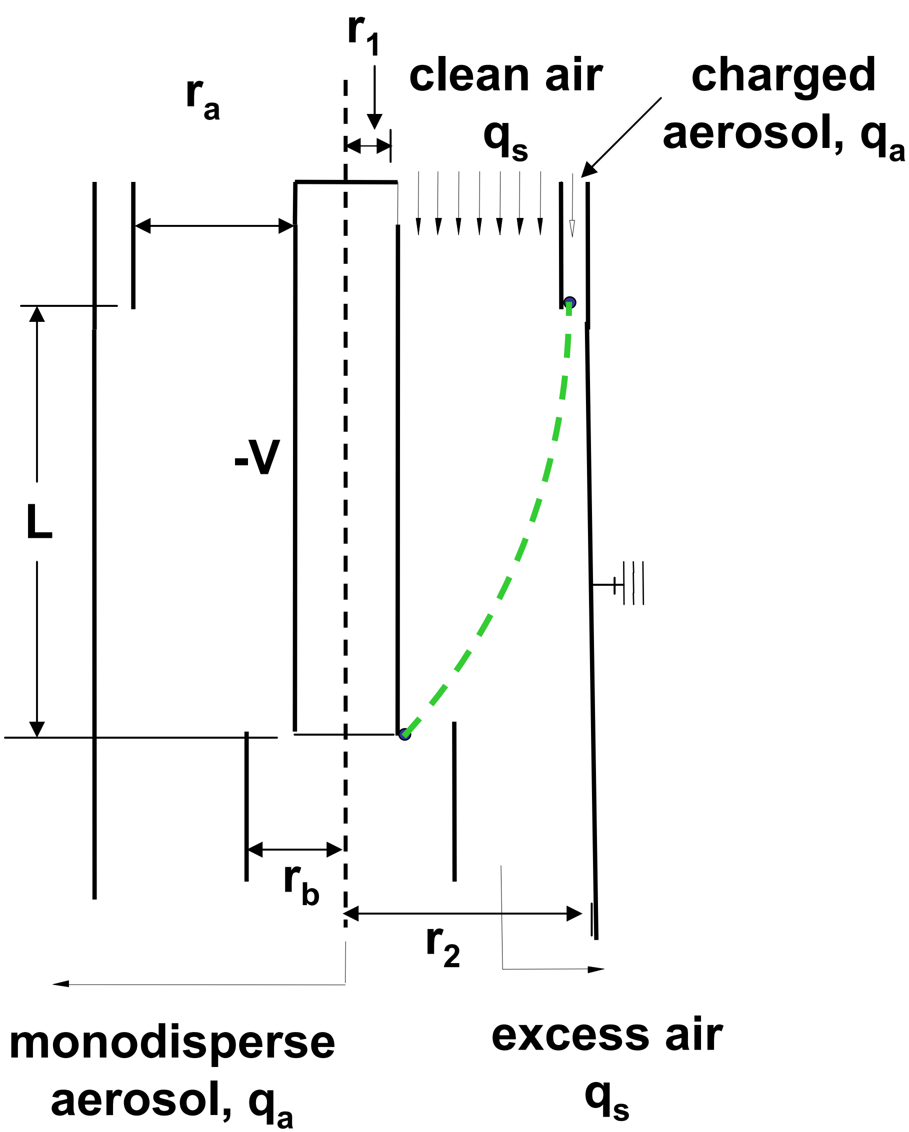
$$\frac{dz}{dt} = U_0 = \frac{L}{\tau}$$

$$\frac{dr}{dt} = Z_p E = \frac{-V}{r\,ln(r_2/r_1)}$$

Radial location at *z* = *L*

$$r(\tau) = \left[r_a^2 - \frac{2Z_p VL}{U_0\, ln(r_2/r_1)} \right]^{1/2}$$

Particle Trajectory through DMA based on Plug Flow



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For *r* = *r₁* and *r* = *r_b* at *z* = *L*)

$$r_a^2 - r_1^2 = \frac{2 Z_p V L}{U_0\, ln(r_2 / r_1)}$$

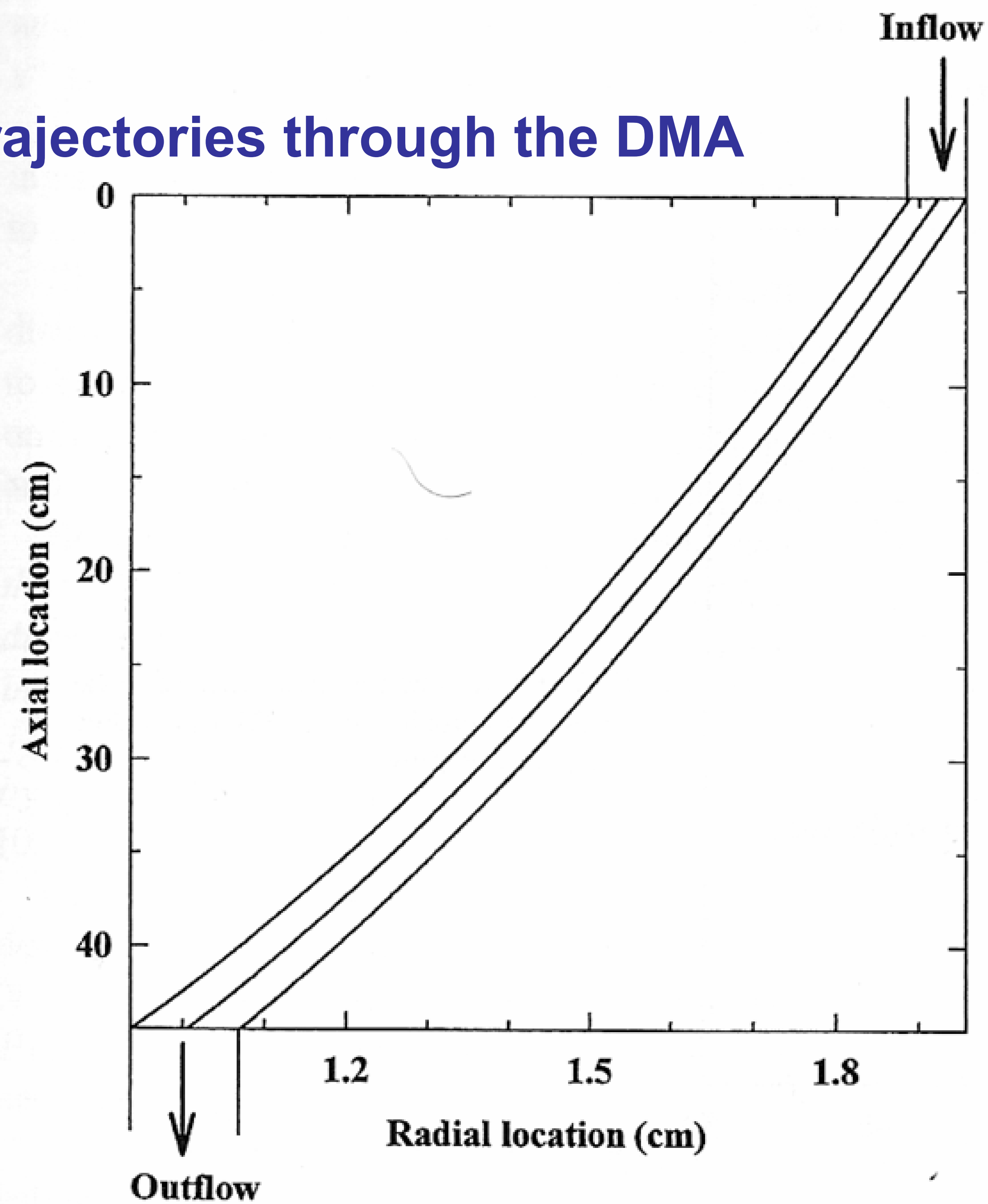
$$r_2^2 - r_b^b = \frac{2 Z_p V L}{U_0\, ln(r_2 / r_1)}$$

Limiting Trajectories through the DMA

Z_p for $r = r_1$ at $z = L$
(centroid)

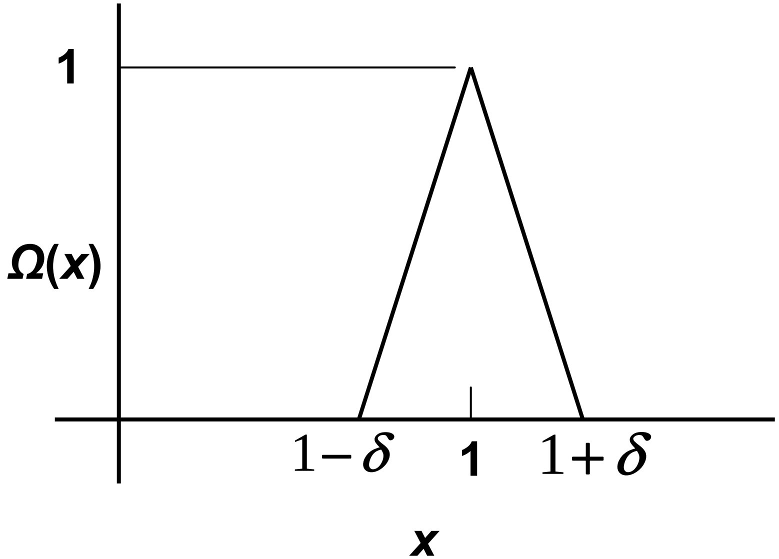
$$Z_p^* = \frac{\left(r_a^2 - r_1^2\right)U_0 \, ln\left(r_2 / r_1\right)}{2VL}$$

$$= \frac{q_s \, ln\left(r_2 / r_1\right)}{2\pi VL}$$



DMA Transfer Function

Ω = probability of particle entering the DMA with mobility Z_p will leave via the sampling flow.



$$x = \frac{Z_p}{Z_p^*}$$

$$\delta = \frac{q_a}{q_s}$$

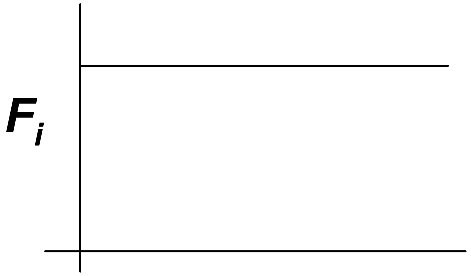
Generalization: the above form of the transfer function is independent of the detailed structure of the flow field provided the flow is laminar.

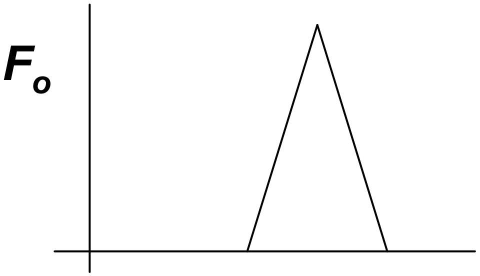
Knutson and Whitby, J. Aerosol Sci., 1975.
Hagwood et al., Aer. Sci. Tech., 1999.

Applications of the Transfer Function

Size distribution passing through DMA

$$F_o\left(Z_p\right)=\left(\frac{dN^+}{dZ_p}\right)_o = F_i\left(Z_p\right)\Omega\left(Z_p\right)$$





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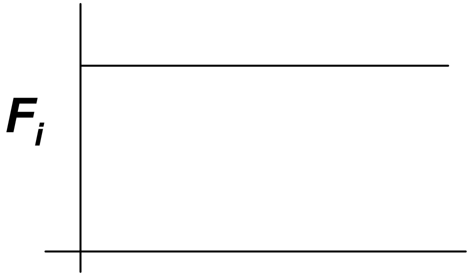
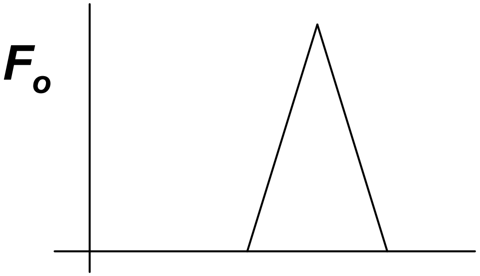
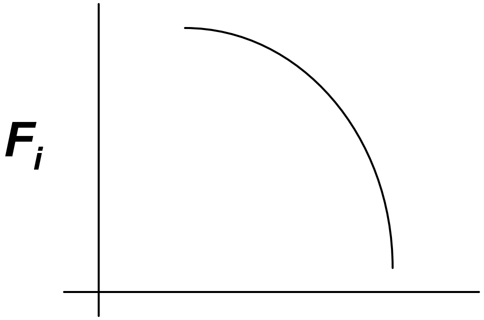
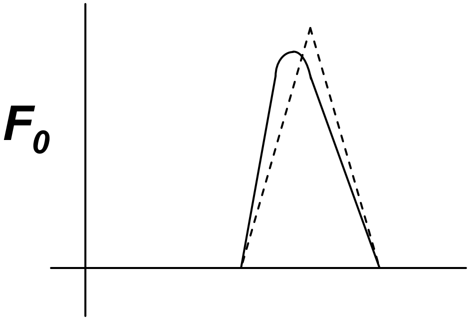
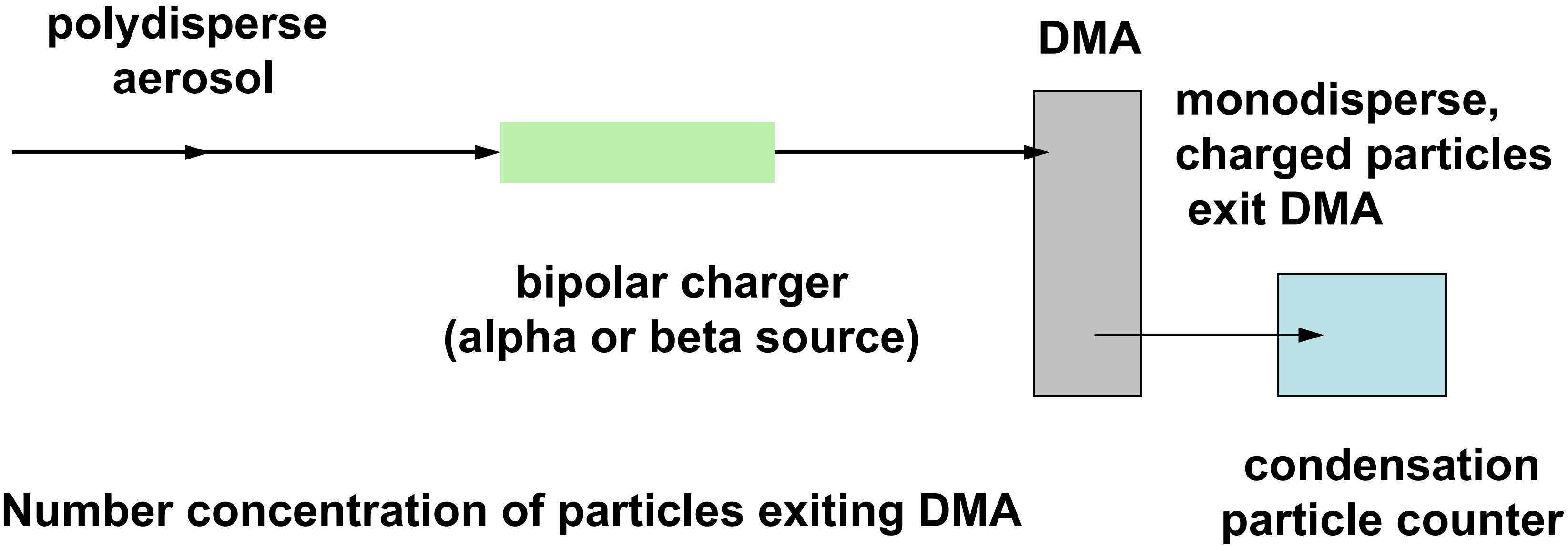








Measurement System



Number concentration of particles exiting DMA

$$N(V) = \delta \int_0^\infty \Omega(Z_p V) F_i(Z_p) dZ_p$$

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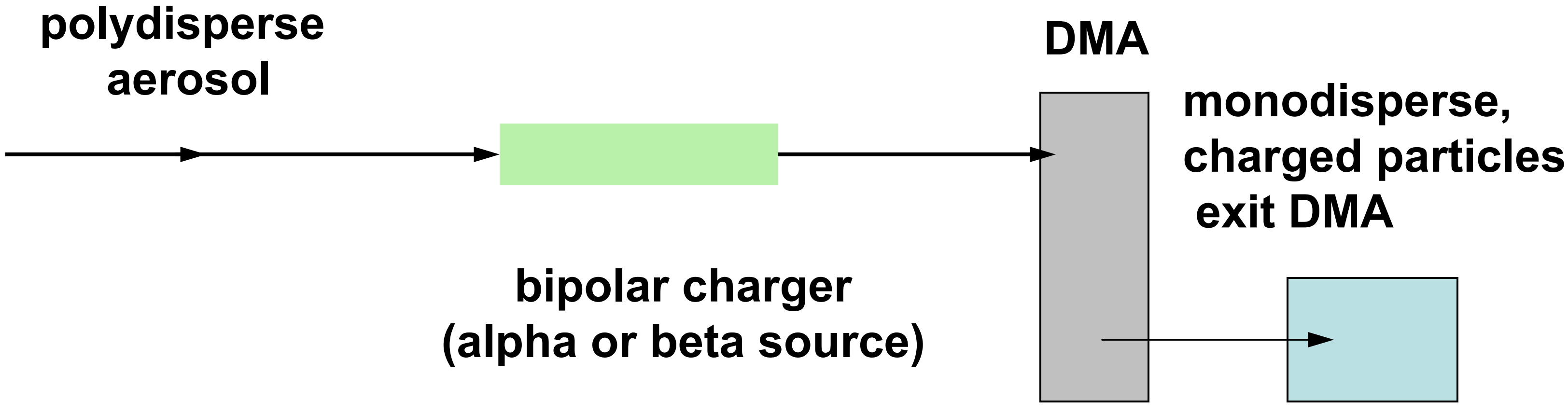


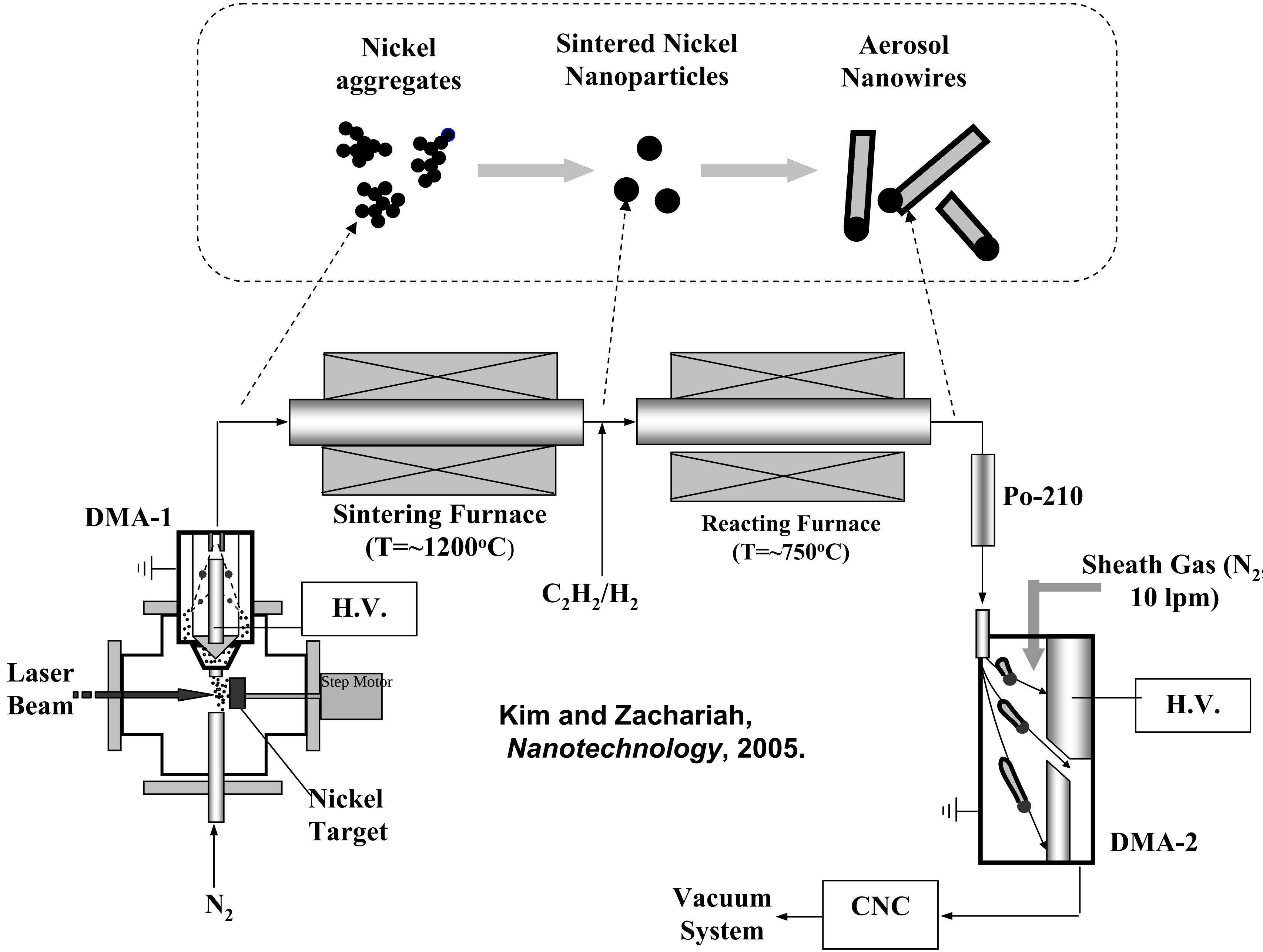
Number concentration of particles exiting DMA

$$N(V) = \delta \int_0^\infty \Omega(Z_p, V) F_i(Z_p) dZ_p$$

Often one is interested in the diameter size distribution of the polydisperse aerosol

$$G(D_p) = \frac{dN}{dD_p} = \frac{dN^+}{dZ_p}\frac{dZ_p}{dD_p}\frac{1}{P(D_p)} = F(Z_p)\frac{dZ_p}{dD_p}\frac{1}{P(D_p)}$$

Generation of Carbon Nanotubes

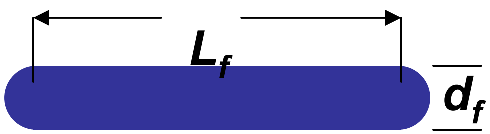


Kim and Zachariah, *Nanotechnology*, 2005.

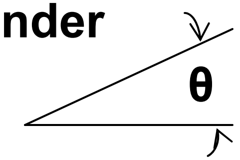
Angle Dependence of Electrical Mobility of Nanowire

Dahneke's expression for the friction coefficient for a cylindrical particle with hemispherical ends in free molecular regime:

$$f_D = \frac{\pi \eta d_f^2}{2\lambda}\left[\left(\beta f + k_2\right) + \beta k_3 \sin^2\theta\right]$$



$\beta = L_f/d_f$, f is the fraction of diffuse gas collisions with the cylinder



and where $k_2 = \frac{\pi f}{6} + \frac{4}{3}$ $\quad k_3 = \left(2 - \frac{6-\pi}{4} f\right)$

We first consider the limiting cases of a totally aligned nanowire and a randomly oriented nanowire.

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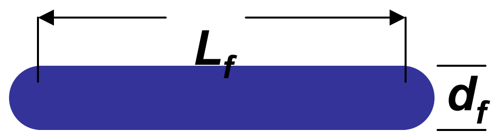

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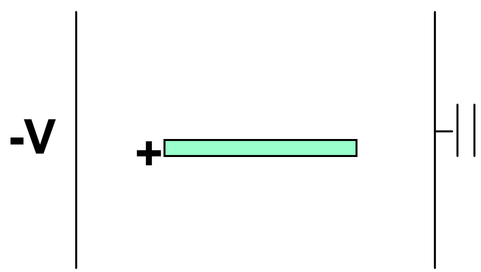
We first consider the limiting cases of a totally aligned nanowire and a randomly oriented nanowire.

For a nanowire oriented by the electric field we have

$$f_D = \frac{\pi \eta d_f^2}{2\lambda}\left(\beta f + k_2\right)$$



Relation between *L* and D_m for Nanowire Aligned by the Electric Field

Mobility of oriented cylinder

$$Z_{cyl} = \frac{2\lambda q}{\pi \eta d_f^2 \left(\beta f + k_2\right)}$$

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Mobility of oriented cylinder

$$Z_{cyl} = \frac{2\lambda q}{\pi\eta d_f^2\left(\beta f + k_2\right)}$$

The mobility diameter, D_m, is the diameter of a sphere with the same mobility as the cylinder.

$$Z_p\left(D_m\right) = \frac{qC\left(D_m\right)}{3\pi\eta D_m} = Z_{cyl}$$

Relation between L and D_m for Nanowire Aligned by the Electric Field

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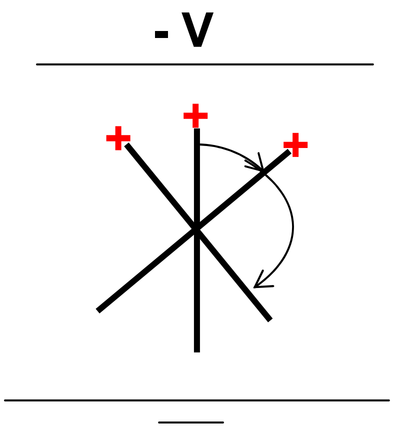
The length of the nanowire, L_f, is then obtained. Note that the DMA in essence measures D_m .

$$L_f = \frac{6\lambda D_m}{f d_f C\left(D_m\right)} - \frac{d_f}{f} k_2$$

Relation between *L* and D_m for Nanowire Randomly Oriented

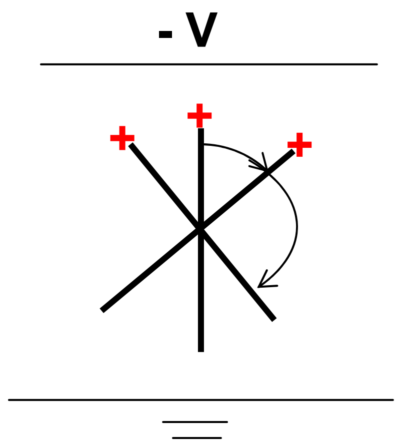


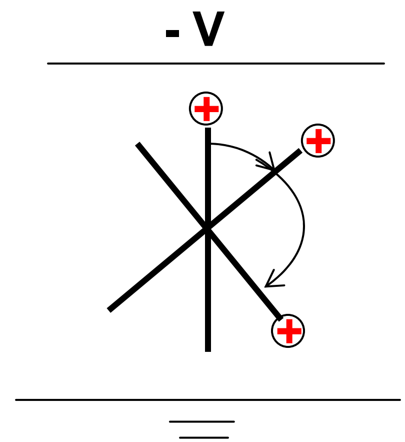
Case 1 – conducting wire

$$-\pi / 2 < \theta < \pi / 2$$

Assume conduction time fast compared to 1μs for β=5 and d_f=15 nm.

Relation between L and D_m for Nanowire Randomly Oriented





Case 1 – conducting wire

$$-\pi / 2 < \theta < \pi / 2$$

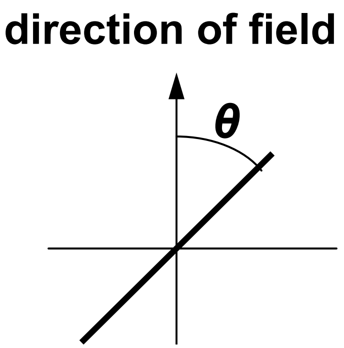
Case 2 – charge trapped on Ni catalyst

$$-\pi < \theta < \pi$$

Randomly Oriented Cylinder

Probability distribution for randomly oriented cylinder:

$$P(\theta)d\theta = c\,\sin\theta d\theta$$



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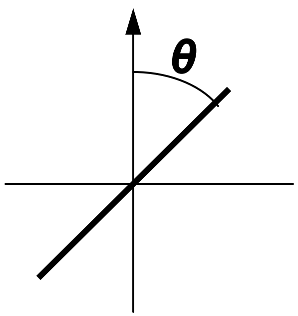


Probability distribution for randomly oriented cylinder:

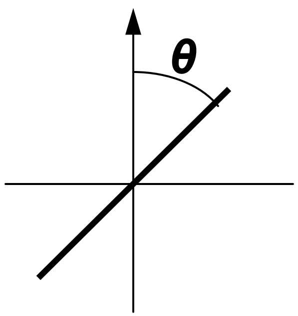
$$P(\theta)d\theta = c\,\sin\theta d\theta$$

Orientation averaged mobility Case 1:

$$\overline{Z}_{cylinder} = \frac{\int_0^{\pi/2} Z_{cylinder} P(\theta) d\theta}{\int_0^{\pi/2} P(\theta) d\theta} = \int_0^{\pi/2} \frac{k_1 c\,\sin\theta}{L\left(1 - a^2 \cos^2\theta\right)} d\theta \Big/ \int_0^{\pi/2} c\,\sin\theta d\theta$$

Randomly Oriented Cylinder

Probability distribution for randomly oriented cylinder:



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Equating Z_{sphere} to $Z_{cylinder}$, one finds an implicit relationship between β (or L_f) and D_m

$$\frac{D_m}{C(D_m)} = \frac{d_f^2 a\left(\beta\left(f+k_3\right)+k_2\right)}{3\lambda \, ln\left[\left(1+a\right)/\left(1-a\right)\right]} \qquad \text{where} \qquad a = \left(\frac{\beta k_3}{\beta\left(f+k_3\right)+k_2}\right)$$

Explicit Relation between *L and Dm* in Limit of Large β

Aligned with Electric Field

$$L_f = \frac{6\lambda D_m}{f d_f C(D_m)}$$

Randomly oriented

$$L_f = \frac{6\kappa\lambda D_m}{d_f C(D_m)}$$

Explicit Relation between *L and Dm* in Limit of Large β

Aligned with Electric Field

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Randomly oriented

$$L_f = \frac{6\kappa\lambda D_m}{d_f C(D_m)}$$

Also we find:

$$L_{aligned} = \frac{1}{f\kappa} L_{random}$$

$$\kappa = \frac{ln[(1+a)/(1-a)]}{2a(f+k_3)} \qquad a = \left[\frac{k_3}{f+k_3}\right]^{1/2} \qquad k_3 = 2 - f\frac{6-\pi}{4}$$

For *f* = 0.9, we find:

$$L_{aligned} = 1.88 L_{random}$$

Explicit Relation between *L and Dm* in Limit of Large β

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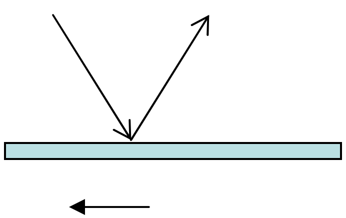
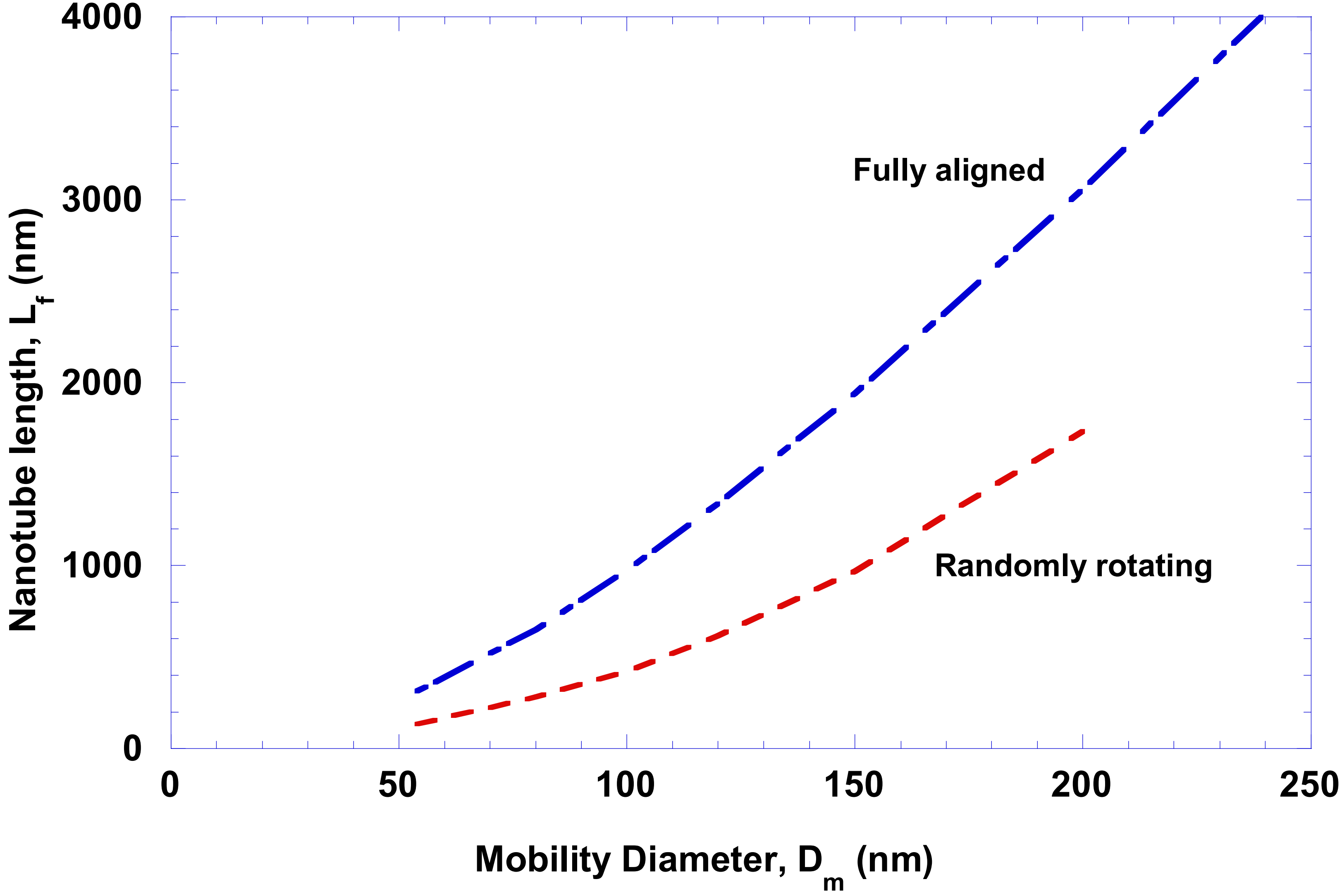
For *f* = 0.9, we find: $L_{aligned} = 1.88 L_{random}$

For *f* = 0.1, we find: $L_{aligned} = 9.05 L_{random}$



Might this apply to single walled nanotubes?

Comparison of Predicted Nanowire Length as a Function of Mobility Diameter for $d_f = 15$ nm



Orientation Probability of Charged Nanowire in Electric Field

The probability of alignment at polar angle *θ*, *P*(*θ*)d*θ* is given by Boltzmann's law:

$$P(\theta)d\theta = c\,exp[-\Phi_t / kT]\,sin\,\theta d\theta$$

$\Phi(\theta)$ is the change in the potential energy as the nanowire rotates by θ. There are two contributions to the potential energy:

Free Charge Φ_q

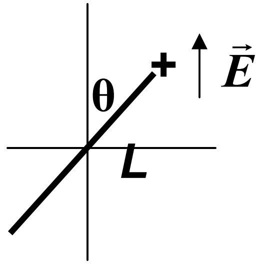
Polarizability Φ_P

Free Charge Potential

$$\Phi_q(\theta) = \int_0^\theta \tau \, d\theta$$

$$\vec{F} = q\vec{E}$$

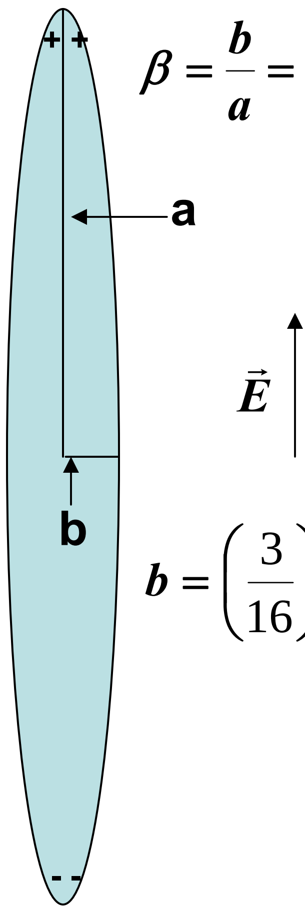
$$\tau = \left|\vec{r} \times \vec{F}\right| = \frac{L}{2} qE_z \sin\theta$$



$$\Phi_q(\theta) = \frac{L}{2} qE_z \int_0^\theta \sin\theta d\theta = \frac{L}{2} qE_z (1 - \cos\theta)$$

Polarizability Potential for a Prolate Spheroid

$$\Phi_p = -1/2\left(2\pi\varepsilon_0\right)\Sigma E^2\left[\frac{\cos^2\theta}{\frac{1}{\varepsilon_k - 1} + \kappa_1} + \frac{\sin^2\theta}{\frac{1}{\varepsilon_k - 1} + \kappa_2}\right]$$



$$\beta = \frac{b}{a} = \frac{L}{d_f}$$

$$b = \left(\frac{3}{16}\right)^{1/3} d_f$$

ε_0 = permittivity in free space
ε_k = dielectric constant of nanowire
Σ = volume of prolate spheroid   same as that of the cylinder

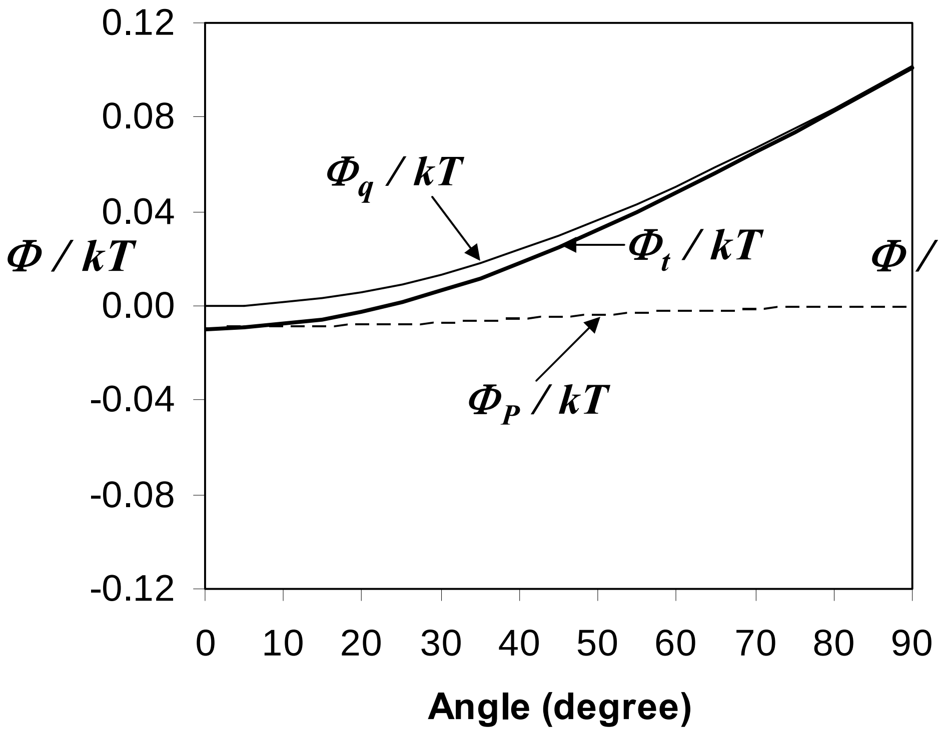
$$\kappa_1 = \frac{1}{\beta^2 - 1}\left[\frac{\beta}{\sqrt{\beta^2 - 1}} \ln\left(\beta + \sqrt{\beta^2 - 1}\right) - 1\right] \rightarrow \frac{\ln\beta}{\beta^2}$$

$$\kappa_2 = \frac{\beta}{2\left(\beta^2 - 1\right)}\left[\beta - \frac{1}{\sqrt{\beta^2 - 1}} \ln\left(\beta + \sqrt{\beta^2 - 1}\right)\right] \rightarrow const$$

Combined Potential Energy for Conducting Nanowire

$$\Phi_t = \frac{L}{2} qE(1 - \cos\theta) - 1/2(2\pi\varepsilon_0)\Sigma E^2 \left[\frac{\cos^2\theta}{\kappa_1} + \frac{\sin^2\theta}{\kappa_2}\right]$$

The first term dominates for smaller *E* and *L* (*or β*) as shown below for 15 nm diameter nanowire:



***β* = 8, *V* = 465**

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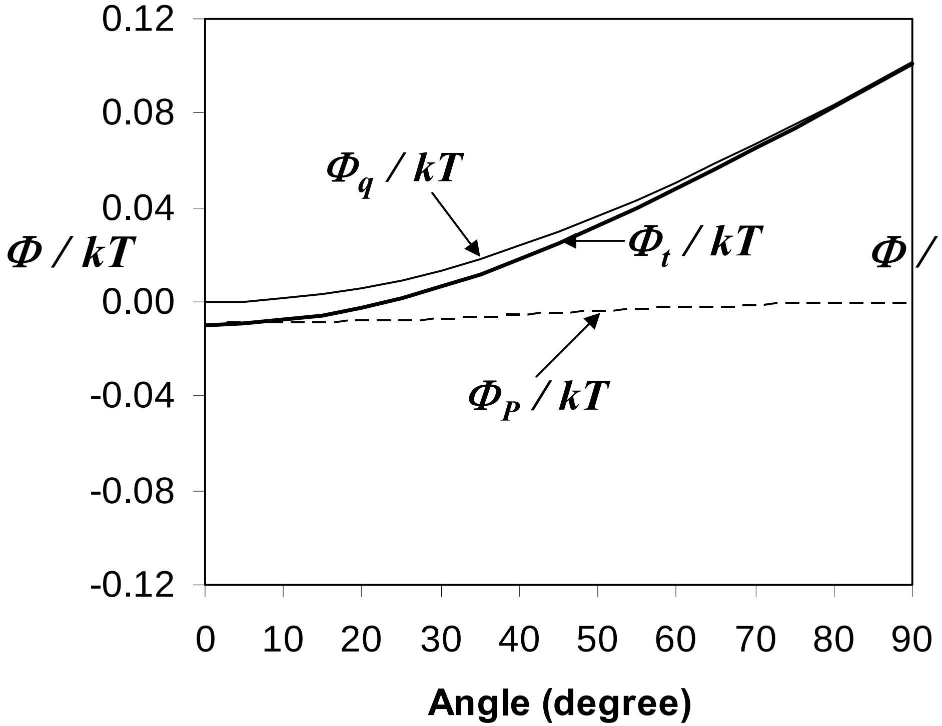

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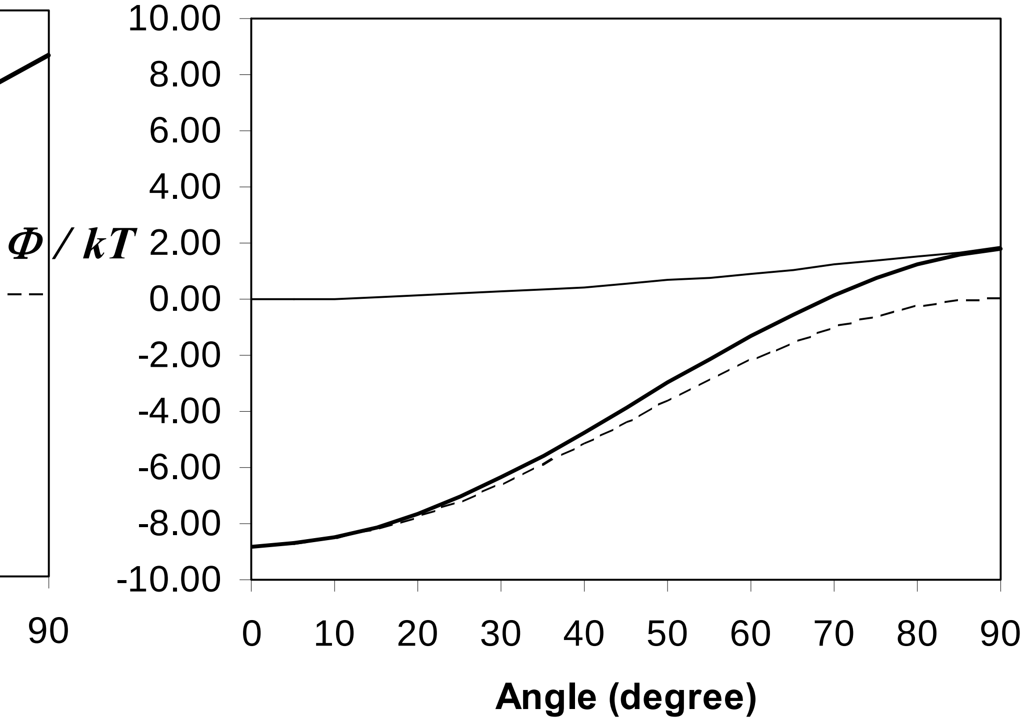
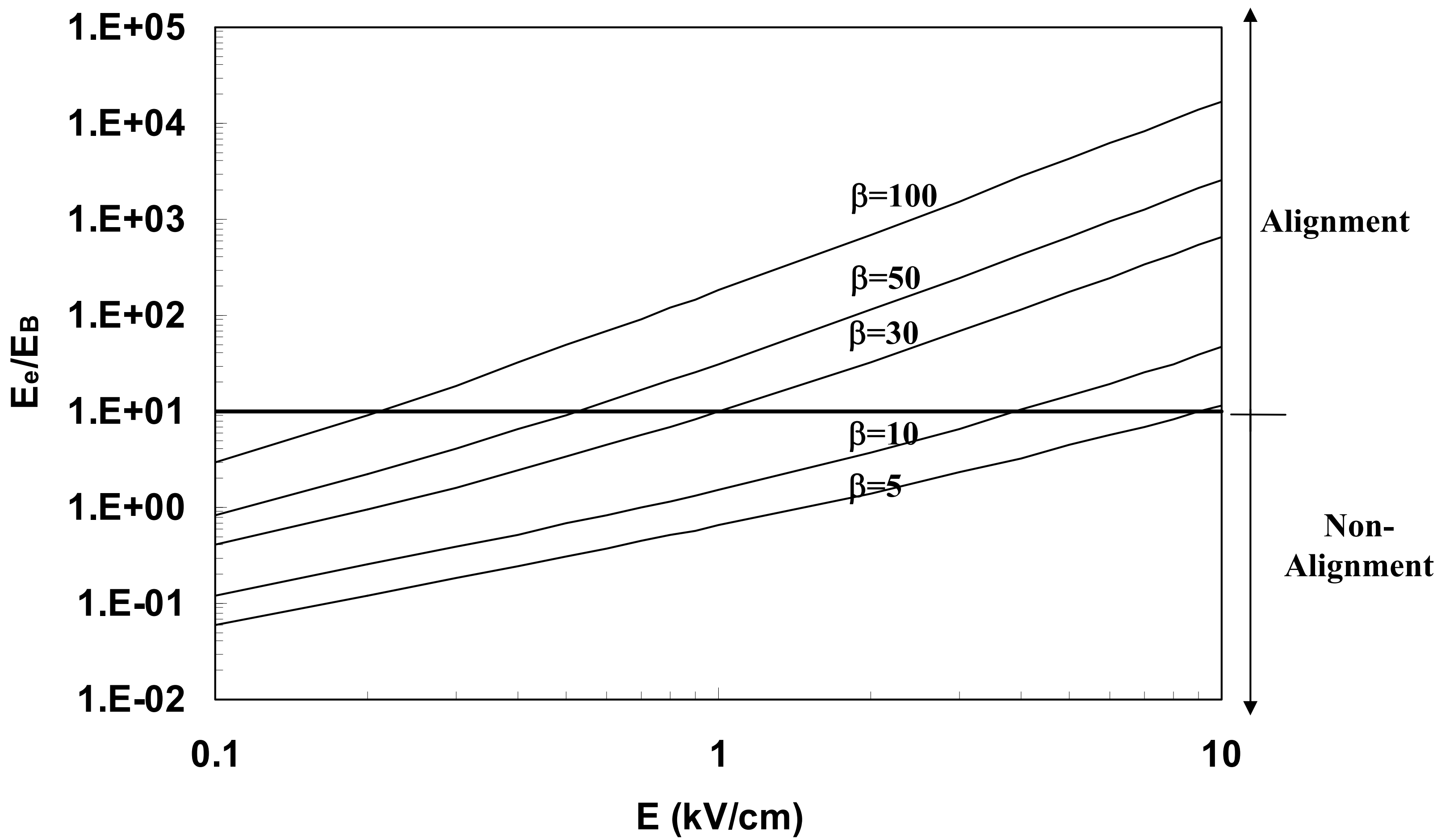


β = 42, *V* = 1614

Tendency of Nanowires to Align in the Electric Field

$$\frac{E_e}{E_B} = \left(\Phi_t\left(\pi / 2\right) - \Phi_t\left(0\right)\right) / kT$$



Orientation Probability

The orientation probability:

$$P(\theta)d\theta = c\,exp\left(-\Phi_t / kT\right) sin\,\theta d\theta$$

It is convenient to express the integral in terms of $x = cos\,\theta$

$$P_1(x)dx = c\,exp\left(-\alpha_1\beta E + \alpha_3\beta E^2\right)exp\left(\alpha_1\beta Ex + \alpha_2\beta E^2 x^2\right)dx$$

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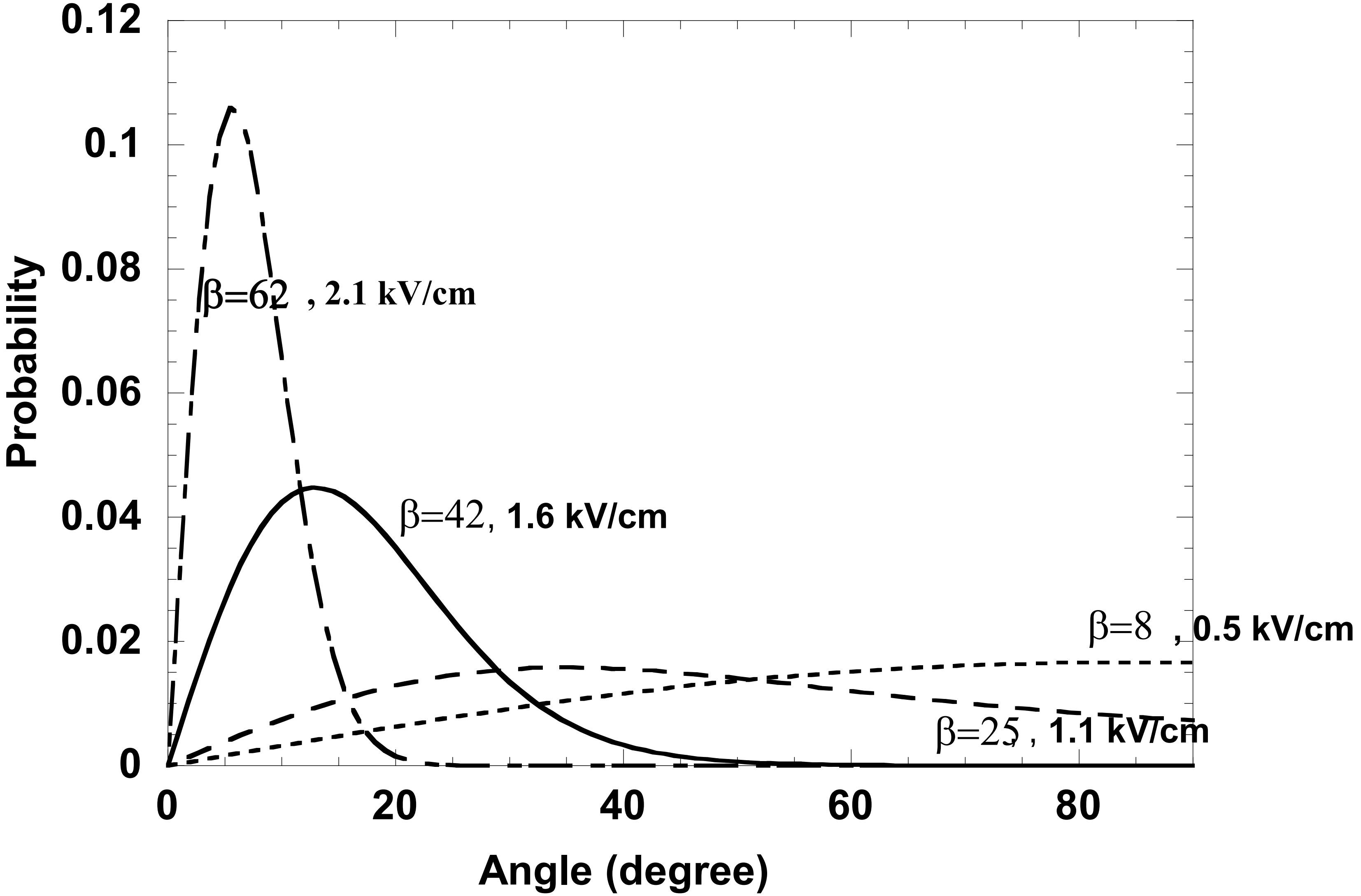
After completing the square, the normalization constant *c is obtained as:*

$$c(\beta, E) = \frac{\left(\frac{4E^2\alpha_2\beta}{\pi}\right)^{1/2} exp\left[\frac{\alpha_1{}^2\beta}{4\alpha_2} + \beta E\left(\alpha_1 - \alpha_3 E\right)\right]}{Erfi\left[\frac{(\alpha_1 + 2\alpha_2 E)\beta^{1/2}}{2\alpha_2{}^{1/2}}\right] - Erfi\left[\frac{\alpha_1\beta^{1/2}}{2\alpha_2{}^{1/2}}\right]} = c_1(\beta, E)\,exp\left[\beta E\left(\alpha_1 - \alpha_3 E\right)\right]$$

where $Erfi(x) = Erf(ix) = \frac{2}{\pi}\int_0^{ix} e^{-t^2}\,dt$

Orientation Probability Dependence on β



Orientation Averaged Mobility

$$\overline{Z_p} = \int_0^{\pi/2} Z_p(\theta) P(\theta) d\theta = \int_0^1 Z_p(x) P_1(x) dx$$

The average mobility is a function of the electric field and aspect ratio for a fixed diameter of the cylinder.

$$\overline{Z_p(E,\beta)} = c_1(\beta,E) k_2(\beta) \int_0^1 \frac{exp(\alpha_1 \beta E x + \alpha_2 \beta E^2 x^2)}{\left(1 - a^2(\beta) x^2\right)} dx$$

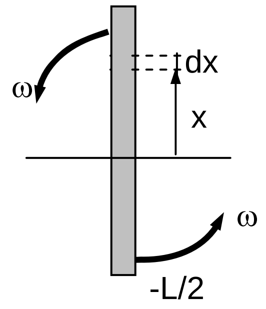
Estimated Rotation Time for Nanowire

Brownian rotation equation:

$$\overline{\theta^2} = \frac{2k_B t_r}{R_d}$$

where R_d = the rotational friction coefficient.

R_d is estimated from the torque exerted by the gas on the rotating nanowire.



$$R_d = \frac{k\pi\eta d_f L_f^{\,3}}{48 \cdot \lambda}$$

with $k \approx 2.0$

The estimated time for a 2π rotation is ≤ 30 ms for β up to 150.

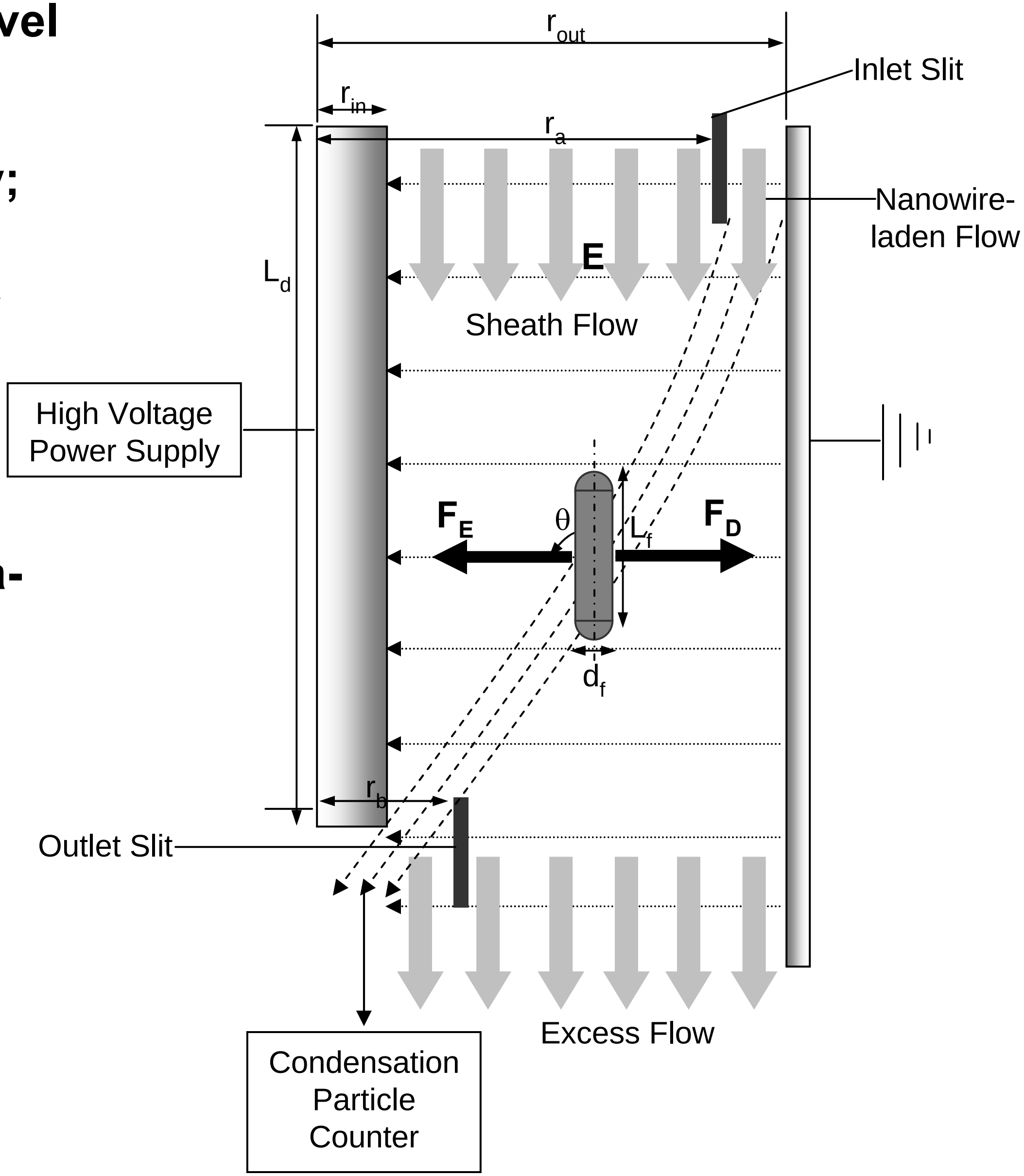
Transit Time through a Differential Mobility Analyzer

t_p = time for the nanowire to travel from inlet slit to outlet slit.

$$t_{p,o} = \int_{r_b}^{r_{out}} \frac{dr}{v_r}$$ **, outermost trajectory;**

$$t_{p,i} = \int_{r_{in}}^{r_a} \frac{dr}{v_r}$$ **, innermost trajectory**

Transfer function = 1 for these trajectories; Hagwood, Sivithanu, Mulholland, *AST*, 1999.



Transit Time through a Differential Mobility Analyzer

t_p = time for the nanowire to travel from inlet slit to outlet slit.

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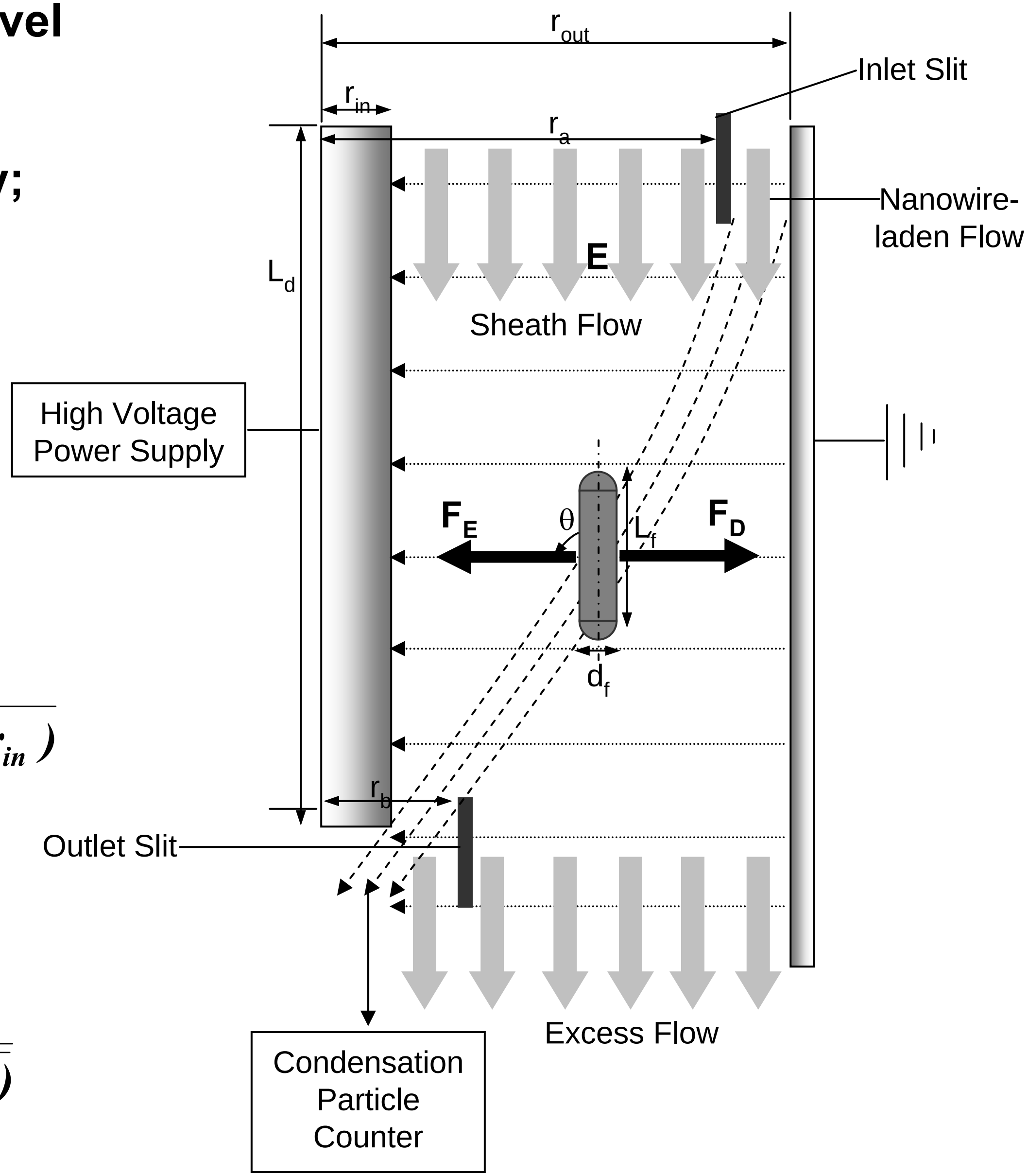


Use the relations:

$$v = E\overline{Z}_p \quad \text{and} \quad E = \frac{V_e}{r\ ln(r_{out}/r_{in})}$$

To express the integral in terms of E and $\overline{Z}_p$.

$$t_{p,o} = \frac{V_e}{ln(r_{out}/r_{in})} \int_{E_{out}}^{E_b} \frac{dE}{E^3\overline{Z_p(E,\beta)}}$$

Connection between Predictions and DMA Measurements

Nanowires are selected by the DMA at several voltages and analyzed by TEM. For these fixed voltages and for a fixed nanowire diameter of 15 nm, the value of the aspect ratio β (length *L*) is adjusted until:

Particle trajectory times, $t_{p,o}$ = fluid transit time, t_f

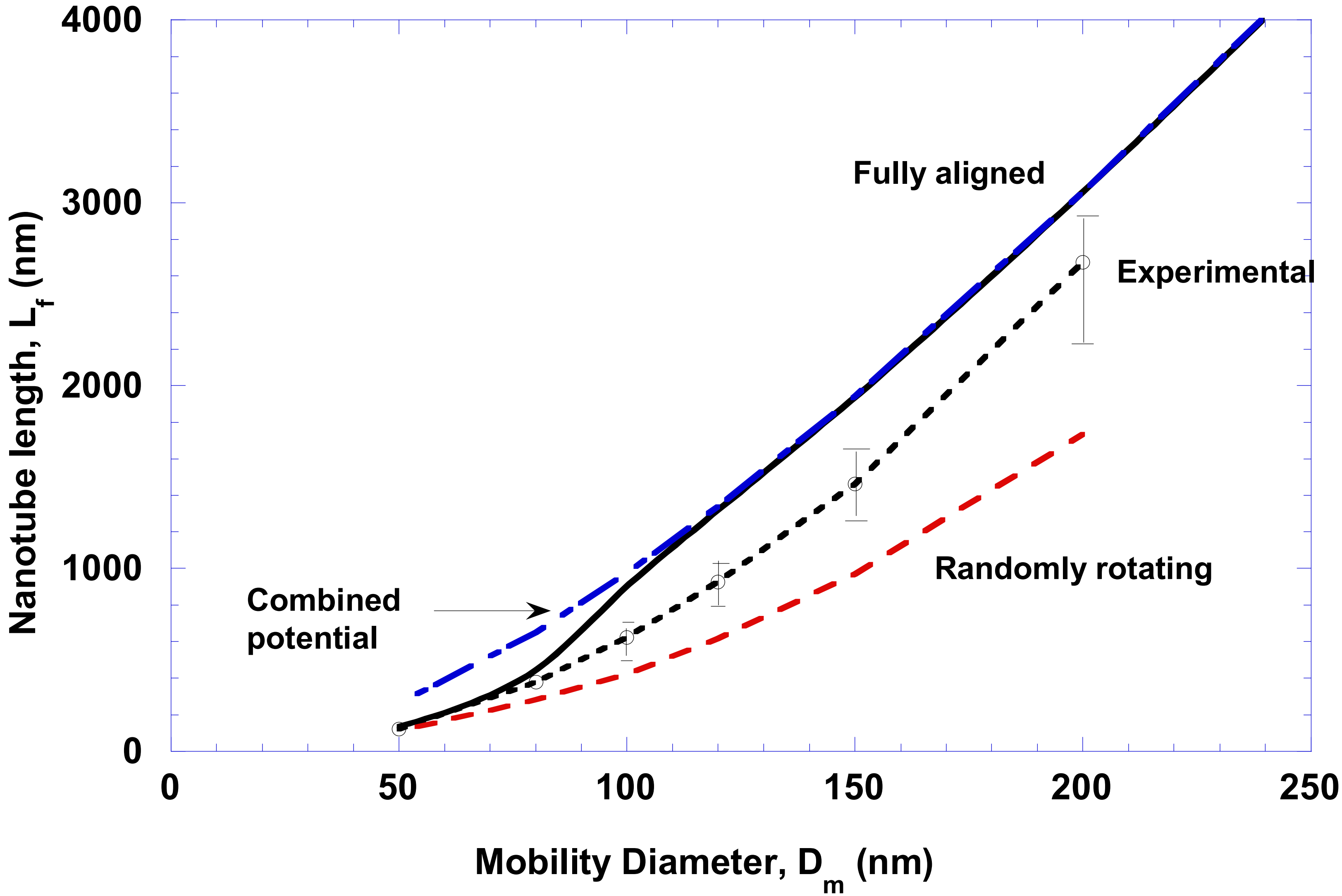
$$t_{p,o} = \frac{V_e}{ln(r_{out}/r_{in})} \int_{E_{out}}^{E_b} \frac{dE}{E^3 \overline{Z_p(E,\beta)}}$$

$$t_F = \frac{\pi(r_{out}^{\;2} - r_{in}^{\;2})L_d}{Q_{total}}$$

Comparison of Predicted and Measured Nanotube Length

t_p, s	V_e, V	D_m, nm	L, TEM nm	L, theory nm
2.26	470	50	121	135
2.26	1089	80	378	405
2.26	1607	100	622	915
2.26	2199	120	926	1320
2.26	3169	150	1460	1935
2.26	4962	200	2674	3060

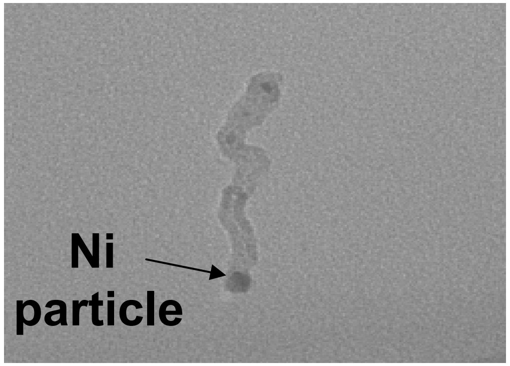
Comparison Experimental and Theoretical Nanotube Length as a Function of Mobility Diameter



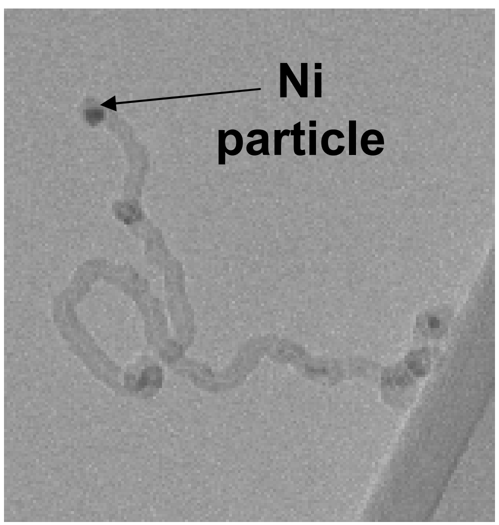
Kim, Mulholland, and Zachariah, J. Aerosol Sci., 38, 823-842, 2007

TEM Micrographs of Carbon Nanotubes

D_m=50 nm for V_e=465 V



L_{CNT}=121±10 nm

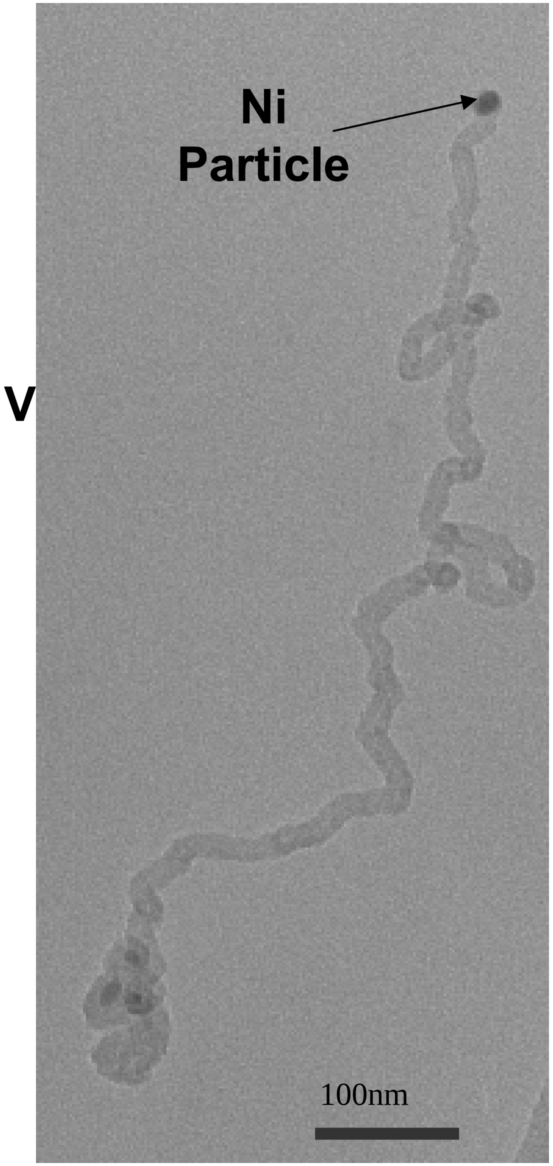


L_{CNT}=378±40 nm

D_m=80 nm

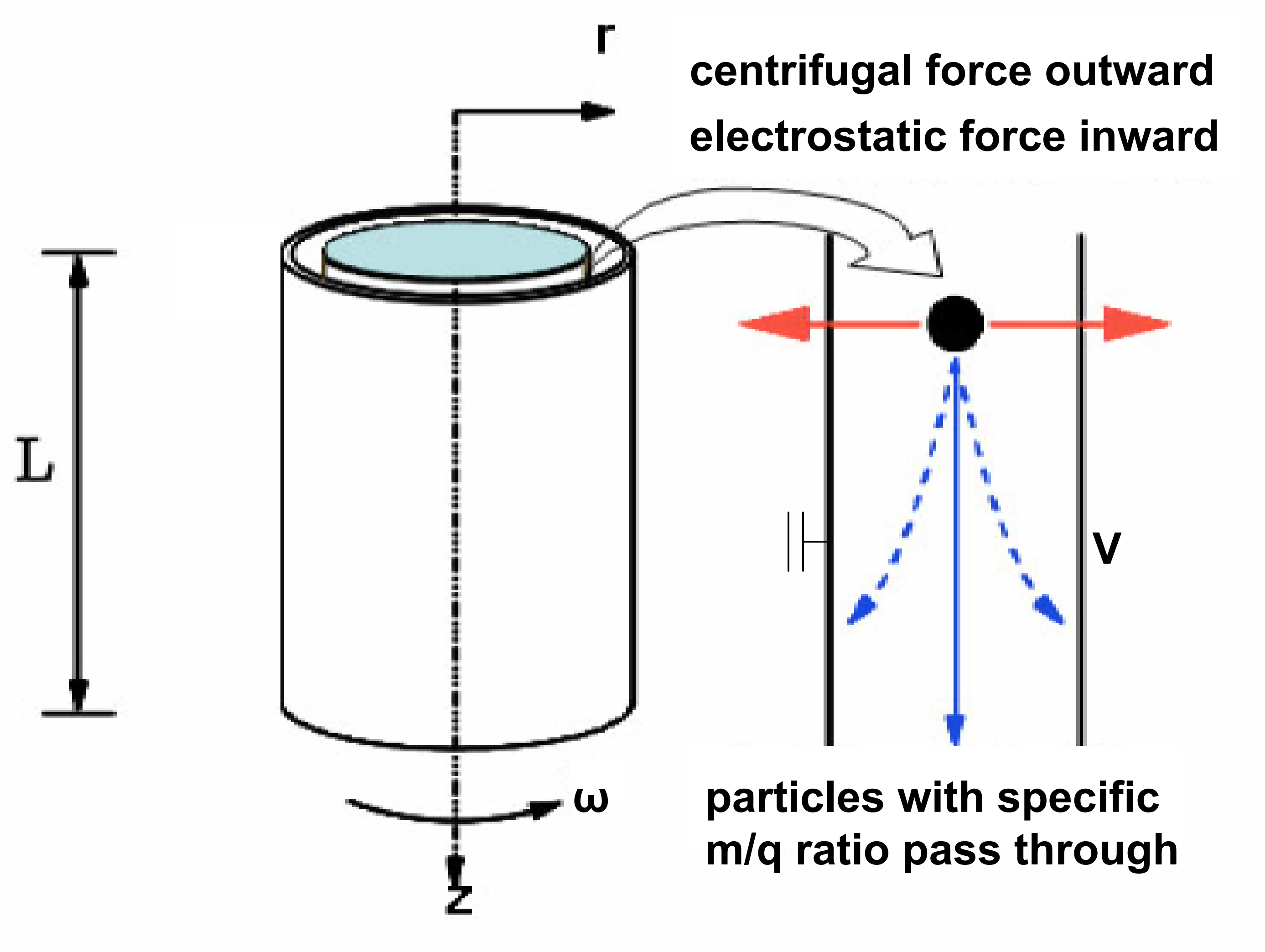
for V_e=1089 V

D_m=120 nm
for V_e=2199 V



L_{CNT}=926±90 nm

Aerosol Particle Mass Analyzer (APM)



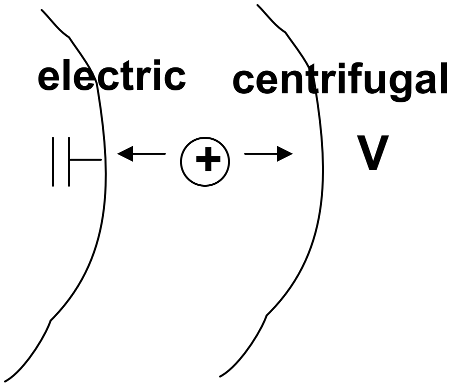
Particle Trajectory in APM

Equation of motion

$$ma_r = m\left(\ddot{r} - r\dot{\theta}^2\right) = -f_d v_r + mr\omega^2 - \frac{qV}{r\,ln\left(r_2/r_1\right)}$$

$u_z\left(r\right) =$ **constant for plug flow**

top view



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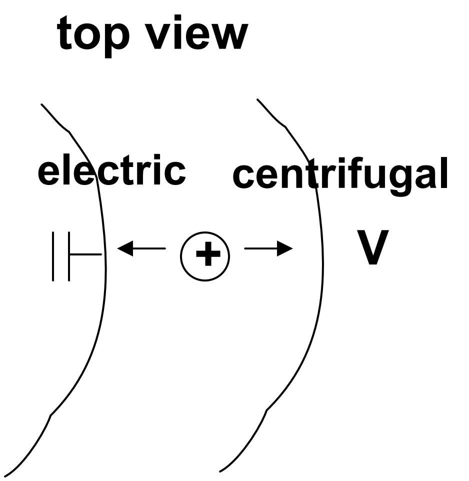


At steady state, a_r=0

$$f_d v_r = f_d \frac{dr}{dt} = mr\omega^2 - \frac{qV}{r\,ln\left(r_2 / r_1\right)}$$

Particle Trajectory in APM

Equation of motion

$$ma_r = m\left(\ddot{r} - r\dot{\theta}^2\right) = -f_d v_r + mr\omega^2 - \frac{qV}{r\,ln\left(r_2 / r_1\right)}$$

$u_z(r) =$ constant for plug flow

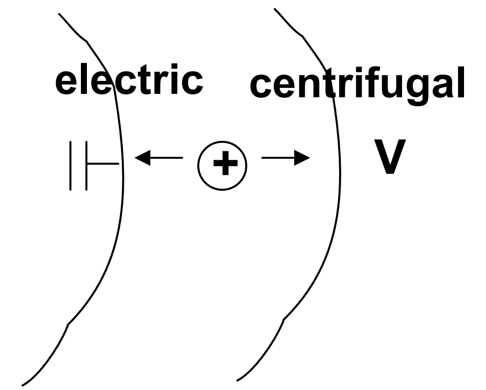
At steady state, a_r=0

$$f_d v_r = m\frac{dr}{dt} = mr\omega^2 - \frac{qV}{r\,ln\left(r_2 / r_1\right)}$$

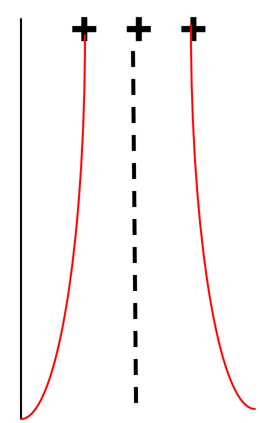
If the two external forces are in balance at r_c =1/2(r_1+r_2),then centroid m/q given by

$$\frac{m}{q} = \frac{V}{r_c^2\omega^2\,ln\left(r_2 / r_1\right)}$$

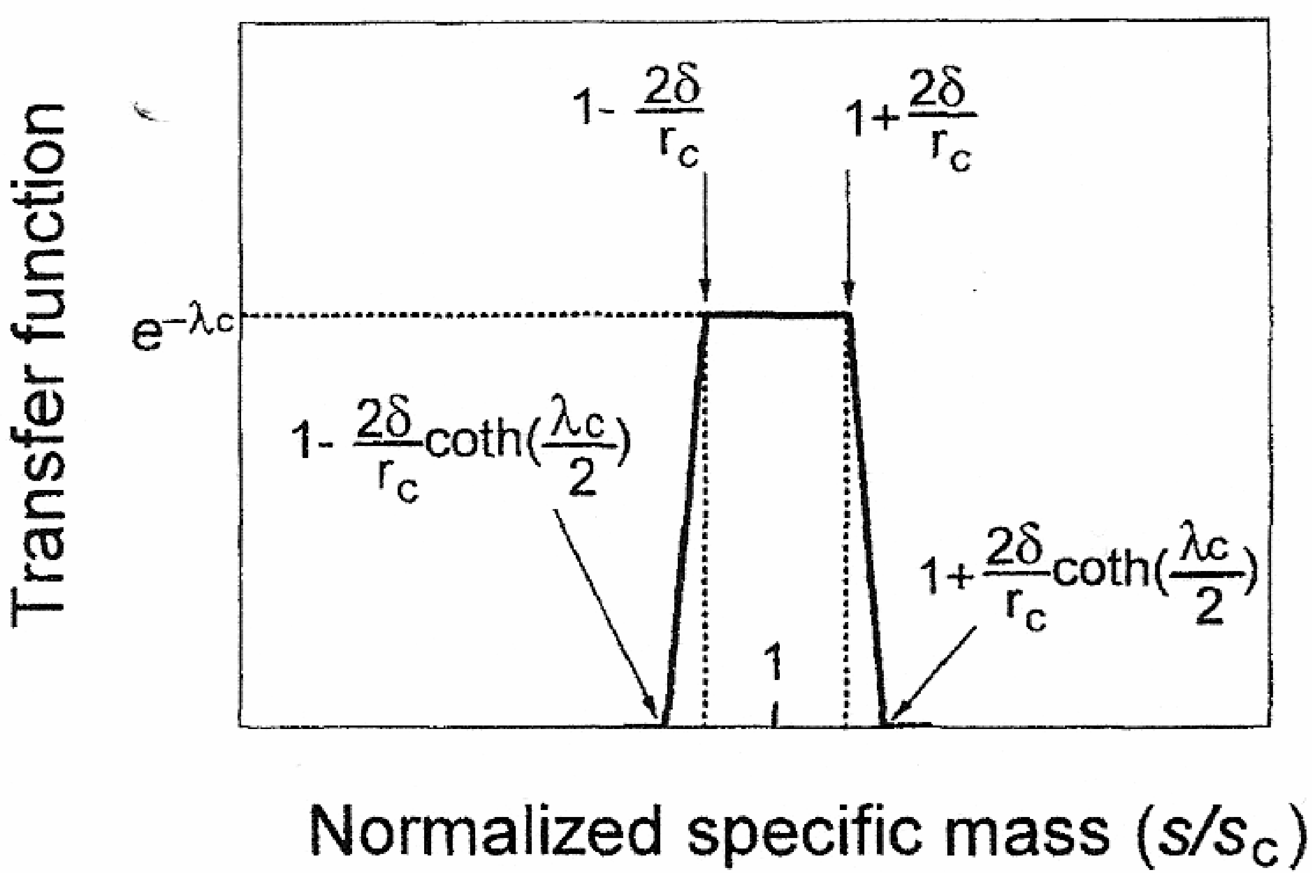
top view



vertical cross section



Transfer Function for Uniform Flow



$$\delta = \frac{r_2 - r_1}{2}$$

$$r_c = \frac{r_1 + r_2}{2}$$

$$\lambda_c = \frac{2\tau(s_c)\omega^2 L}{v_z}$$

Ehara, Hagwood, and Coalkey, J. of Aerosol Sci., 1996

Results for DOP and NaCl

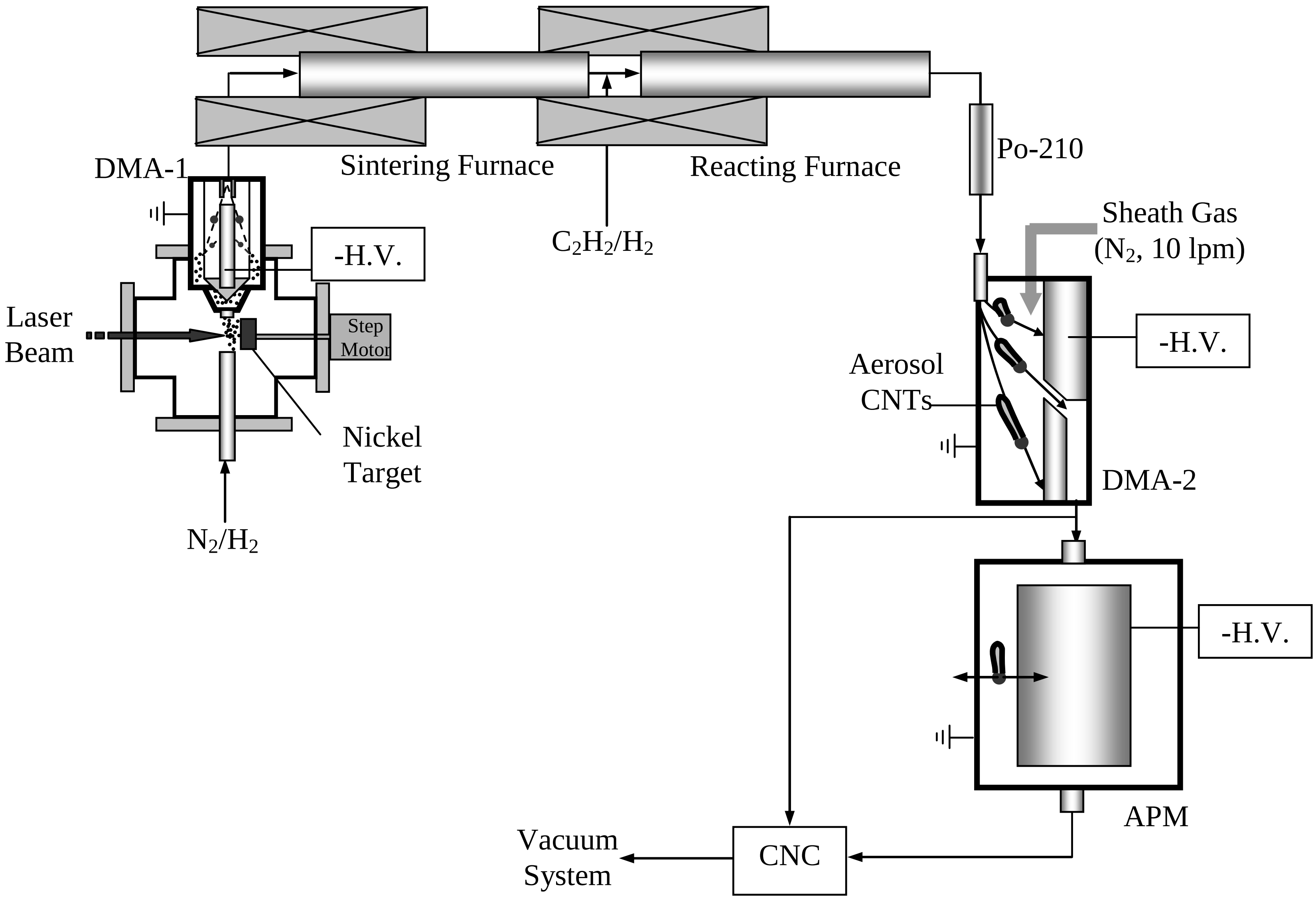
Monodisperse DOP and NaCl aerosols are selected with a DMA and then the mass classification was obtained using the APM.

Material	Mobility Diameter, nm	ρ, g/cm^3
DOP	50	1.07
	100	1.01
	200	1.01
NaCl	50	2.29
	100	2.10
	150	2.09

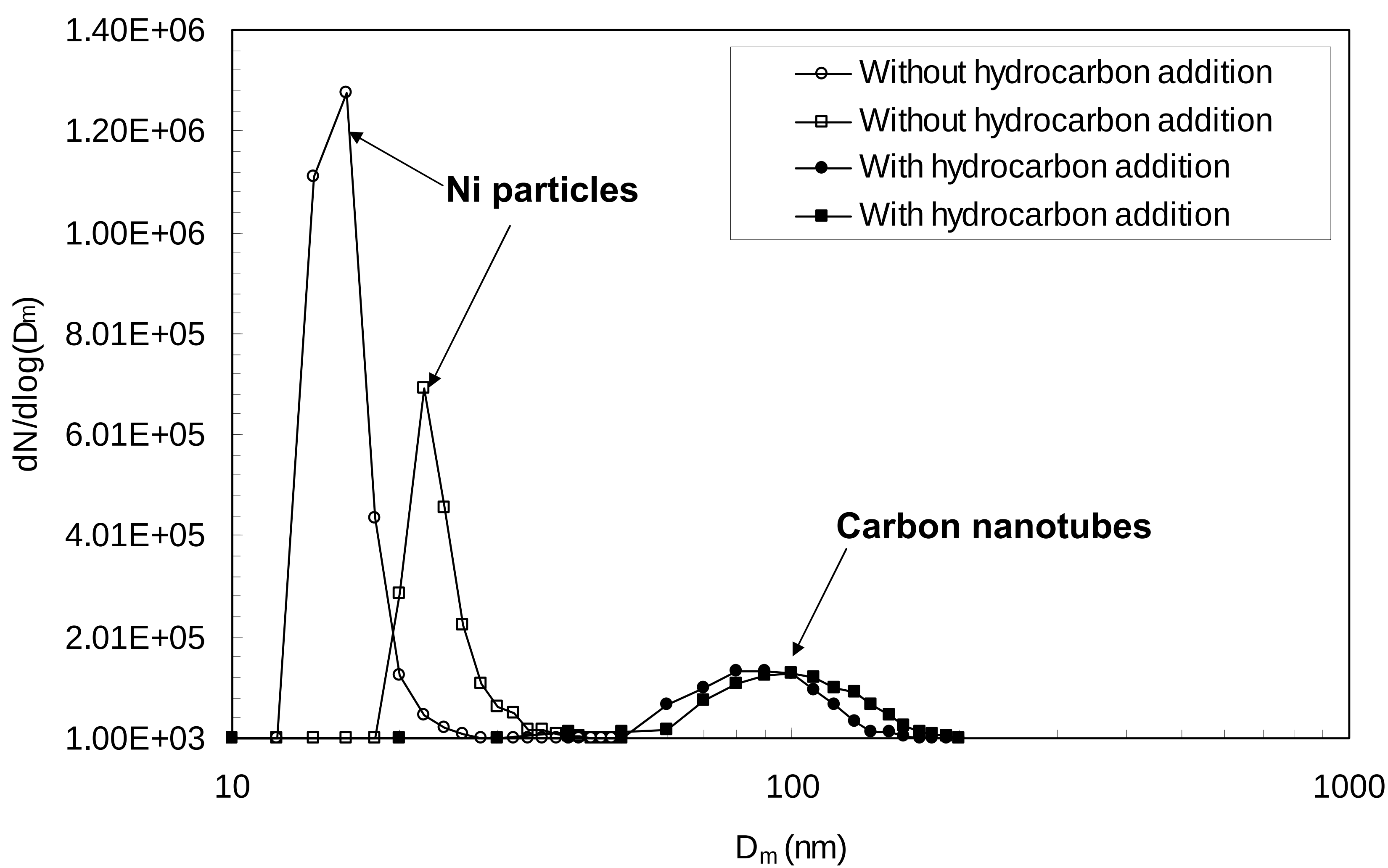
$\bar{\rho}(DOP)$ **= 1.03 g/cm3 vs 0.986 g/cm3**

$\bar{\rho}(NaCl)$ **= 2.16 g/cm3 vs 2.165 g/cm^3**

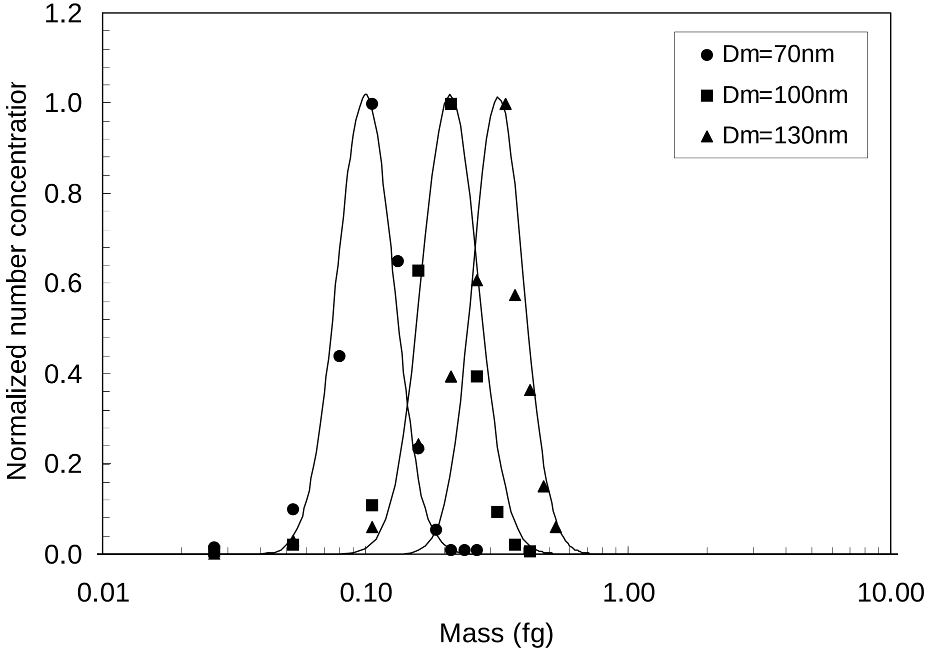
Generation, Size, and Mass Classification of CNTs

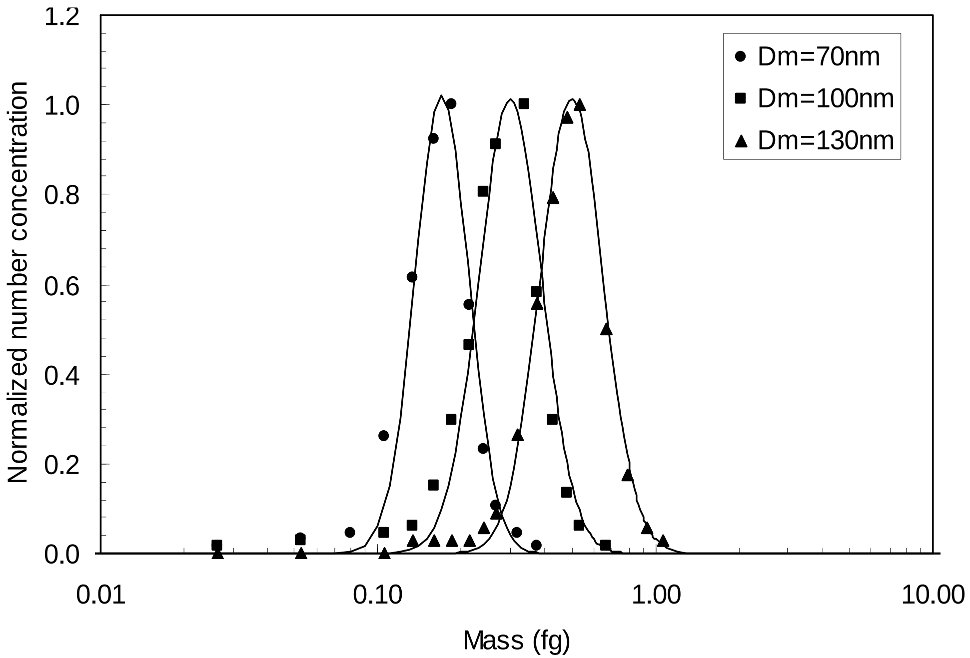


Mobility Size Distributions of Size-Selected Nickel Nanoparticles with and without Hydrocarbon (i.e. C2H2) Reaction for Growing CNTs



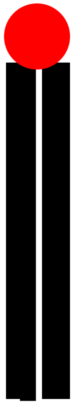
Mass Distributions of Mobility-Classified aerosol CNTs with Tube Diameter of 15 nm (upper) and 22 nm (lower).





Procedure for Computing the Density

1. Compute mass of CNT
   a. Measure the mass of combined CNT and Ni particle.
   b. Use the density of Ni, 8.9 g/cm^3, and diameter of seed particle to compute its mass.
   c. The difference of these two masses is the CNT mass.



2. Compute volume of CNT
   a. Measure the CNT diameter by TEM (15 nm or 22 nm).
   b. Determine the projected area of the CNT from the correlation between the projected area diameter and the mobility diameter.

3. Compute density

$$\rho_{CNT} = \frac{m_{CNT}}{(1/4)\pi D_{CNT} A_{pCNT}}$$

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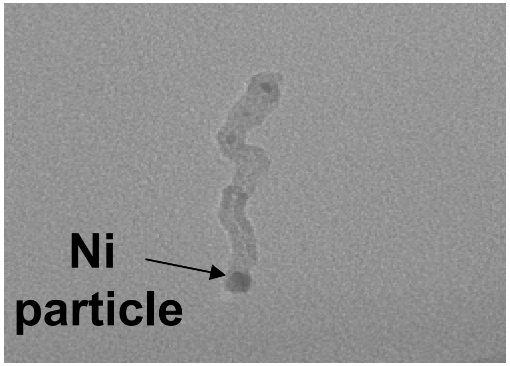

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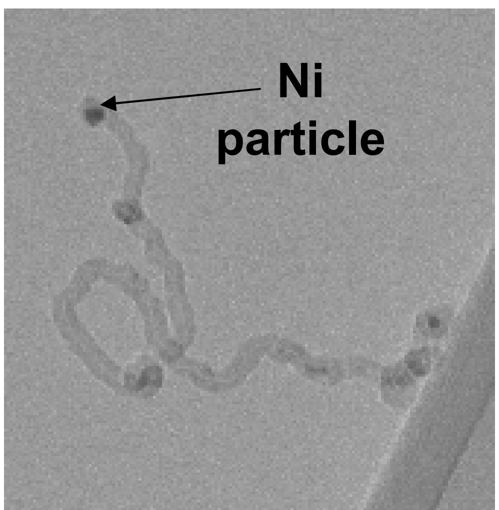

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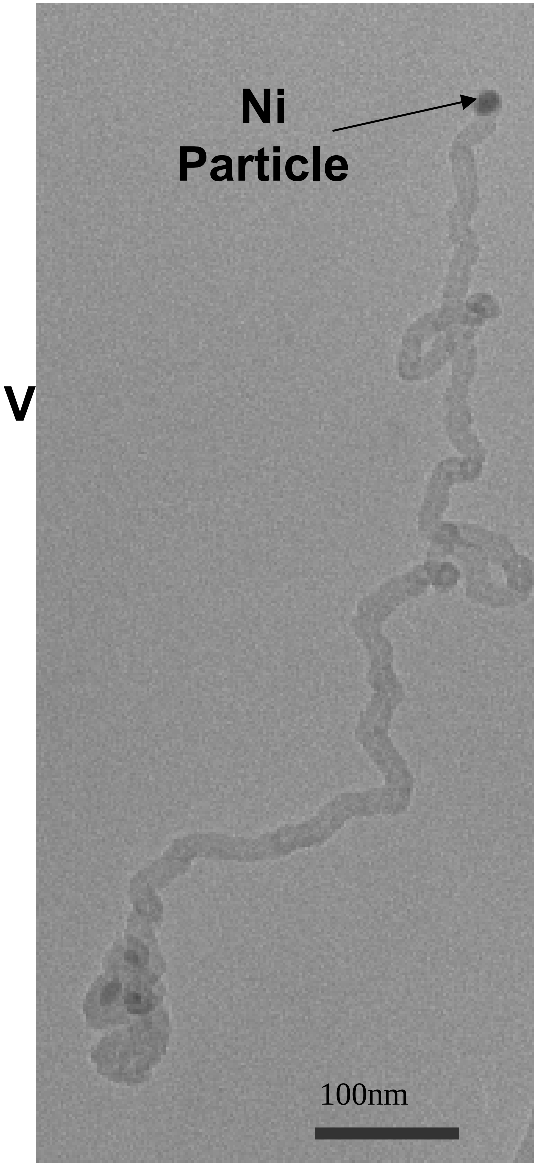
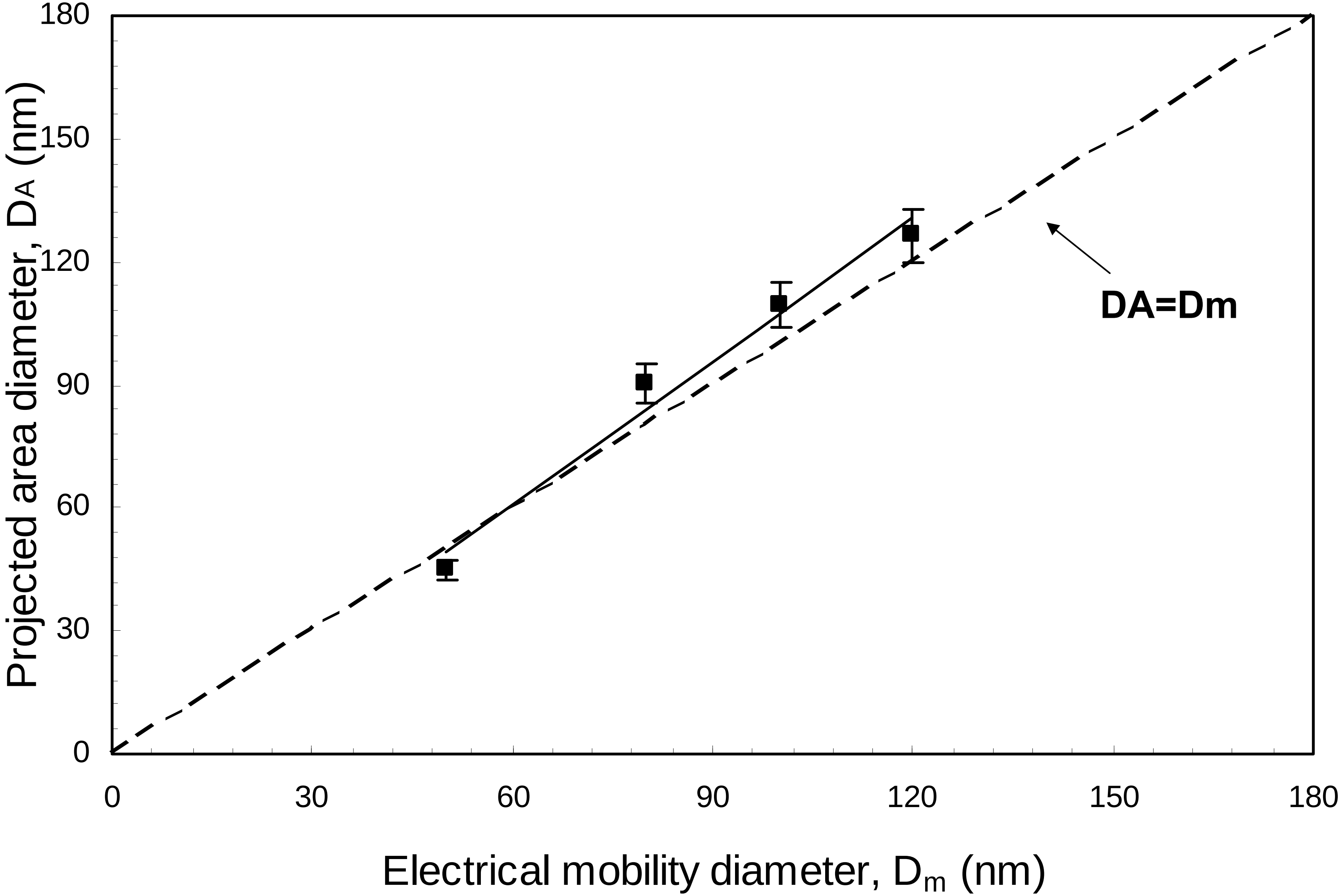


L_{CNT}=926±90 nm

Comparison of Projected Area Diameter of Nanotubes (from TEM) with Electrical Mobility Diameter



Density of MWCNTs for 2 Diameters and 3 Lengths Each

D_{CNT}, nm	D_m, nm	ρ_{CNT}, g/cm^3
15	70	1.74
	100	1.73
	130	1.63
22	70	1.69
	100	1.67
	130	1.75

Mean density ± uncert. = 1.70 g/cm^3 ± 0.15 g/cm^3

Graphite density =2.22 g/cm^3
Carbon black density = (1.84 – 2.06) g/cm^3

I have not been able to find data or predicted densities for MWCNTs

Simultaneous Measurement of L and d_f

Heuristic Example

APM: $m = L d_f^2$

DMA: $A_p = L d_f$

From these two expressions, we can estimate *L* and d_f:

$$d_f = \frac{m}{A_p} \qquad L = \frac{A_p^2}{M}$$

Could these two measurements be useful for monitoring the production of nanowires?

Acknowledgements

Dr. Michael R. Zachariah
University of Maryland/NIST

Dr. Soo H. Kim
Pusan National University