Energy Track: Material Needs in Alternative & Renewable Energy for the Automotive Industry

Lithium ion traction batteries and the role of ceramics

Mark Verbrugge and Mike Balogh

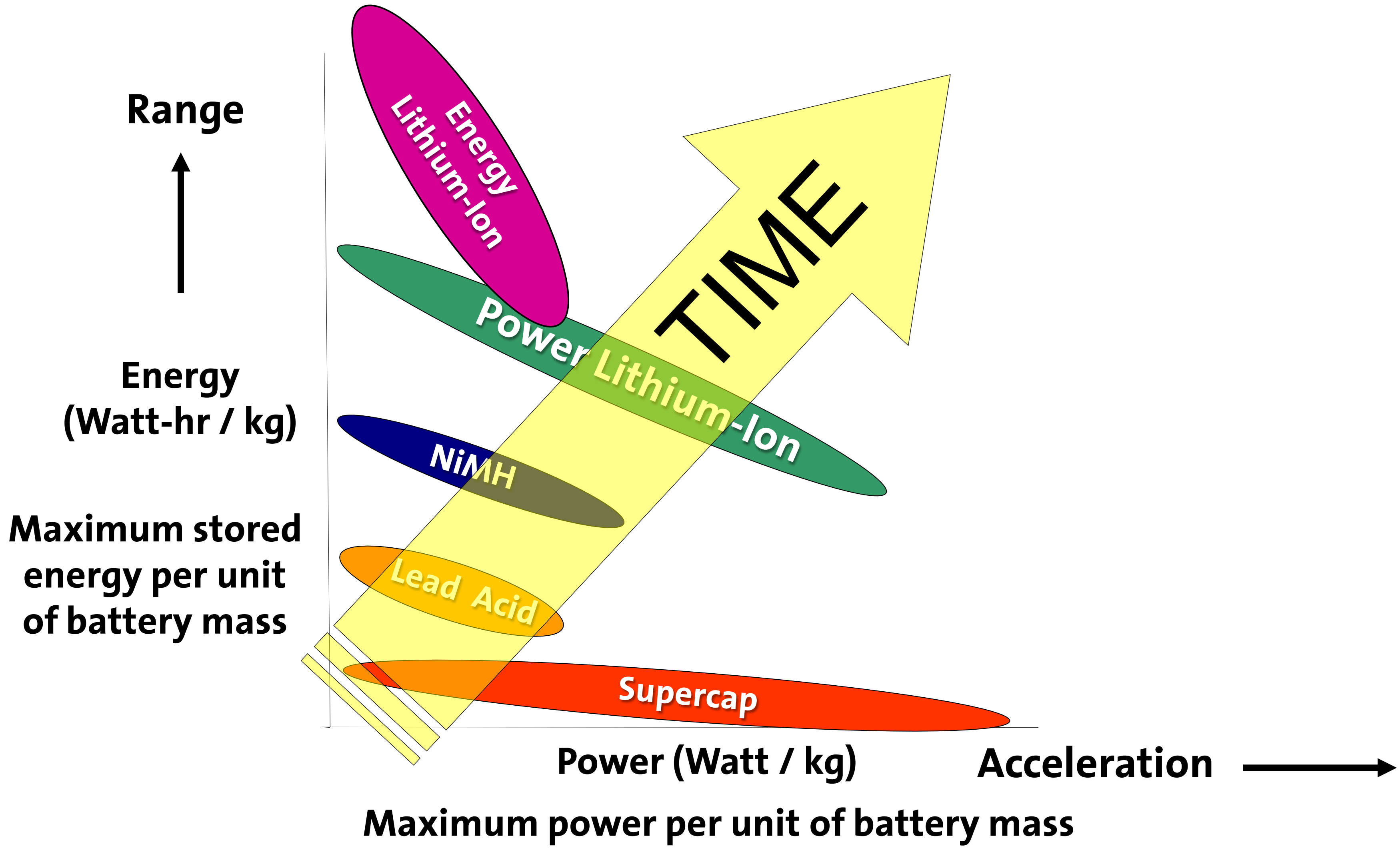
General Motors Research & Development

Ceramic Leadership Summit. August 1-3, 2011, Baltimore, MD

Outline

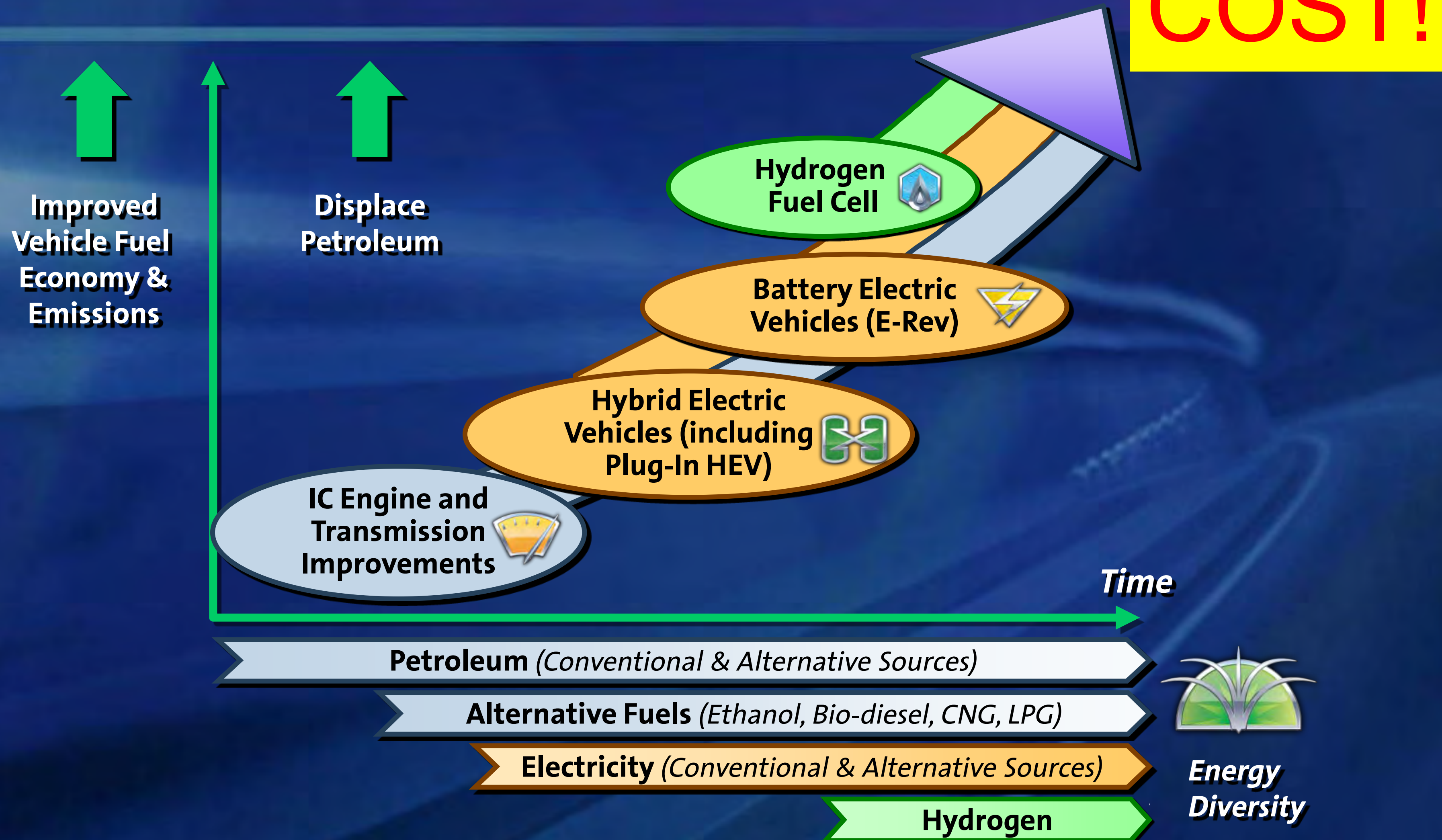
- ❑ **Automotive context**
- ❑ **Short overview/primer on lithium ion batteries**
- ❑ **Ceramics…now and in the future of lithium ion battery technology**
- ❑ **Summary**

Battery Technology Trajectory



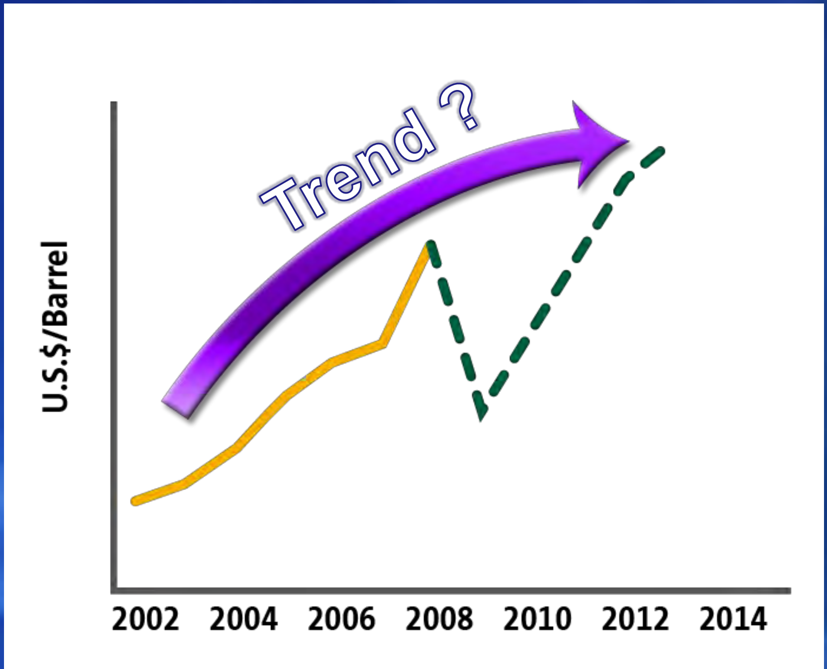
Future vehicles will use alternative energy sources like bio-fuel, grid electricity, and hydrogen

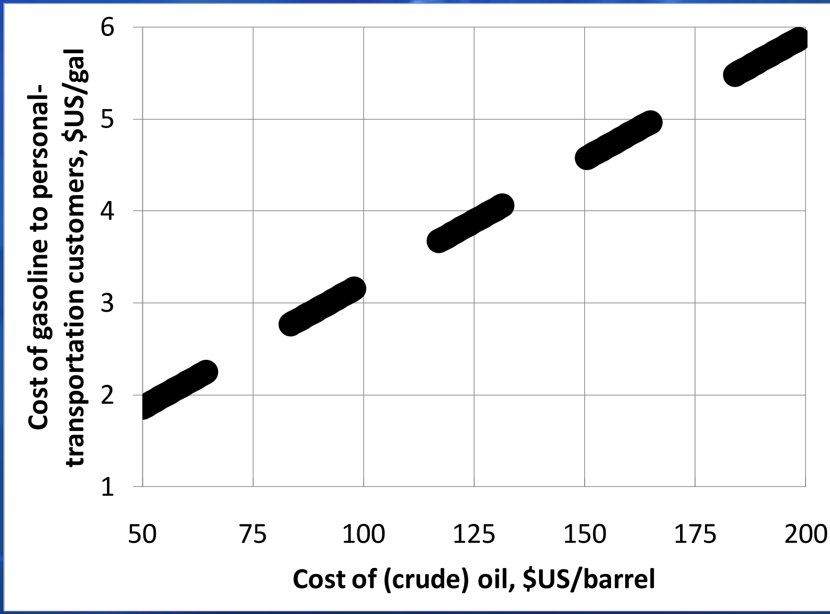
COST!



Really BIG questions

- Liquid fuels
  - Future price & availability of oil?
  - Efficacy of bio-derived fuels?
- ***What is the relative importance of zero on-vehicle "regulated emissions" vs. fuel cost, CO_2 emissions , & energy security?***
- Fuel cell vision offers
  1. Range
  2. Short re-charge times
  3. Zero emissions
  4. Technical efficacy now
- **Another vision: EREV with bio-derived fuels**
  - City: EV (~40 miles)...zero emissions
  - Between cities
    - Liquid fuel: high Wh/kg
    - Regulated emissions from ICE range extender, but greatly reduced today and low for highway driving
- ➢ Energy security, affordability, and reduced unwanted emissions (including CO_2)

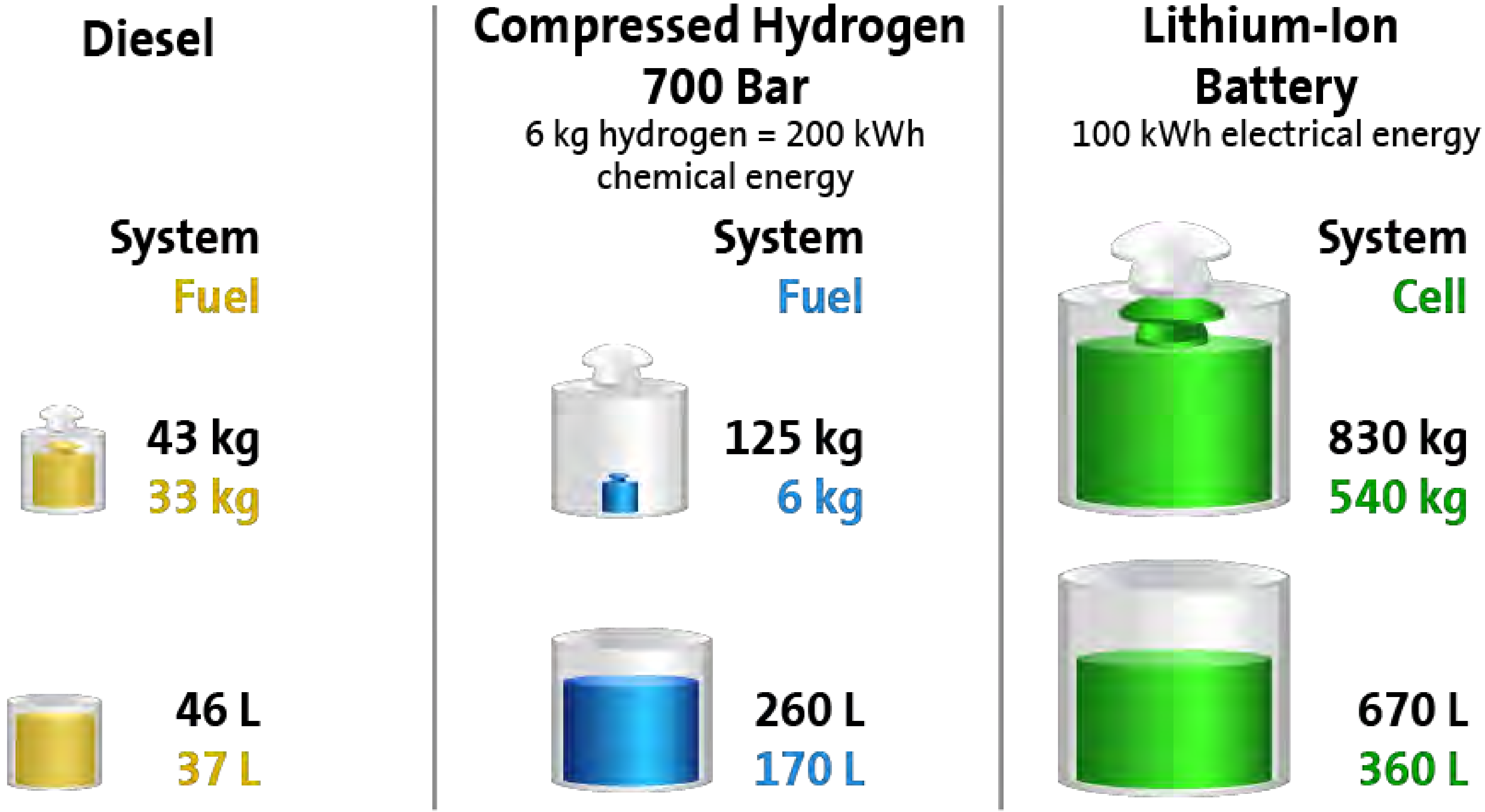




ENERGY CARRIER PROPERTIES: ONBOARD STORAGE

WHY IS PETROLEUM THE DOMINANT TRANSPORTATION FUEL?

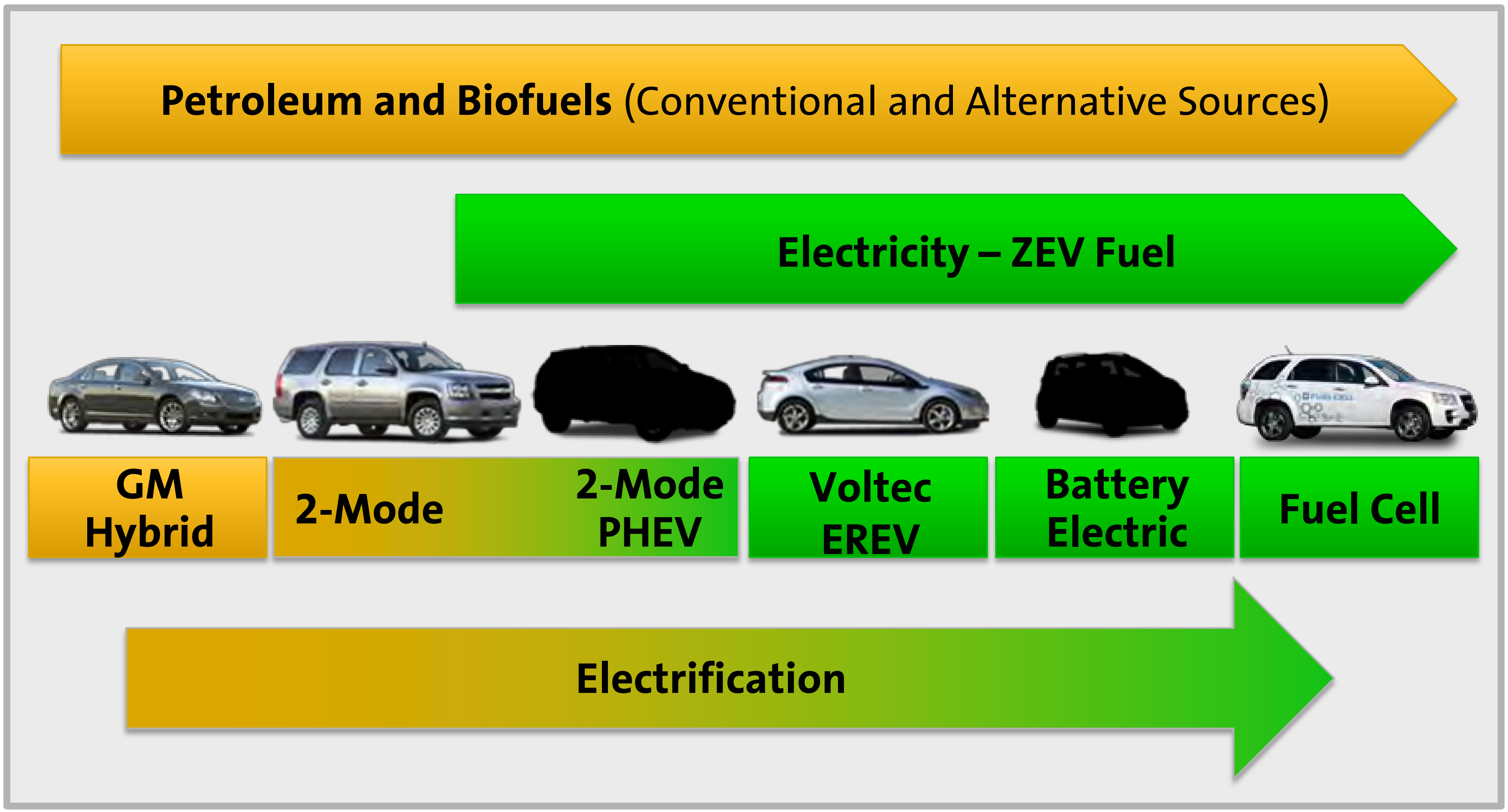
Weight & Volume of Energy Storage System for 500-km Range



EREV: SMALL ZEV BATTERY + LIQUID FUEL FOR RANGE EXTENSION

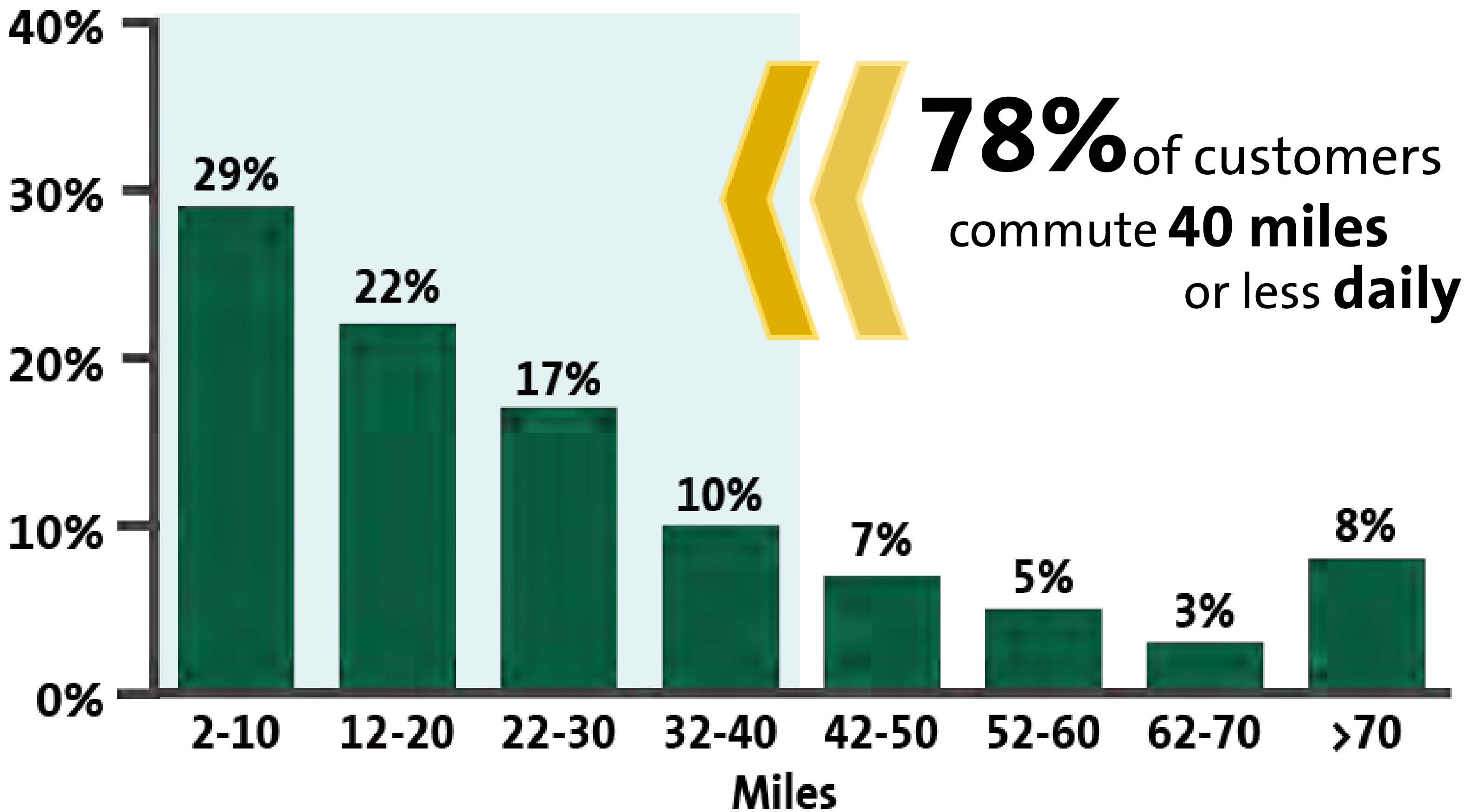
GM Vehicle Electrification Strategy

Portfolio of solutions for full range of vehicles that provide customer choice



Typical Commute

Why Target 40 Miles? → 40 Miles Is the Key



Based on U.S. Department of Transportation 2003 Omnibus Household Survey

Electric Vehicle with RANGE-EXTENDER

VOLT

Up to **40** miles
BATTERY
Electric Drive

\+

HUNDREDS of miles
EXTENDED-RANGE
Driving

Variations on Electric Vehicles

Chevrolet Volt: The Electric Vehicle with Extended Range

http://www.youtube.com/watch?v=JZYN3TK3Fmo

Chevrolet Volt Wins 2011 Motor Trend Car of the Year!.flv (3 minute Motor Trend Video)

PHEV

Plug-in Hybrid Electric Vehicle

- All-electric at low speed/power
- Blended electric/gas at higher speed/power
- Primary fuel is gasoline supplemented with electricity

(typical)

EV with Extended Range

Electric Vehicle with "Extended-Range"

- All-electric for up to 40 miles
- Gas generator for +300 miles extended driving range
- Primary fuel is electricity supplemented with gasoline

(Volt)

Pure EV

Pure Electric Vehicle

- All-electric for ~100 miles
- Fuel is electricity

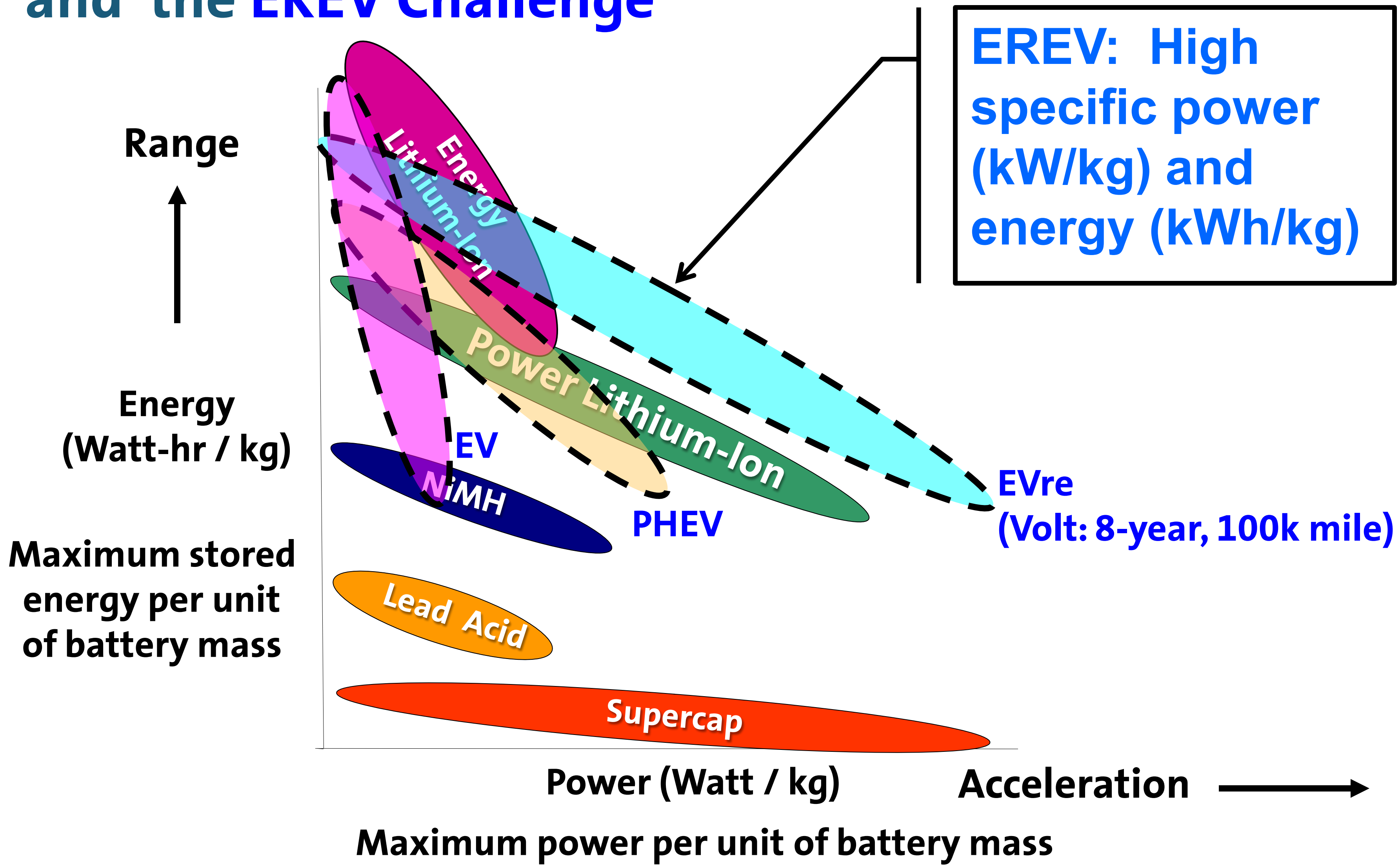
(typical)

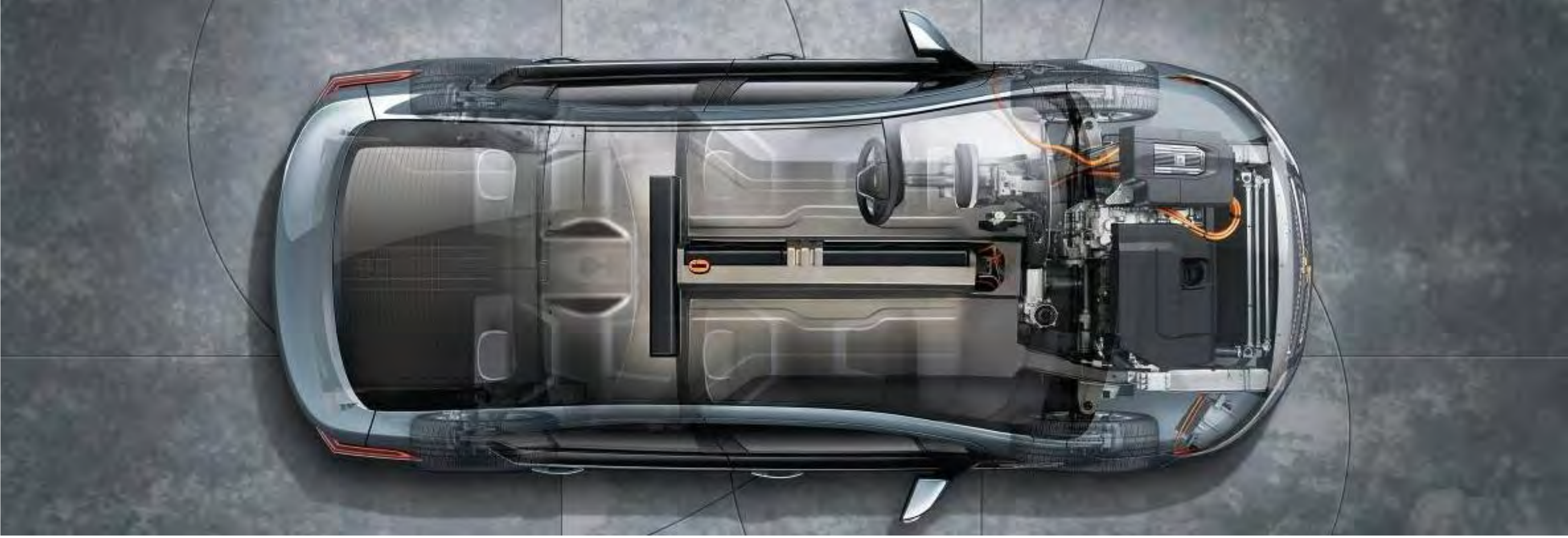
Electricity as Low-Cost Fuel (US costs)

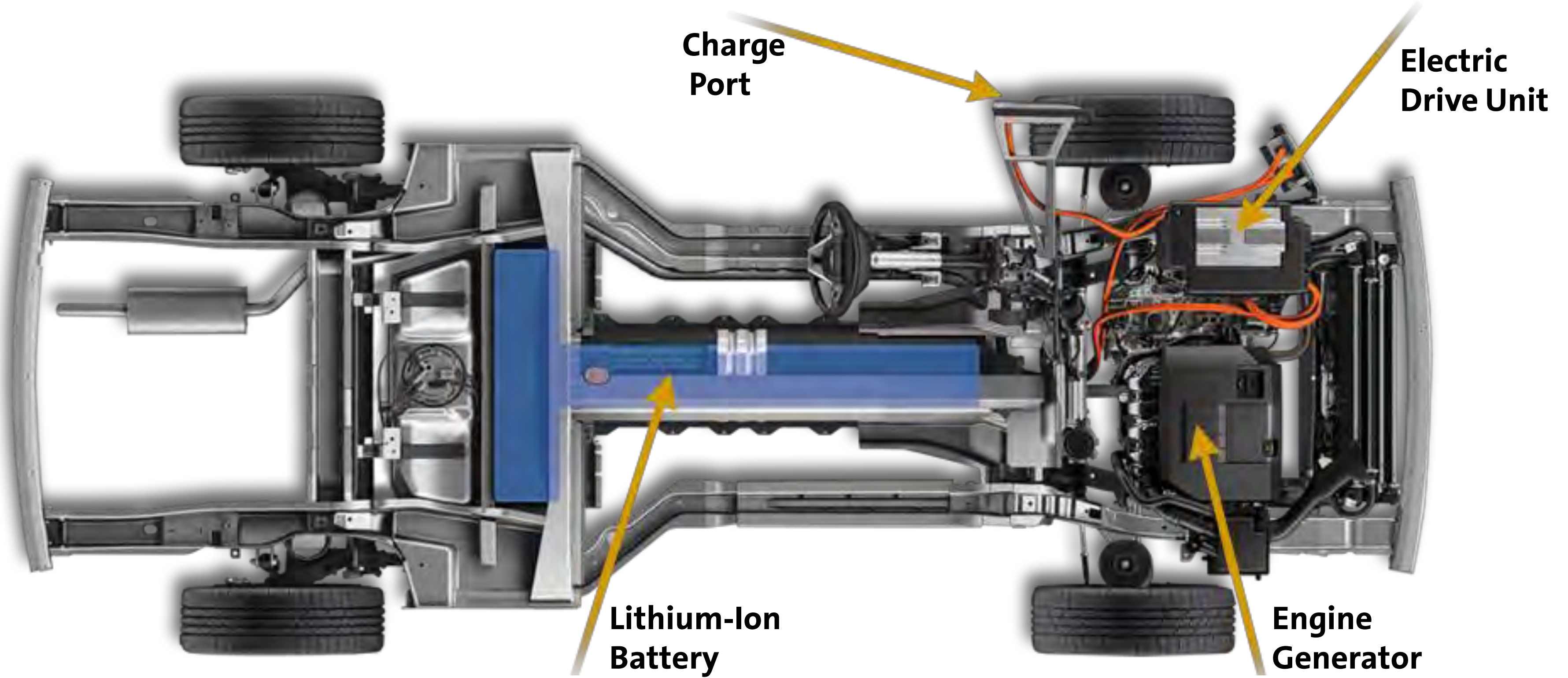
7-13¢ per mile

1-2¢ per mile

Battery Technology Improvements and the EREV Challenge







Charge Port
Electric Drive Unit
Lithium-Ion Battery
Engine Generator

North American Car of the Year for 2011

Motor Trend 2011 Car of the Year

Green Car Journal 2011 Green Car of the Year

Car and Driver 10 Best for 2011

Ward's AutoWorld 10 Best Engines for 2011

AUTOMOBILE Magazine 2011 Automobile of the Year

2010 Breakthrough Technology, by Popular Mechanics

THE ALL NEW 2011 CRUZE

Introducing the Cruze Eco and amazing highway fuel economy at 42 MPG that doesn't sacrifice the sculpted exterior design that sets Cruze apart from the competition.

Kelley Blue Book Consumer Reviews

2011 - 2011 Chevrolet Cruze models

Overall Rating:	**4.6** out of **5**
Value:	**4.4** out of **5**
Reliability:	**4.4** out of **5**
Quality:	**4.7** out of **5**
Performance:	**4.3** out of **5**
Styling:	**4.6** out of **5**
Comfort:	**4.3** out of **5**

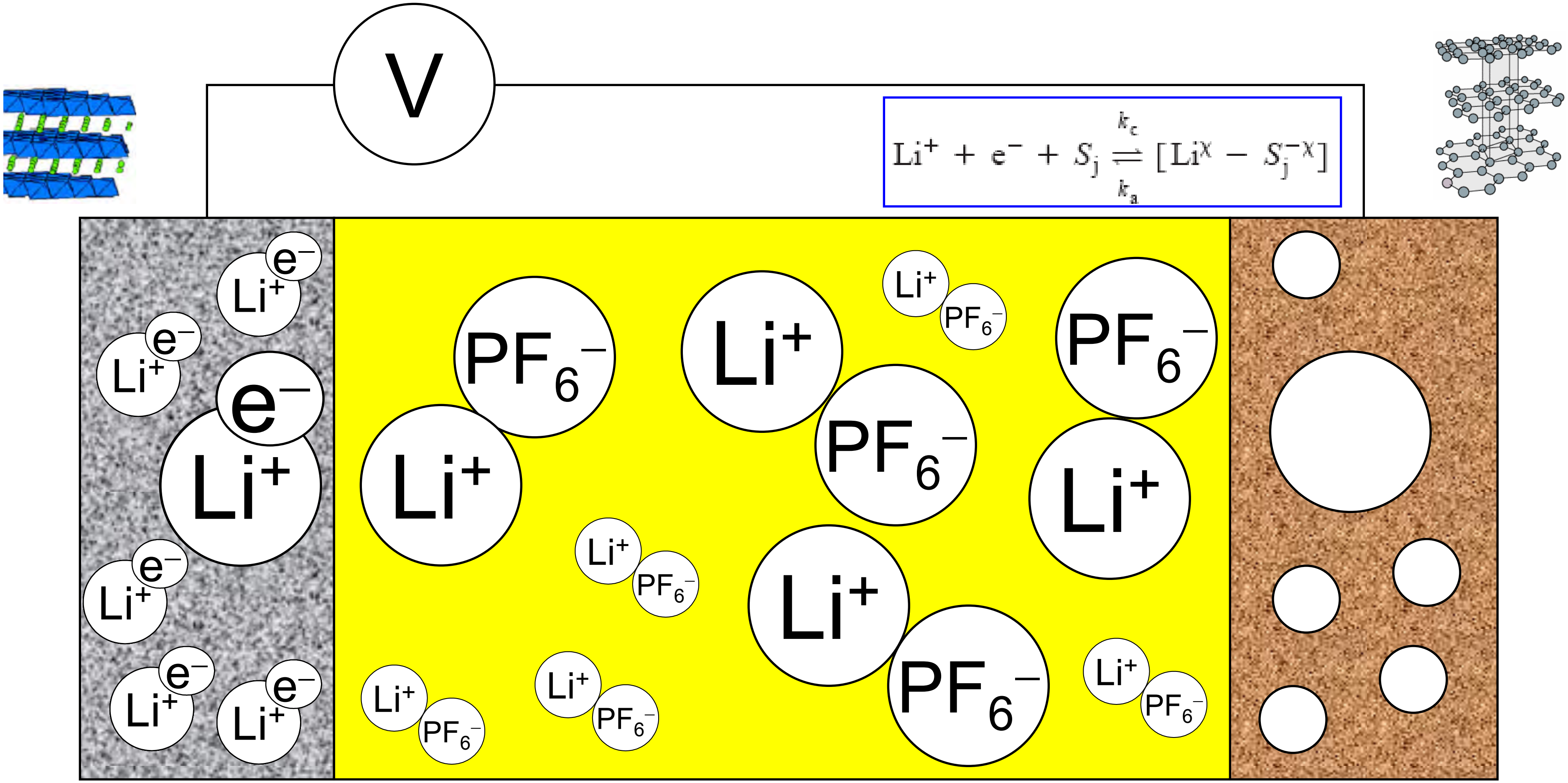
Based on 28 Ratings

Cruze 2LT in Silver Ice Metallic with available 17" 5-spoke machined-face alloy wheels.

Lithium ion battery challenges

- **Cost**
  - Can we size pack closer to end-of-life requirements?
  - Can we reduce materials & processes costs?
- **Life**
  - How do electrodes fail?
  - Can we develop an accelerated life test?
- **Temperature tolerance**
  - Can we improve low temperature power?
  - Why is battery life shorter at higher temperatures?

Charge mechanism (reverse of discharge)



(+) Metal oxide, phosphate, or silicate

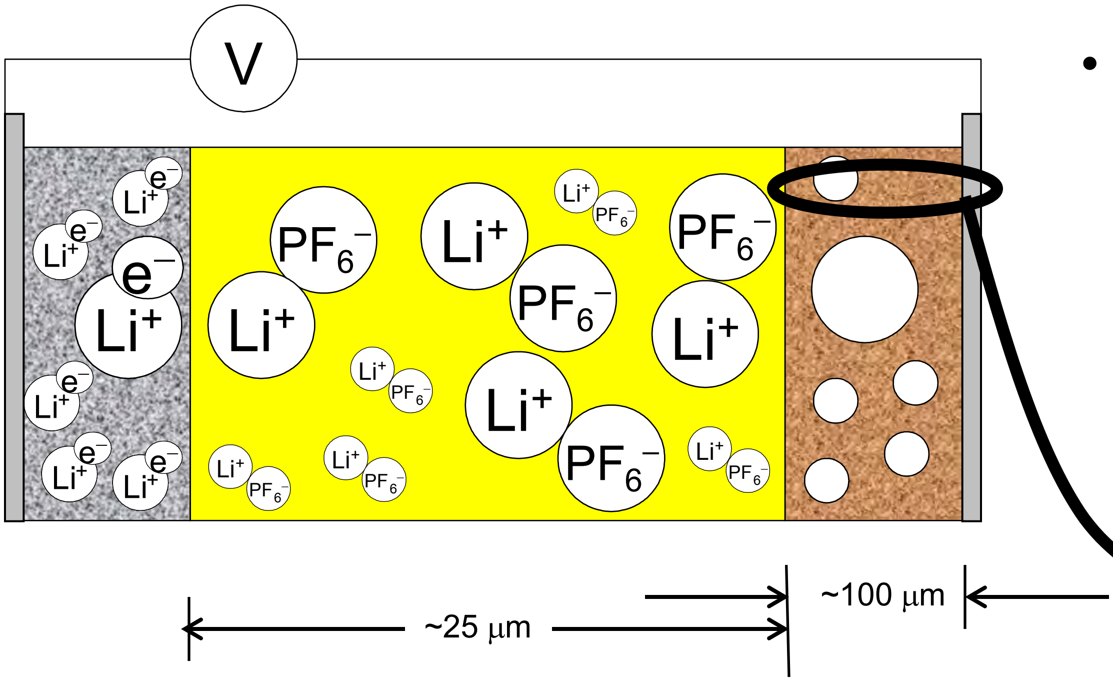
Separator (Solvent + Salt), often ceramic enhanced

(–) Carbon, titanate, etc.

Positive is full of lithium at the end of discharge

By putting energy into the cell for charging, lithium is forced out of the positive and into the negative.

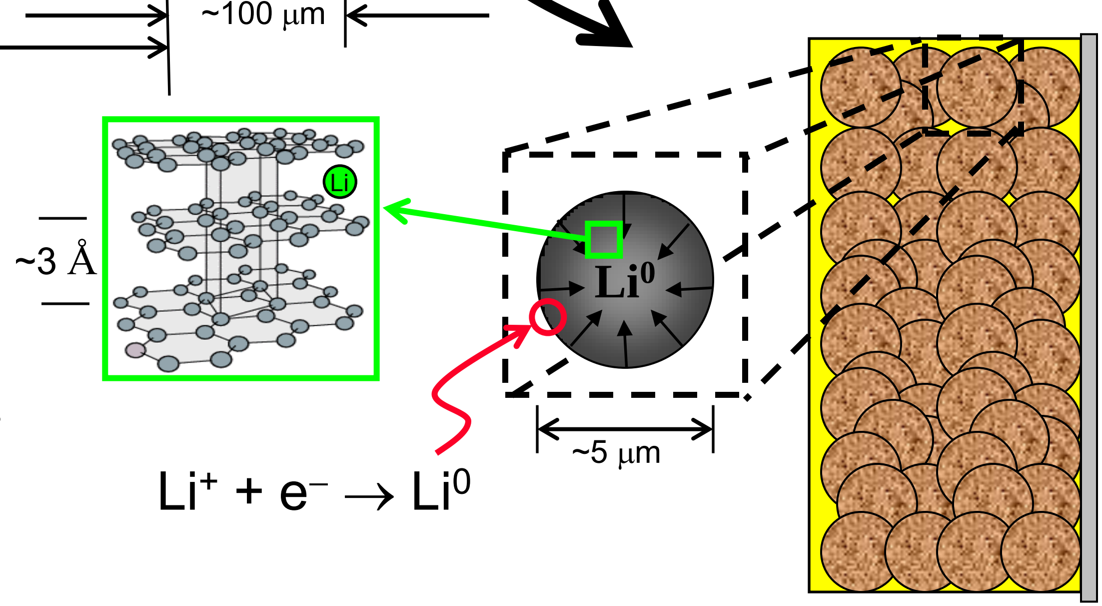
Electrode microstructure



- Porous electrodes (~100 µm thick) composed of host particles (~1 to 5 µm diameter) are used to
  1. increase the surface area for reaction
  2. reduce lithium diffusion resistance

Negative charge reaction

1. At the particle surfaces:
   $Li^+ + e^- \rightarrow Li^0$
2. Lithium diffuses *rapidly* into host particle
   - Opposite reactions takes place at positive particle surfaces
   - Li plating must be avoided



Positive electrode materials

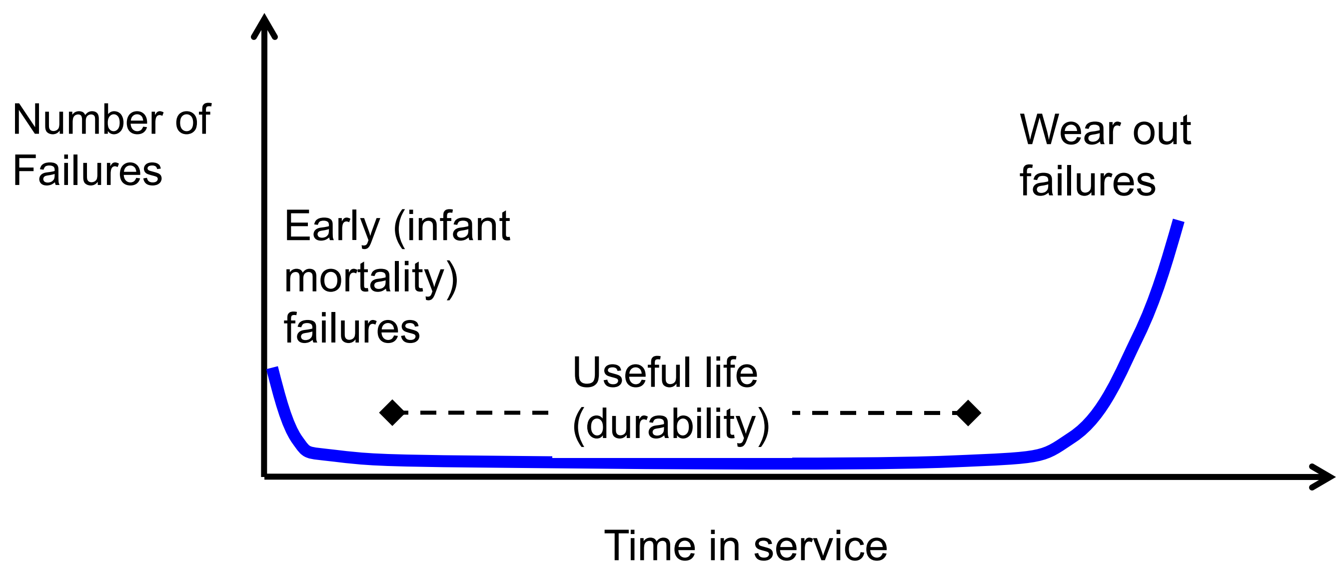
- **Often ceramics**
  - $LiMO_2$ (with M = Ni, Co, Mn, Al … or combinations thereof) is the most used positive material (includes $LiCoO_2$, NCM, LNCA)
  - $LiMn_2O_4$ (spinel) is low cost and provides high power density along with good abuse tolerance
  - $LiMPO_4$ (with M = Fe, Mn, Mg, … or combinations thereof)
  - Li_2MnO_3-$Li(Ni_xCo_yMn_z)O_2$ (with x + y + z = 1) is of strong interest currently
  - $LiMSiO_4$ (with M = Fe, Mn, … or combinations thereof) is showing promise as a low cost, high capacity positive
  - The positive electrode material is a major cost driver in LiIon batteries
  - The potential for solvent oxidation at the positive electrode leads to abuse tolerance concerns

Ceramic coatings to suppress unwanted side reactions

Particle cracking…back to the ceramics literature

Durability…terminologies, bathtub curves

- **Chemical degradation**
  - Critical role of SEI (solid electrolyte interface) to impede deleterious degradation reactions within lithium ion cells
  - Calendar life determined by chemical degradation
- **Mechanical degradation**
  - Cyclic expansion and contraction of insertion or alloy materials leads to fatigue, cracking, and structural changes
  - Cyclic life issues are affected by mechanical degradation and chemical degradation



Electroanalytical cell for characterization of graphite negative

$$\underset{x=0}{|}\quad Li_yC_6 \text{ electrode} \quad \underset{x=\ell_{Li_yC_6}}{|} \quad \text{Electrolyte} \quad \underset{x=\ell_{Li_yC_6}+\ell_e}{|} \quad Li^0 \text{ electrode}$$

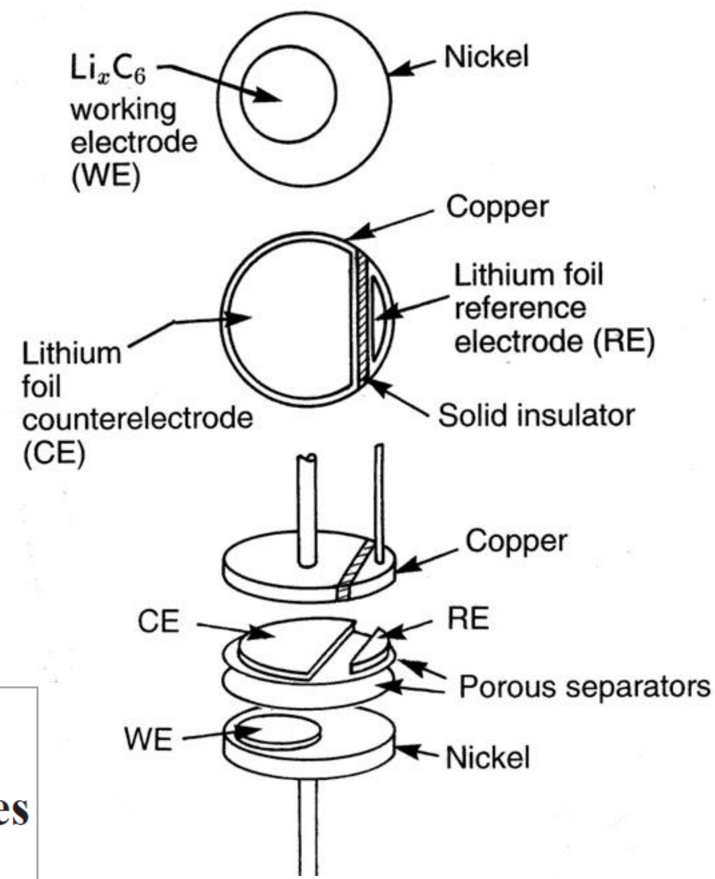
- **Substantially uniform current distribution and cell pressure**
  - Li reference in the counter-electrode plane
  - Electrochemical reaction on the surfaces of the graphite particles (WE):

$$Li^+ + e^- + S \underset{k_a}{\overset{k_c}{\rightleftharpoons}} [Li^{\delta} - S$$

  - At the Li electrodes (CE, RE),

$$Li^{+} + e \rightleftharpoons$$

Three Electrode Cell



Journal of The Electrochemical Society, **150** (3) A374-A384 (2003)
0013-4651/2003/150(3)/A374/11/$7.00 © The Electrochemical Society, Inc.

Electrochemical Analysis of Lithiated Graphite Anodes

Mark W. Verbrugge,[a,*,z] and Brian J. Koch[b,*]

Negative electrode…the solid electrolyte interface (SEI)

Mathematical modeling of high-power-density insertion electrodes for lithium ion batteries

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$$\mathrm{Li}^{+} + \mathrm{e}^{-} + S_{\mathrm{j}} \underset{k_{\mathrm{a}}}{\overset{k_{\mathrm{c}}}{\rightleftharpoons}} [\mathrm{Li}^{\chi} - S_{\mathrm{j}}^{-\chi}]$$

Journal of Power Sources 110 (2002) 295–309

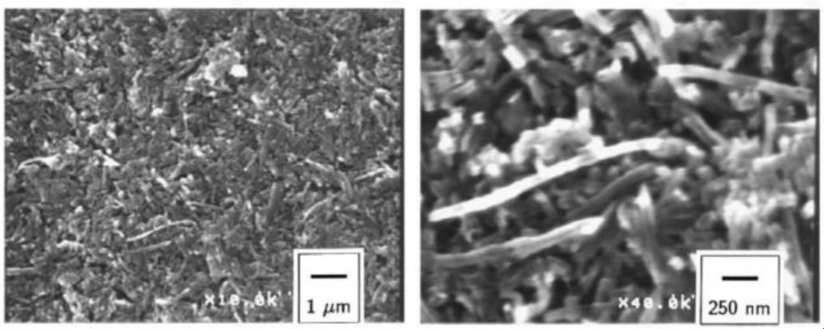
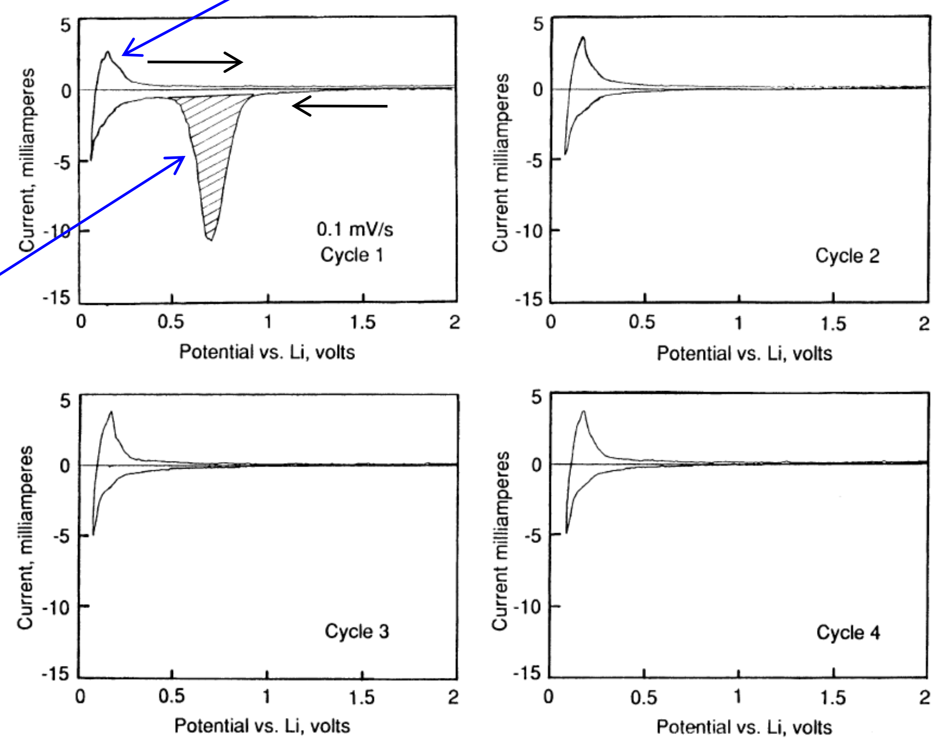


Fig. 3. High-magnification electrode micrographs.



- Solvent reduction at ~0.8V vs Li on first cycle
- Then ~100% Coulombic efficiency

Electrochemical and Solid-State Letters, **13** (9) A128-A131 (2010)
1099-0062/2010/13(9)/A128/4/$28.00 © The Electrochemical Society

Application of Hasselman's Crack Propagation Model to Insertion Electrodes

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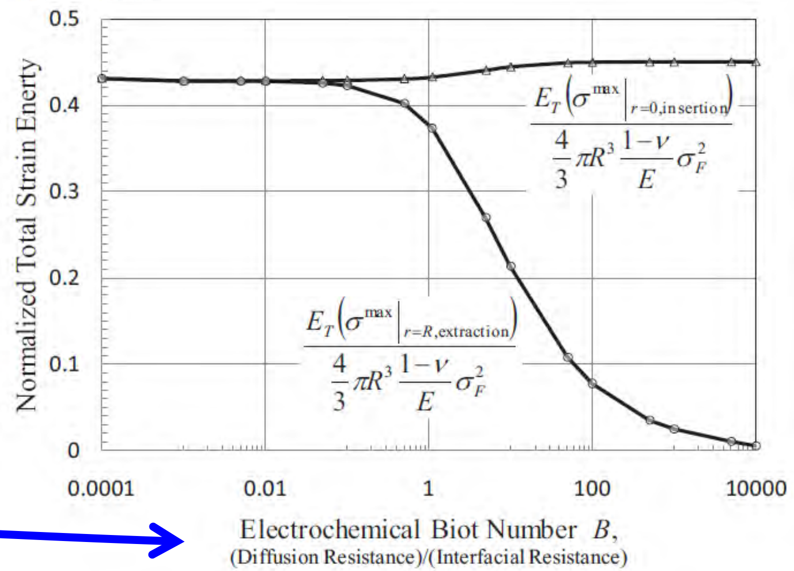
[c] *General Motors Research and Development Center, Chemical Sciences and Materials Systems Laboratory, Warren, Michigan 48090, USA*

Electrode failure, in the form of fracture or decrepitation, can occur as a result of repeated volume expansion and contraction in insertion electrodes used in lithium-ion batteries. Deleterious reactions with the electrolyte may also occur at the newly cracked surface, causing capacity fade. In this article, we derive an analytic expression for the total elastic energy stored within a spherical particle due to diffusion-induced stresses. Using this equation and taking an approach similar to that of Hasselman [*J. Am. Ceram. Soc.*, **46**, 535 (1963)], we establish a criterion for crack propagation within a spherical insertion electrode.
 [DOI: 10.1149/1.3455179]

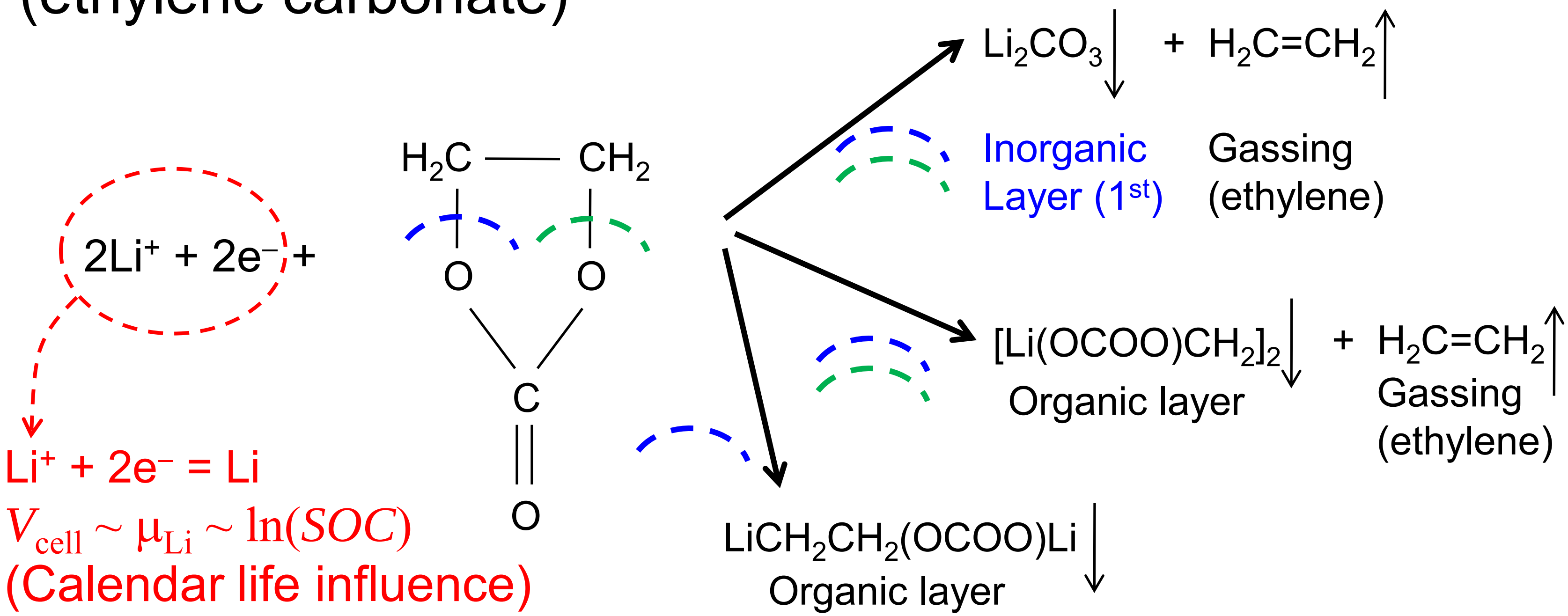
Following Hasselman's approach and using Eq. 12, we equate the total surface energy required for the propagation of the cracks to the elastic energy stored at fracture

$$E_T = f(B)\frac{4}{3}\pi R^3 \frac{1-\nu}{E}\sigma_F^2 = 2AN\gamma_{eff}$$

$$B = k_a^{\ \beta} k_c^{\ 1-\beta}\left[e^{(1-\beta)f(V-U^{\theta})} - e^{-\beta f(V-U^{\theta})}\right]\frac{R}{D_I c}$$



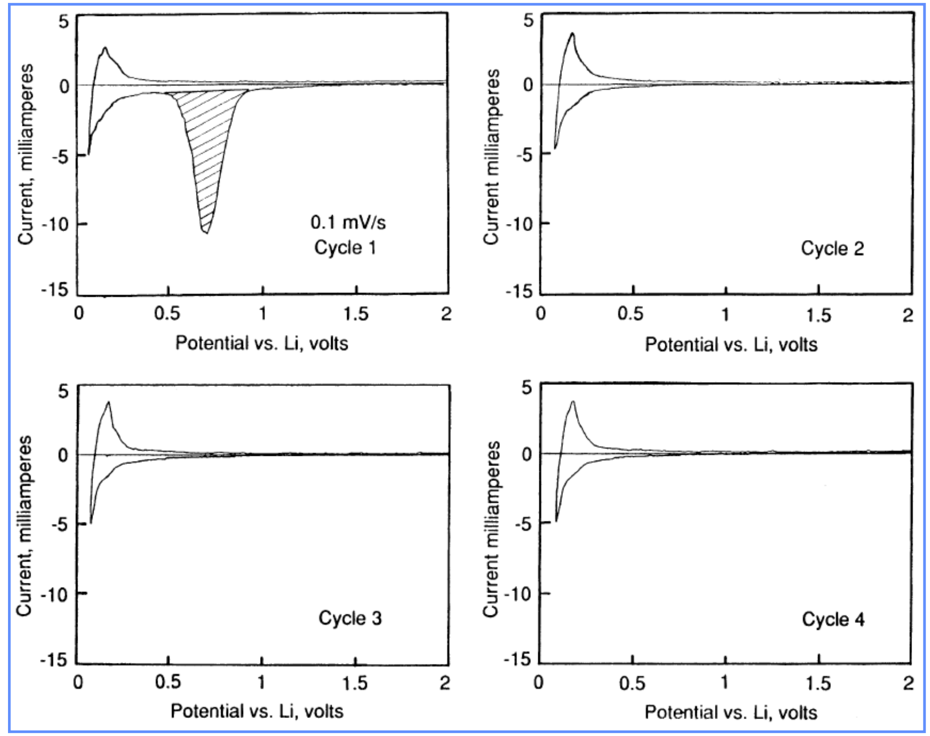
Formation of the SEI…solvent reduction on the negative
(ethylene carbonate)



- **Example reactions only…many others contribute to the formation of the solid electrolyte layer**
  - For computed IR spectra of surface species in an EC electrolyte, see S. Matsuta, T. Asada, and K. Kitaura. *J. Electrochem. Soc*. 147(2000)1695-1702…dimers found to be lowest energy
  - Experimental FTIR data indicates predominance of $[Li(OCOO)CH_2]_2$ for EC and EC+DEC systems with 1M $LiPF_6$, see C. R. Yang, Y. Y. Wang, C. C. Wan, *J. Power Sources*, 72(1998)66.

On the importance of Coulombic efficiency η_I

$\frac{}{2}$ $LiCH_2CH_2OCO_2Li$



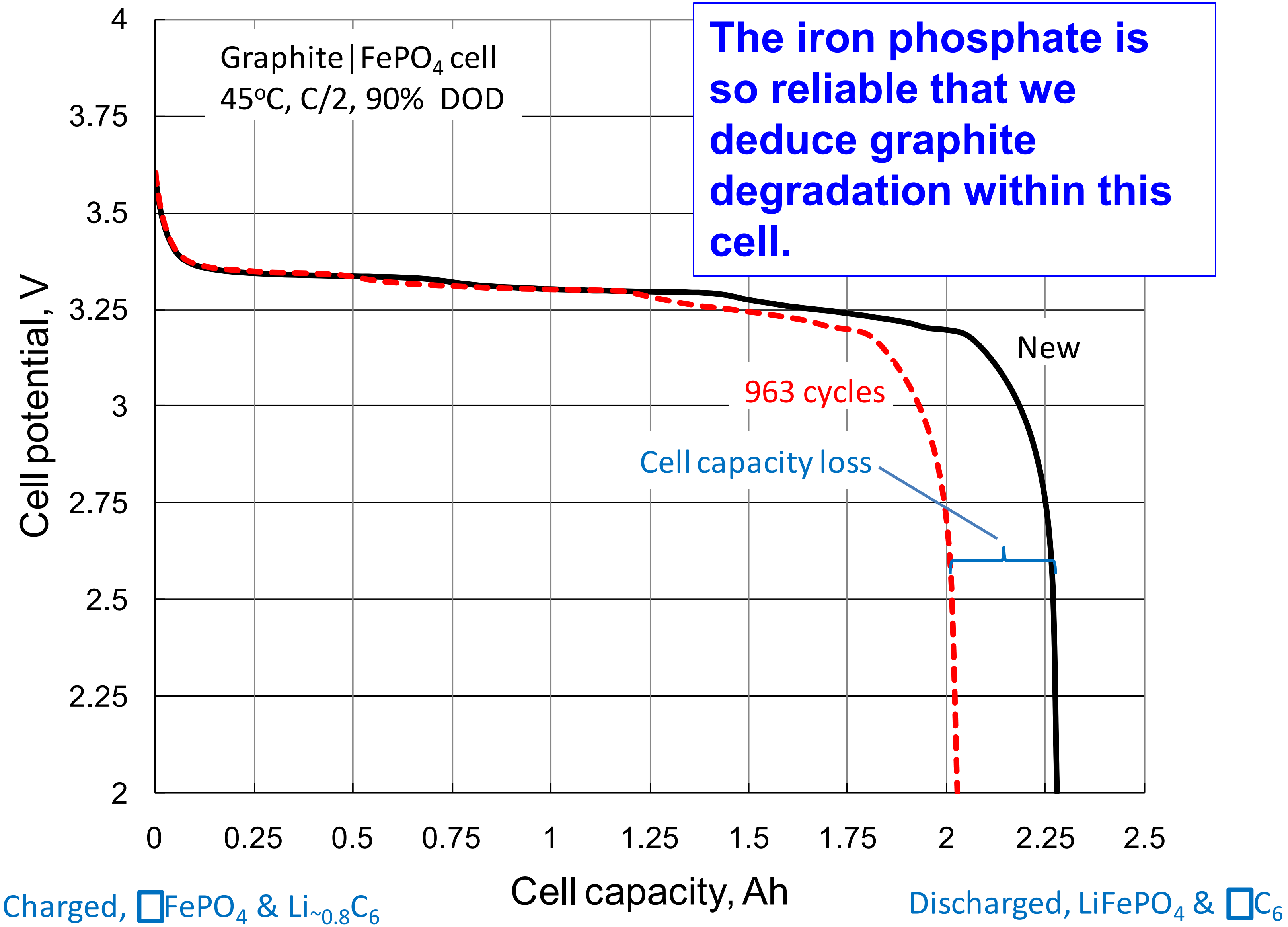
Cycle	Capacity
1	$(Ah_0)\eta_I$
2	$[(Ah_0)\ \eta_I\ ]\eta_I$
3	$[(Ah_0)\ \eta_I\ \eta_I\ ]\eta_I$
⋮	
N	$(Ah_0)(\eta_I)^N$

For N = 5000 cycles and a 12/16 or 75% capacity retention, the current efficiency per cycle must be such that

$$[Ah_0(\eta_I)^N\ ]/Ah_0 > 0.75, \text{ or } \eta_I > (0.75)^{(1/5000)}\text{, hence } \eta_I > \mathbf{\underline{0.99994}}.$$

- This is why very low rates of lithium-consuming reactions can lead to premature cell failure. The rates can be so low that they are not measureable in terms of seeing current maxima associated with solvent reduction.
- Note: high capacity negatives (Si, Sn based)…large challenge!

Graphite|iron-phosphate cell…excellent power density, life, and potential for low cost. Challenged on energy density.



The iron phosphate is so reliable that we deduce graphite degradation within this cell.

Journal of The Electrochemical Society, **157** (4) A499-A507 (2010)
0013-4651/2010/157(4)/A499/9/$28.00 © The Electrochemical Society

Aging Mechanisms of $LiFePO_4$ Batteries Deduced by Electrochemical and Structural Analyses

Ping Liu,[a,*,z] John Wang,[a] Jocelyn Hicks-Garner,[a,*] Elena Sherman,[a] Souren Soukiazian,[a] Mark Verbrugge,[b,*] Harshad Tataria,[b,*] James Musser,[c] and Peter Finamore[c]

[a] *HRL Laboratories, LLC, Malibu, California 90265, USA*
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[c] *John Deere Southeast Engineering Center, Charlotte, North Carolina 28241, USA*

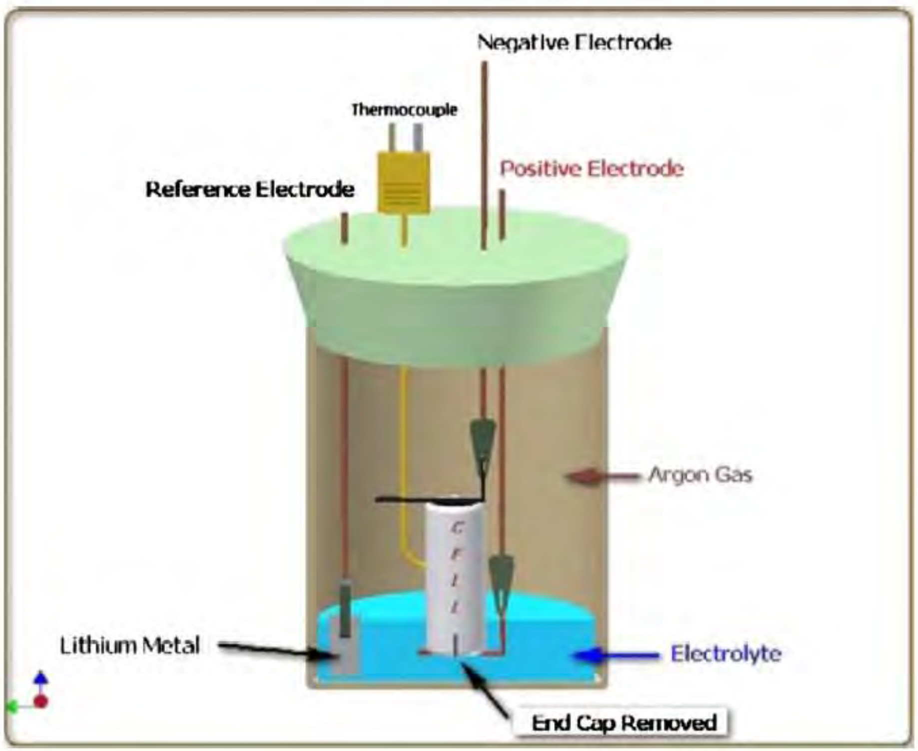

Figure 1. (Color online) Schematic of an in situ reference electrode measurement setup. The end cap of the cylindrical cell was removed. The cell is immersed in a liquid electrode, and a lithium reference electrode is located next to the cell. During constant current charge and discharge, the potential of the carbon negative can be recorded.

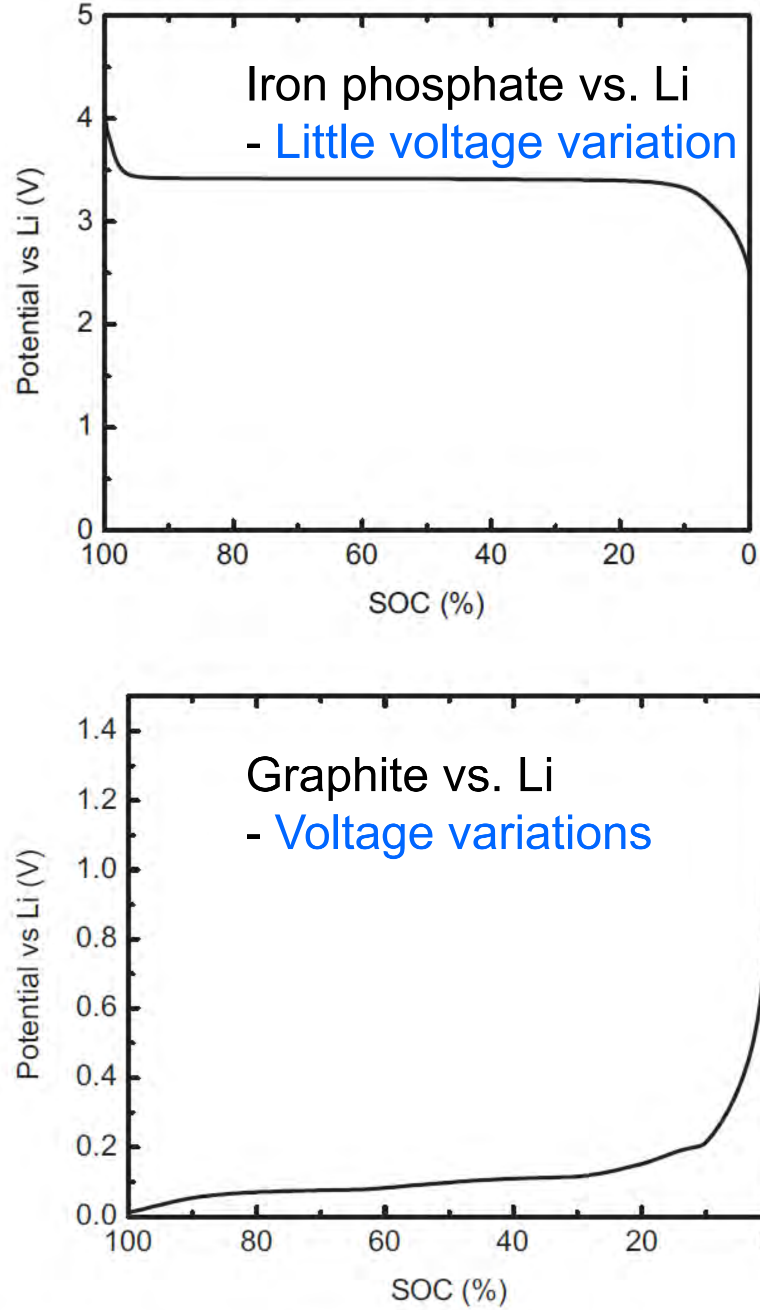

Figure 5. C/20 discharge curves for $LiFePO_4$ (top) and graphitic carbon (bottom) when measured against metal lithium.

Analysis of $FePO_4$/ graphite cells

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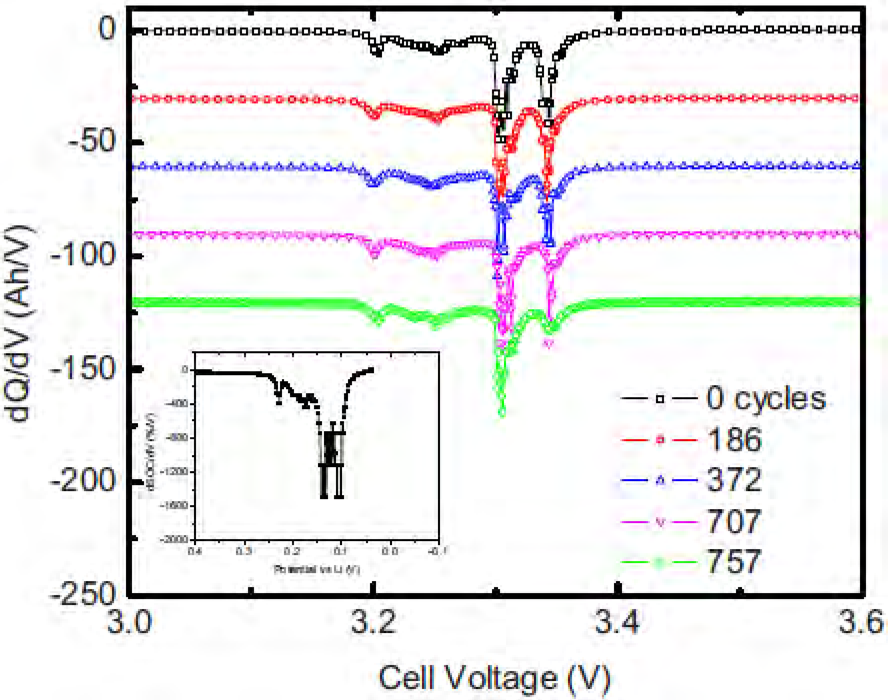
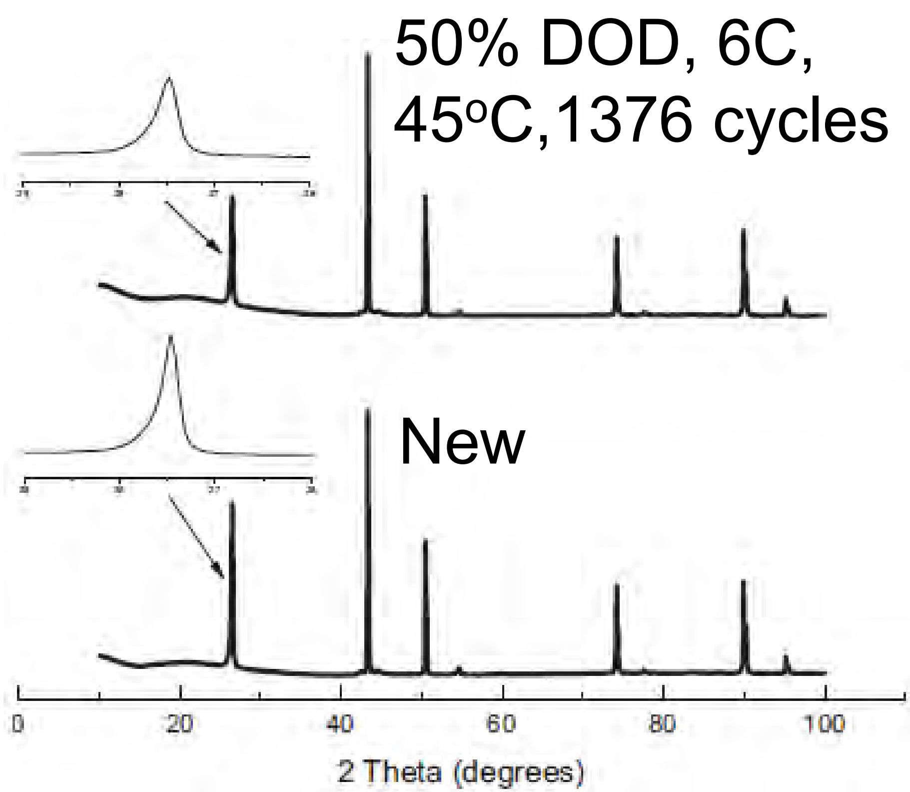
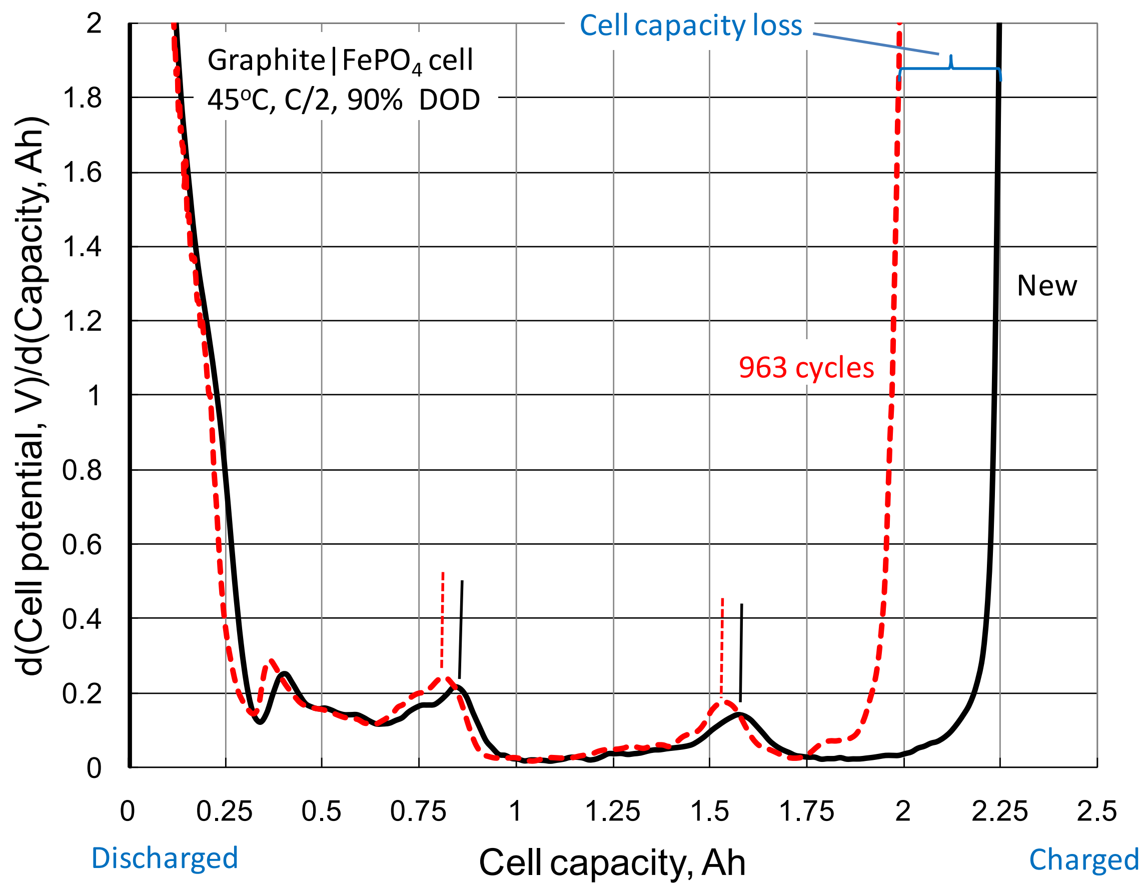




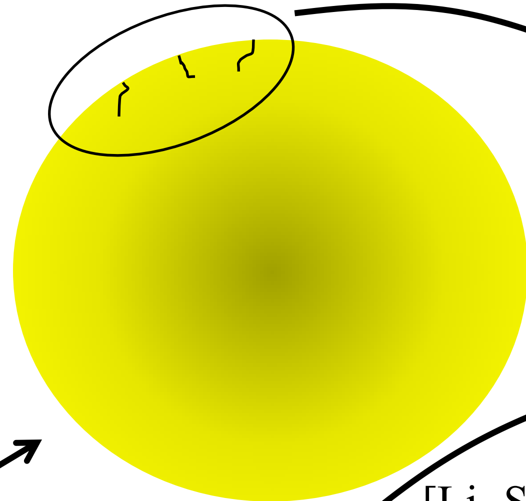
- Conventional differential voltage spectroscopy, but here on the full $FePO_4$-graphite cell
- Peaks result from graphite staging (next slide)
- Peak broadening indicating reduction in crystallite size

Utility of dV/dQ vs Q, uniform shifting of peaks for graphite/$FePO_4$ cells

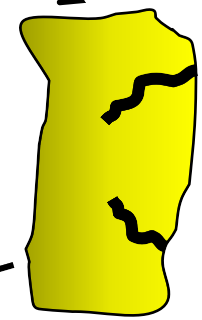


- ❑ **Same as previous plot with the exception that origin now is at the fully discharged state…clear that distance between graphite peaks is nearly constant**
- ❑ **Conclusion: lithium consumption (at the negative electrode surface) is leading to capacity decline**

Chemical-mechanical degradation at the negative electrode

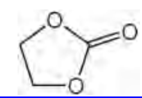


Cracks
via cycling



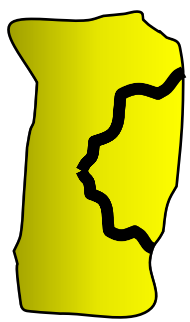
SEI forms on newly exposed surfaces (cracks)

$$[\mathrm{Li\text{-}S}] + \mathrm{R\text{-}H\text{-}O} \rightarrow \mathrm{SEI} + \mathrm{gasses} + \ldots$$

$[\mathrm{Li\text{-}S}] +$  $\rightarrow \mathrm{S} + \mathrm{LiCO_3} \downarrow + \mathrm{H_2C{=}CH_2} \uparrow$

Increased disorder and cracking.
d_{002} peak-width at half max amplitude increases with cycling. Supported by Raman analyses.

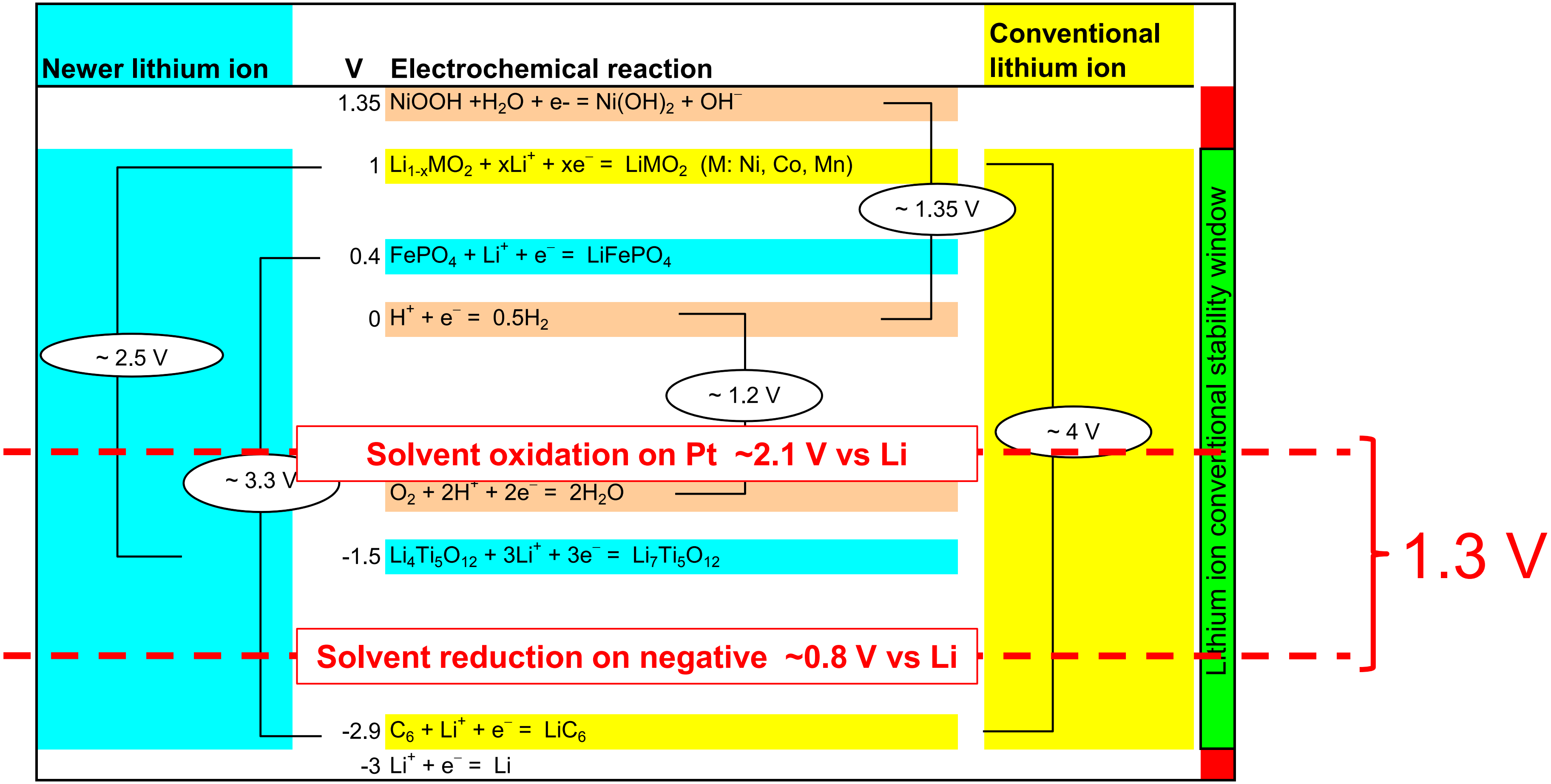
- Consistent with loss of active lithium.



Electrode isolation and loss of active material when cracks join

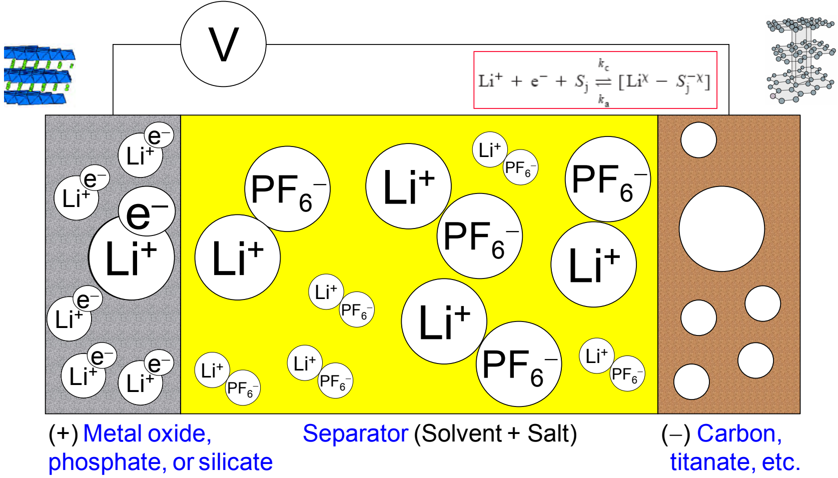
- Consistent with additional loss of negative capacity

Summary: *role of surface layers on + and –*



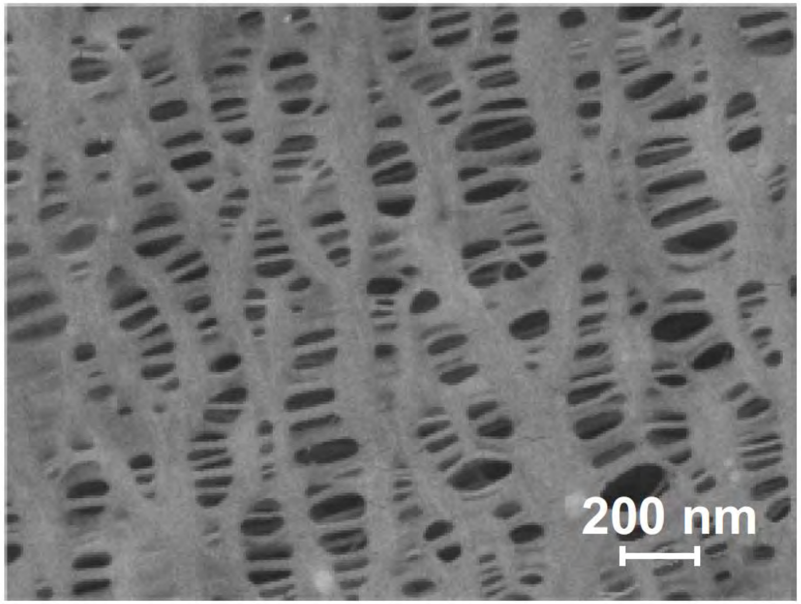
- **Underscores the importance of the SEI**
  - Disruption of the SEI (e.g., due to dilation, crack propagation, etc.) is deleterious to cell life…even low reaction rates are a problem
    - – Loss of Li
    - – Gas generation

Separators and ceramics



- **Function**
  - "Zero" electronic conduction
    - Requires mechanical integrity
    - Low porosity helps to mitigate dendrite shorting
  - Facile ionic conduction
    - High porosity is desired
    - Wetted by conventional solvent+salt systems (e.g., $LiPF_6$ in EC+DEC)
- **Strong element of cell abuse-tolerance strategy**

- **Current separator costs are significant**
  - Poly(propylene) and poly(ethylene)
- **Relatively new development: ceramic enhancement**

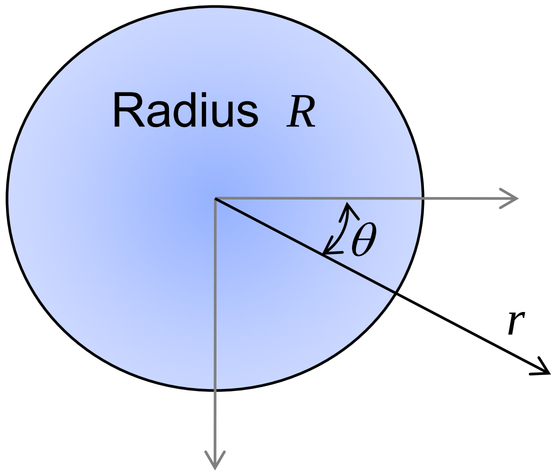


Conventional separator
PP or PP|PE|PP

Ceramic (alumina and/or silica) enhanced separator

- More on expansion and contraction of active materials due to diffusion induced stress
  - ➢analogous to thermoelasticity analyses of ceramics

Potential step, $\Theta_0 \rightarrow \Theta_R$



$$S_1 = \frac{1-\dfrac{(1+\nu)}{E}\dfrac{K^s}{R}}{1+\dfrac{(1-2\nu)}{E}\dfrac{2K^s}{R}}\ ,\quad S_2 = -\frac{2\dfrac{\tau^0}{R}}{1+\dfrac{(1-2\nu)}{E}\dfrac{2K^s}{R}}$$

Surface modulus K^s Surface stress τ^0

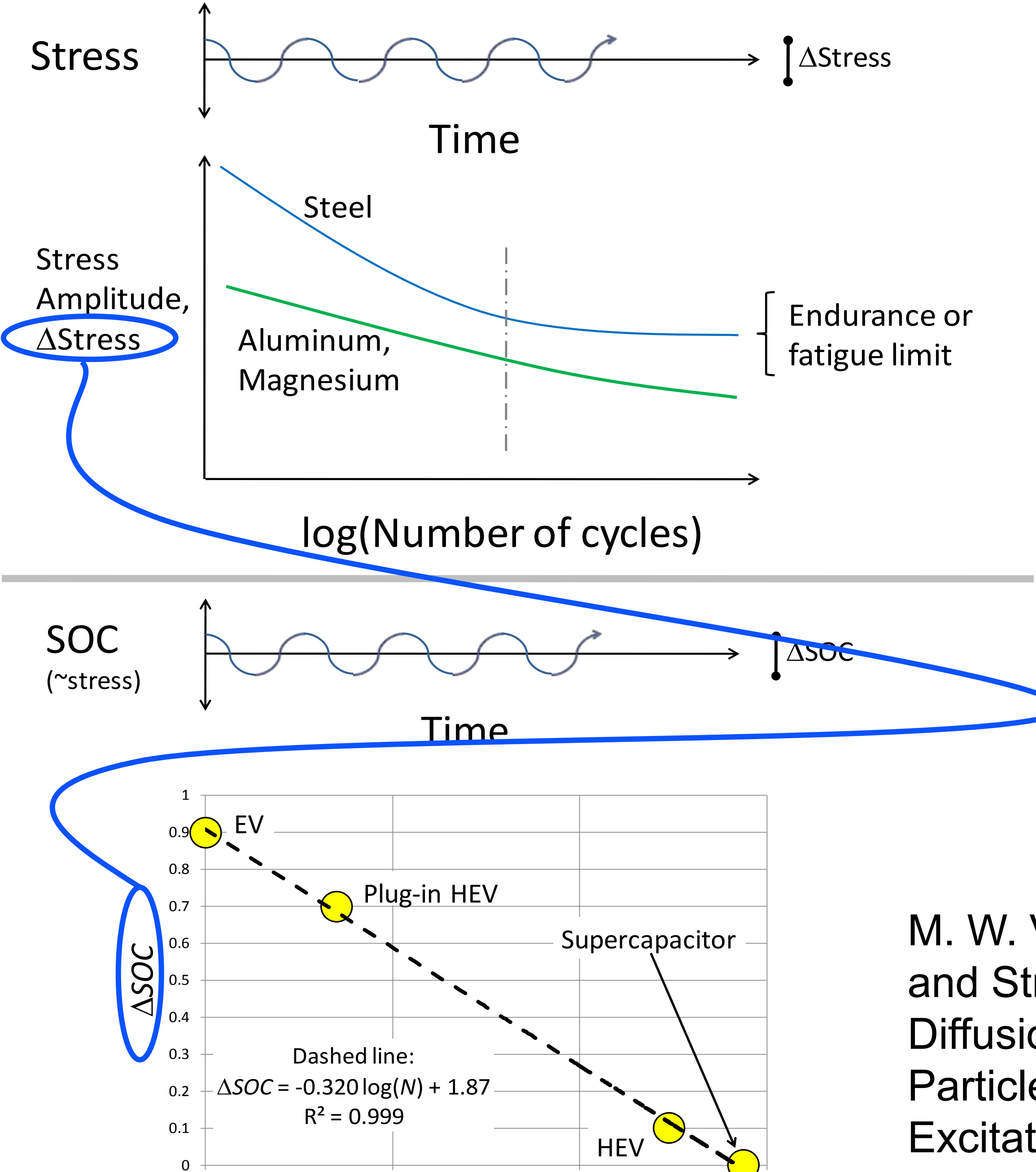
1. **Influence of both terms vanish as $R \rightarrow \infty$**
2. **Results now consistent with nano-particle and thin film electrodes yielding enhanced cycle life**

$$\frac{3(1-\nu)}{E\Omega C_S}\sigma_r(t,r) = \frac{2}{3}\Theta_R(S_1-1) - 4(\Theta_R-\Theta_0)\sum_{n=1}^{\infty}\left[\frac{S_1}{(n\pi)^2} + (-1)^n\frac{\sin(n\pi x) - n\pi x\cos(n\pi x)}{(n\pi x)^3}\right]e^{-n^2\pi^2\tau} + \frac{3(1-\nu)}{E\Omega C_S}S_2$$

$$\frac{3(1-\nu)}{E\Omega C_S}\sigma_\theta(t,r) = \frac{2}{3}\Theta_R(S_1-1) - 2(\Theta_R-\Theta_0)\sum_{n=1}^{\infty}e^{-n^2\pi^2\tau}\left[\frac{2S_1}{(n\pi)^2} - (-1)^n\frac{(\sin n\pi x - n\pi x\cos n\pi x)}{(n\pi x)^3} + (-1)^n\frac{\sin(n\pi x)}{n\pi x}\right] + \frac{3(1-\nu)}{E\Omega C_S}S_2$$

- **For the stress functions, the transient terms are proportional to ΔSOC (ΔSOC ∝ stress)**

Y-T Cheng and M. W. Verbrugge, "The Influence of Surface Mechanics on Diffusion Induced Stresses within Spherical Nanoparticles," *J. Appl. Phys.*, 104(2008)83521.



- Direct analogy to the lower cycle-life fatigue…stress amplitude is replaced by ΔSOC
- Model result: **maximum stress is proportional to the maximum difference in SOC, or ΔSOC**

M. W. Verbrugge and Y-T. Cheng, "Stress and Strain-Energy Distributions within Diffusion-Controlled Insertion-Electrode Particles Subjected to Periodic Potential Excitations," *J. Electrochem. Soc.*, 156(2009)A927.

Problem statement

Single Particle Model…particle diffusion resistance dominates

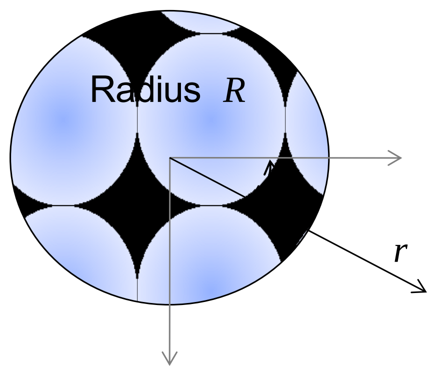
- Phenomenological stress-strain relations (cf. thermoelasticity)

$$\varepsilon_r = \frac{1}{E}\left(\sigma_r - 2\nu\sigma_\theta\right) + \frac{1}{3}\Omega c$$

$$\varepsilon_\theta = \frac{1}{E}\left[\left(1-\nu\right)\sigma_\theta - \nu\sigma_r\right] + \frac{1}{3}\Omega c$$

- Mechanical equilibrium

$$\frac{d\sigma_r}{dr} + 2\frac{\sigma_r - \sigma_\theta}{r} = 0$$



- Boundary conditions
  - Finite stresses at particle center
  - Surface

$$\sigma_r\Big|_R = -\frac{2}{r}\sigma_\theta^{surf}\Big|_R = -\frac{2}{r}\left(\tau^0 + K^s\varepsilon_\theta\right)\Big|_R$$

    - surface tension and surface modulus

- Phenomenological guest lithium (intercalate) diffusion

$$\frac{\partial c}{\partial t} = D\left(\frac{\partial^2 c}{\partial r^2} + \frac{2}{r}\frac{\partial c}{\partial r}\right)$$

$$c(0,r) = c_0$$

$$c(t,R) = \begin{cases} c_{\max} & \text{for } t - nt_{cycle} < t_1 \\ c_{\min} & \text{for } t - nt_{cycle} > t_1 \end{cases}$$

$$c(t,0) = \textit{finite}$$

- The equation system can be solved analytically
  - ✓ Simplified problem statement
  - ✓ Decoupled concentration problem
  - ✓ No variable physical properties
  - ✓ Idealized (spherical geometry)

Stress profiles (stationary state)

- **Define**

$$S_1 = \frac{1 - \frac{(1+\nu)}{E}\frac{K^s}{R}}{1 + \frac{(1-2\nu)}{E}\frac{2K^s}{R}} \quad \text{and} \quad S_2 = -$$

- **Solution.** Simple in structure and similar to step
  Analogous expressions result for the second port

$$\frac{3(1-\nu)}{E\Omega(c_{\max} - c_{\min})}\sigma_x(\tau, x; \tau < T_1) = \frac{3(1-\nu}{E\Omega(c_{\max} -}$$

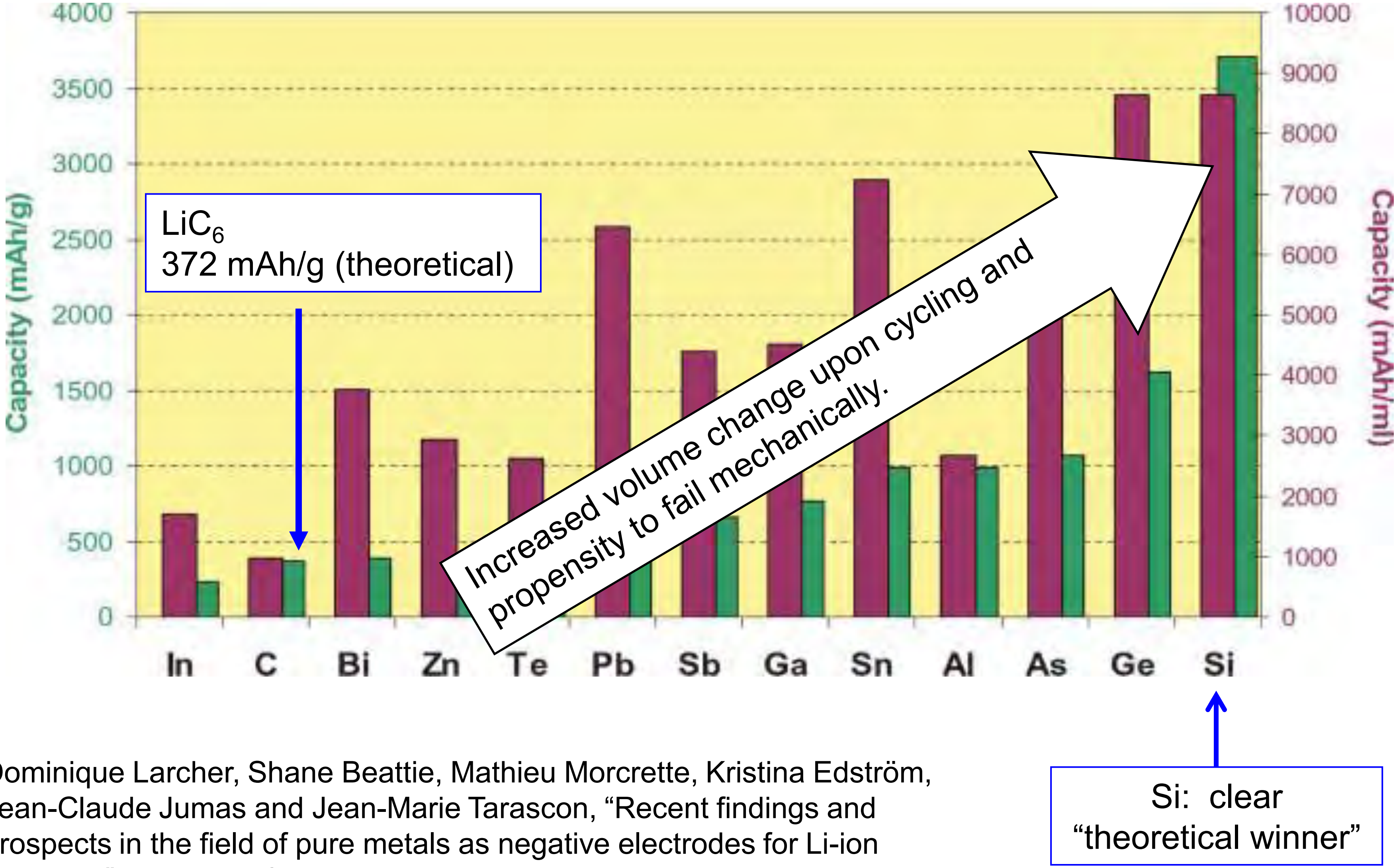
$$-4\sum_{j=1}^{\infty}\left[\frac{e^{-\beta_j(T+\tau-T_1)} - e^{-\beta_j \tau}}{e^{-\beta_j T} - 1}\right]\left[\frac{S_1}{(j\pi)^2} + (-\right.$$

$$\frac{3(1-\nu)}{E\Omega(c_{\max} - c_{\min})}\sigma_\theta(\tau, x; \tau < T_1) = \frac{3(1-\nu)}{E\Omega(c_{\max} - c_{\min})}S_2 + \frac{2}{3}(S_1 - 1)\frac{c_{R,\max}}{(c_{\max} - c_{\min})}$$

$$-2\sum_{j=1}^{\infty}\left[\frac{e^{-\beta_j(T+\tau-T_1)} - e^{-\beta_j \tau}}{e^{-\beta_j T} - 1}\right]\left[\frac{2S_1}{(j\pi)^2} - (-1)^j\frac{\sin(j\pi x) - j\pi x\cos(j\pi x)}{(j\pi x)^3} + (-1)^j\frac{\sin(j\pi x)}{j\pi x}\right]$$

Stresses are proportional to ΔSOC

Current and next generation negative electrodes



Dominique Larcher, Shane Beattie, Mathieu Morcrette, Kristina Edström, Jean-Claude Jumas and Jean-Marie Tarascon, "Recent findings and prospects in the field of pure metals as negative electrodes for Li-ion batteries," *J. Mater. Chem.*, 2007, 17, 3759 – 3772

215th ECS Meeting | San Francisco, CA | May 24-29, 2009

❑ ***Why is there such interest in nano-scaled insertion & alloy electrodes?***

1. High power capability…shorter solute(intercalate) diffusion distance and higher surface area for electrochemical reaction
2. Absent surface-area-enhanced degradation reactions, smaller particles should be more (mechanically) robust

B — Batteries, Fuel Cells, and Energy Conversion

B1 — Battery / Energy Technology Joint General Session (M-F) — *N. J. Dudney, S. R. Narayanan, and C. R. Walk*

B2 — Battery Modeling at Cell Level (W) — *V. Srinivasan, A. M. Sastry, and K. Zaghib*

B3 — Characterization of Porous Materials 2 (Tu-W) — *B. Lakshmanan, G. Brisard, and A. Lasia*

B4 — Fuel Cells for Portable Power (M-Th) — *S. R. Narayanan, D. Chu, and E. Plichta*

B5 — Hydrogen Production, Transport, and Storage 3 (M-Tu) — *M. C. Williams, M. Heben, S. N. Lvov, M. Manivannan, P. H. Maupin, S. R. Narayanan, E. D. Wachsman, and J. W. Weidner*

B6 — Measurement and Diagnostics for Energy Systems (Tu) — *S. R. Narayanan, S. Mukerjee, R. Mukundan, and P. Strasser*

B7 — Nanostructured Materials for Energy Storage and Conversion (M-Th) — *K. Zaghib, K. M. Abraham, and C. Julien*

B8 — Photoelectrochemical Energy Conversion (M-Tu) — *K. Rajeshwar, J. Hupp, and B. Parkinson*

B9 — Advanced Materials and Concepts for Energy Harvesting (M-W) — *X. Zhou, M. Manivannan, and J. C. Nino*

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Summary

- ❑ **Automotive context**
- ❑ **Short overview/primer on lithium ion batteries**
- ❑ **Ceramics…now and in the future of lithium ion battery technology**
  - ➢ Positives…most are ceramics
  - ➢ Negatives…SEI; LTO
  - ➢ Separators…ceramic enhanced
  - ➢ Dilation and fatigue…classical ceramic investigations are proving helpful

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- ❑ **University of Kentucky**
  - YT Cheng
- ❑ **Brown University**
  - H. Haftbaradaran and H.Gao