Lifecycle Characterization of a High Magnetostriction Cobalt Iron Electroplating Chemistry

SAND2016-10348C

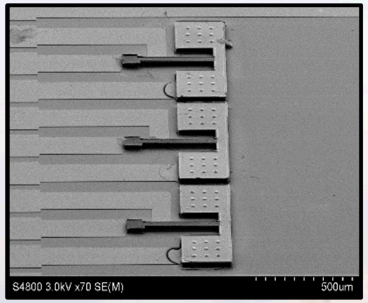
Christopher R. St. John

cstjohn@sandia.gov

http://www.sandia.gov/mstc/fabrication/metal-micromachining.html

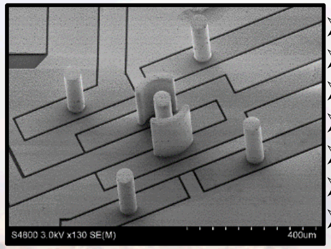
Metal Micromachining Team - MESA Light Labs

Lab #1 – Lithography



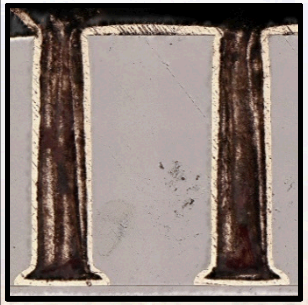
- mm range
- Can be electrofilled
- Micromolding or cast w/ PDMS
- Accommodates topography
- Polymer or glass

Lab #2 – Electroplating



- 2.5D structures
- Precision coatings
- Magnetic materials
- Insulating materials
- Conductive polymers
- >10:1 aspect ratio
- Multi-layer

Lab #3 – Cross-Sectioning and Planarization



- R_a as low as 50nm
- Wafer/die thinning
- Provide flat/parallel surface for processing
- Beveled edges
- Vacuum, pad, wax mount

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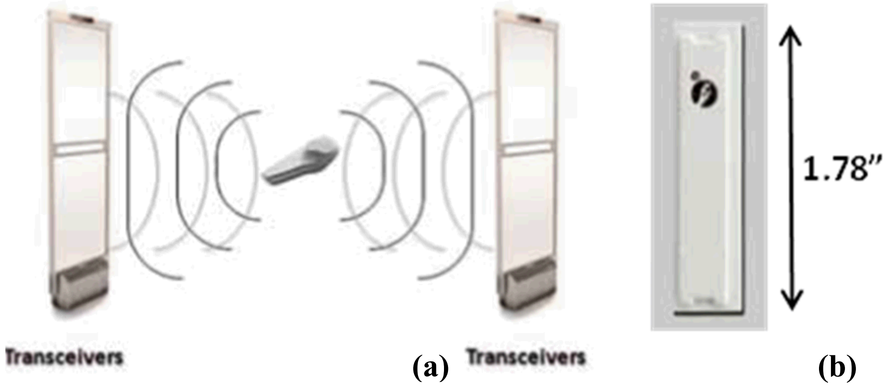
Magnetostrictive sensors background

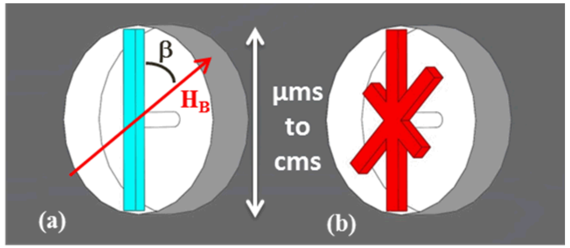
"Dumb Tag"



a) magnetic dipole antenna interrogation zone
b) magnetoelastic tag in plastic package

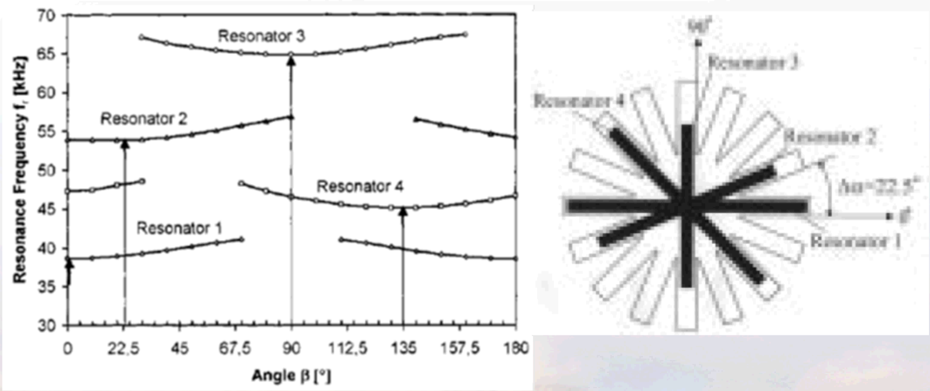
- Used to prevent theft in retail stores
- Operates via Joule magnetostriction, $\lambda_S = \Delta L/L$
- Generates an AC magnetic response signal when subjected to an externally applied AC magnetic interrogation signal
- Single frequency devices – can only convey information that a tag is magnetically activated (limited utility to this simple function)
- Made from strips of an amorphous magnetic material such as METGLAS™ ($Ni_{40-50}Fe_{40-50}Mo_{5-10}B_{1-5}$), with low magnetostriction (λ_S = 12 ppm)

"Smart Tag"



a) Single frequency resonator
b) Multi-Frequency resonator (3)

$$f_r = \frac{1}{2L}\left[\sqrt{\frac{\rho}{E_0} + \frac{9\lambda^2{}_S\rho\left(\left(|H_B|\cos(\beta)\right)\right)^2}{J_S H_A{}^3}}\right]^{-1}$$



M. Arndt and L. Kiesewetter, "Coded labels with amorphous magnetoelastic resonators," Magnetics, IEEE Transactions on, vol. 38, pp. 3374-3376, 2002.

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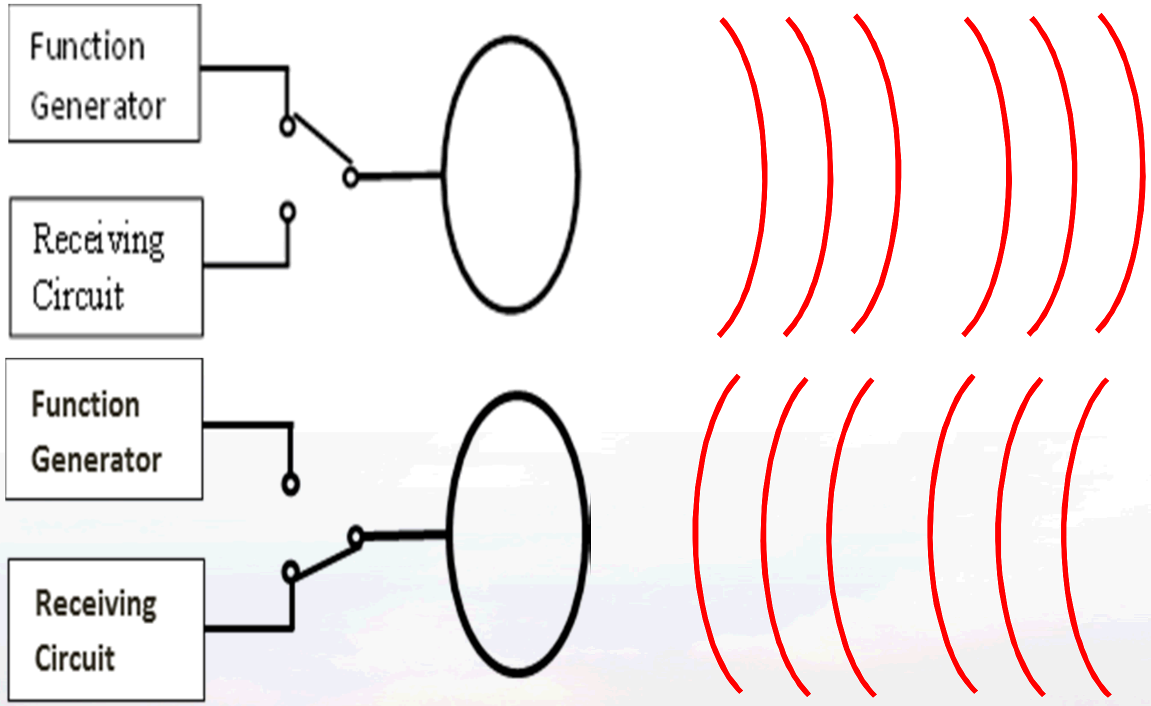
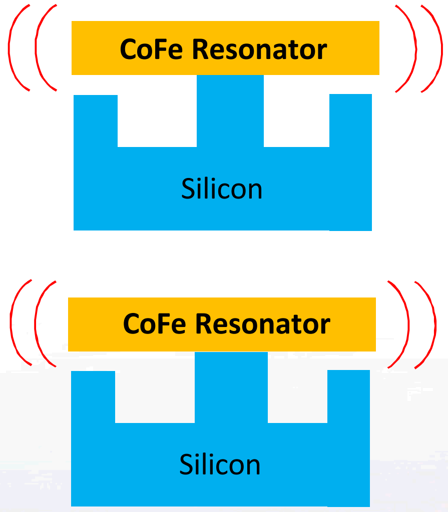
Magnetostrictive sensors function

$$f_r = \frac{1}{2L}\left[\sqrt{\frac{\rho}{E_0} + \frac{9\lambda_{sat.}{}^2\rho\left(\left(|H_B|\,cos(\beta)\right)\right)^2}{J_s H_A{}^3}}\right]^1$$

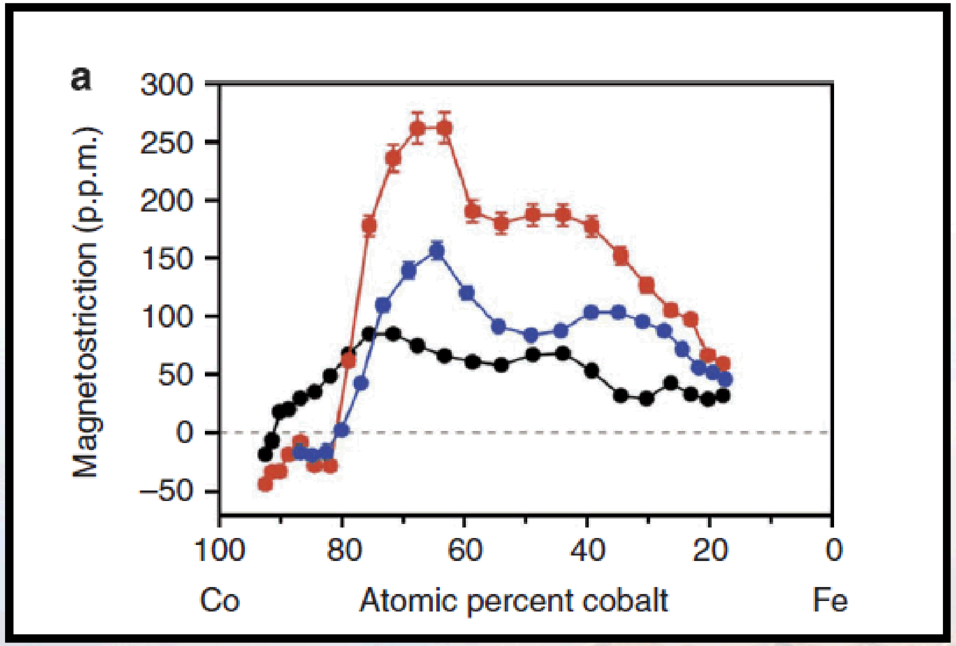


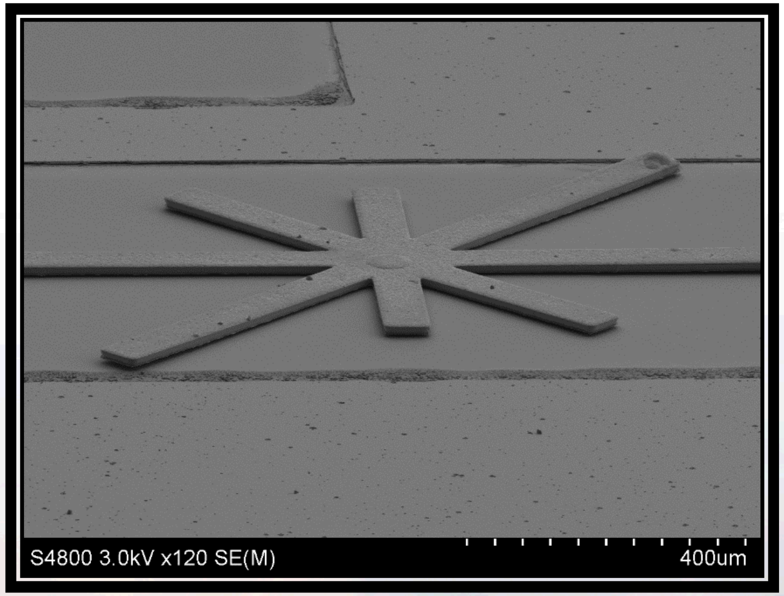


Cross section view

Electrodeposition to Realize CoFe devices

- Sputtered $Co_{0.7}Fe_{0.3}$ ratio as identified as 'giant magnetostriction' of >250 ppm by Hunter *et al* (Nature 2011)
- Electroplate CoFe for high magnetostrictive films
- Increase magnetostriction performance for MEMS applications and sensors





Hunter, D., et al. (2011). "Giant magnetostriction in annealed $Co_{1-x}Fe_x$ thin-films." Nature Communications **2**.

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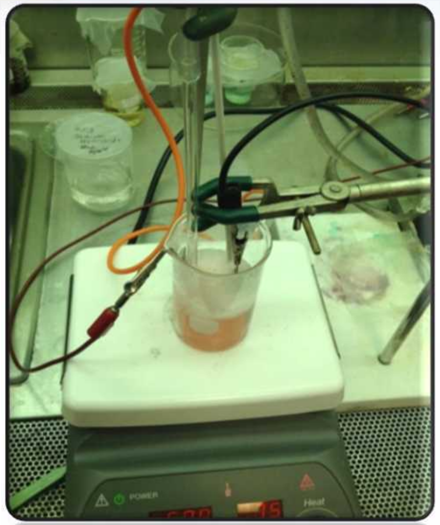
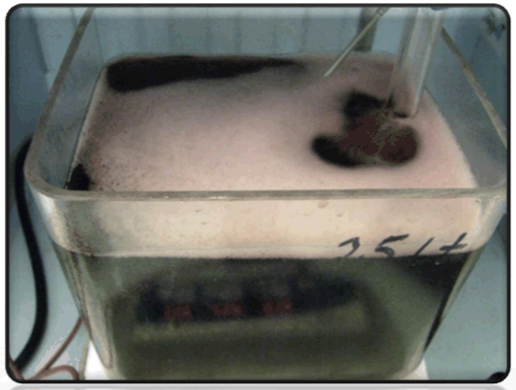
Scaling up to wafer-level chemistry



7 cm^2
sample

100 mm
wafer



Volume = 450 mL → 6x → Volume = 2.5 L

Surface Area = 57 cm^2 → 4x → Surface Area = 236 cm^2

Heat $Added_{Water}$ = 47 kJ → 6x → Heat $Added_{Water}$ = 262 kJ

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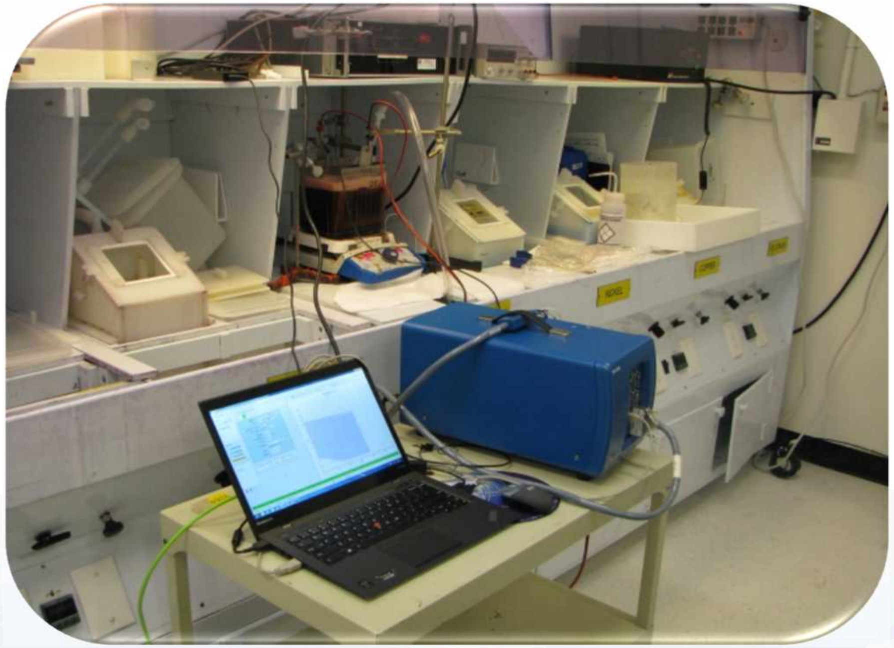
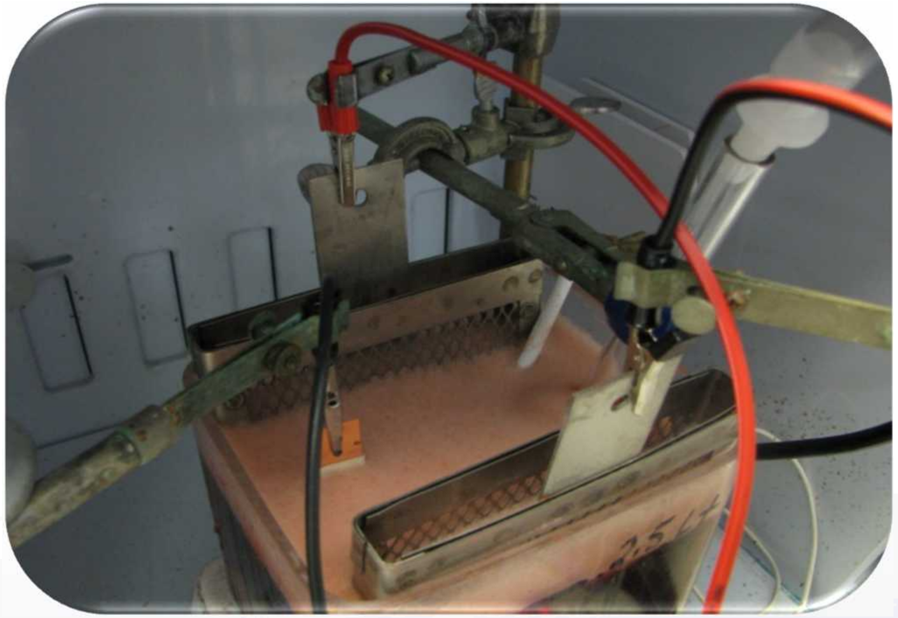
Experimental Setup

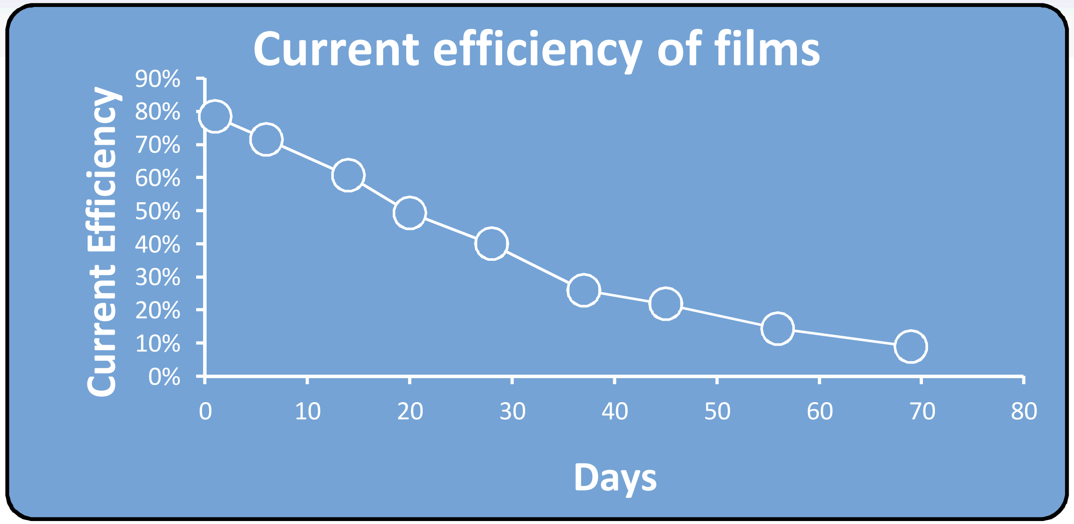


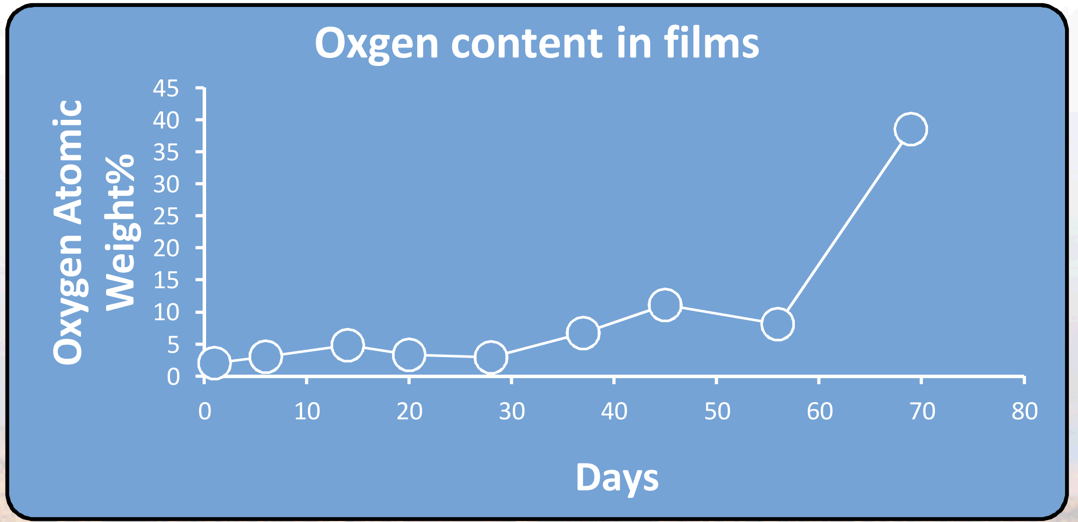


$$Current\ Efficiency = \frac{Actual\ thickness}{Theoretical\ thickness} = Film\ thickness/\frac{MW * I * t}{n * F * \rho * A}$$

2.5-liter bath magnetostriction results

Current Efficiency	Oxygen Atomic%
78%	2%
72%	3%
61%	5%
49%	3%
40%	3%
26%	7%
22%	11%
14%	8%
9%	39%





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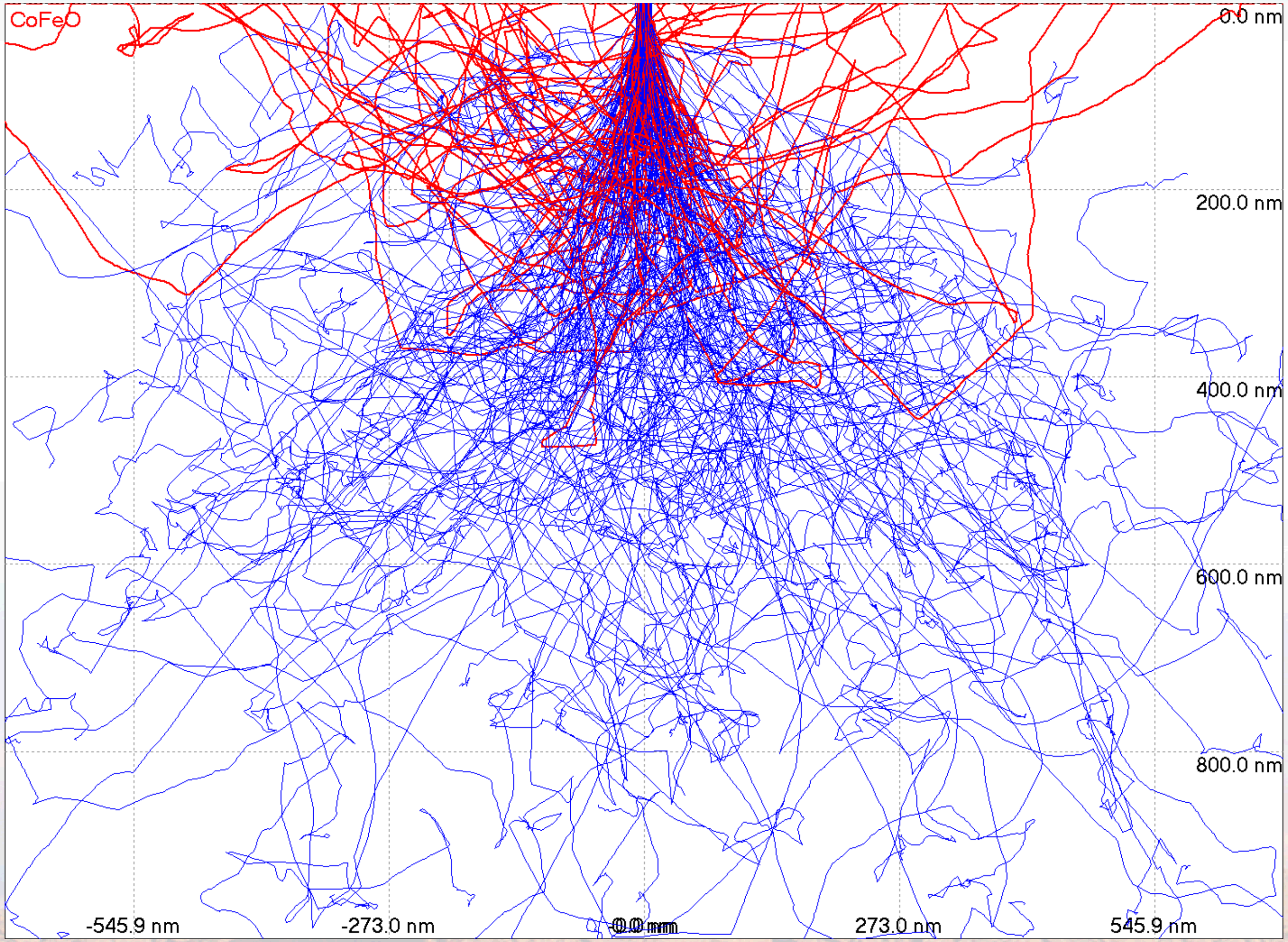
EDS Monte Carlo Simulation



Simulation in Casino 2.4.8.1
20 keV beam in $Co_{0.7}Fe_{0.21}O_{0.09}$

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Why the decrease in current efficiency?

Electrodeposition of iron alloy metals is problematic due to the undesired oxidation of Fe^{+2} to Fe^{+3}

Side Reactions:

$$Fe^{2+} \rightarrow Fe^{3+} + e^-$$

$$Fe^{3+} + O_2 \rightarrow Fe_2O_3$$

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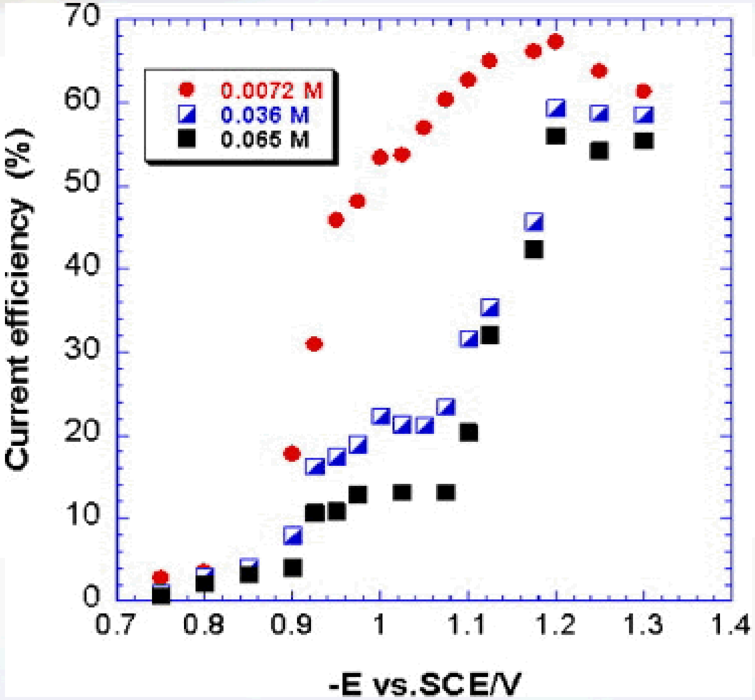
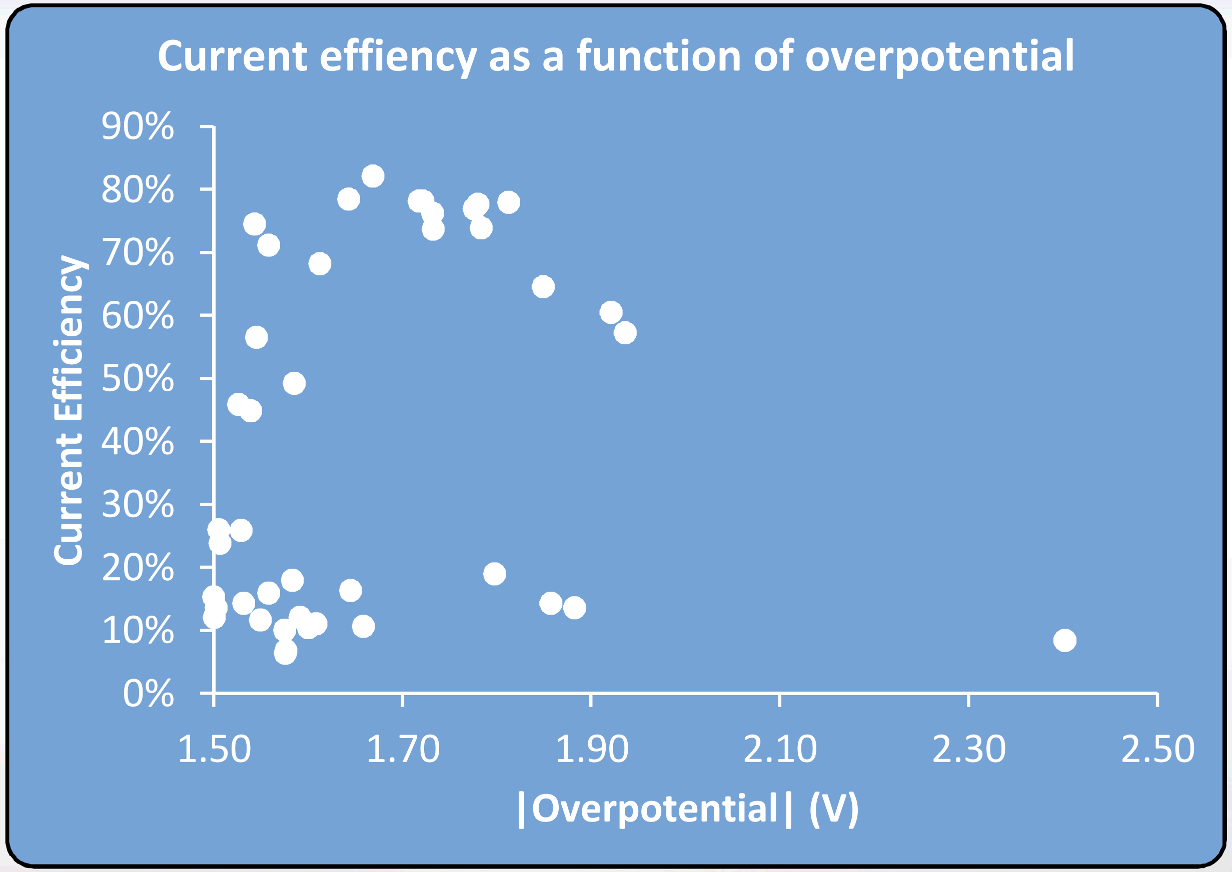
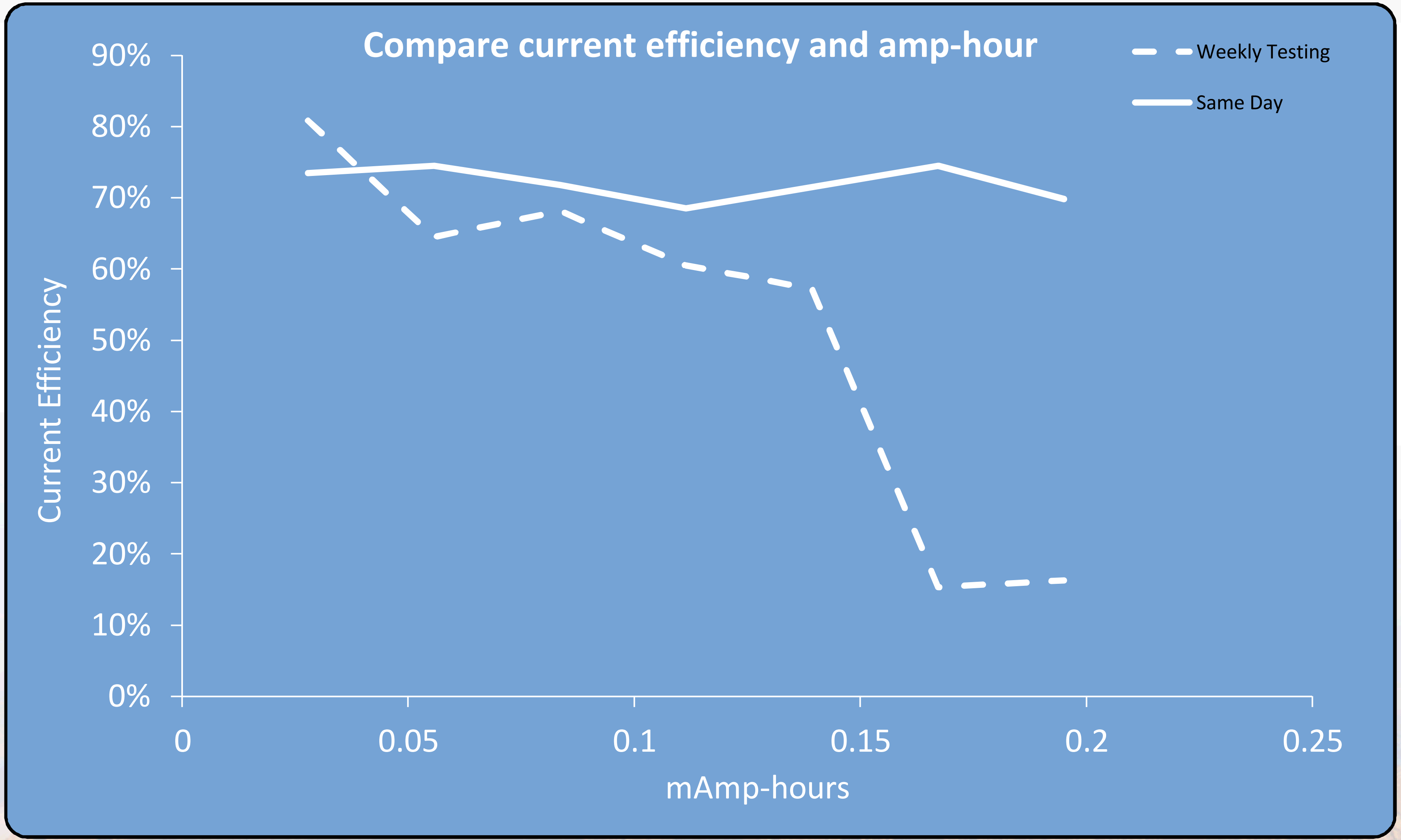




CE tends to decrease at the potentials more negative than -1.2 V vs. SCE, where reduction of H_2O starts more intensively

I. Tabakovic, S. Riemer, N. Jayaraju, V. Venkatasamy, J. Gong, "Relationship of Fe2+ concentration in solution and current efficiency in electrodeposition of CoFe films," Electrochimica Acta, vol. 58, pp. 25-32, 2011.

CoFe Bath aging results



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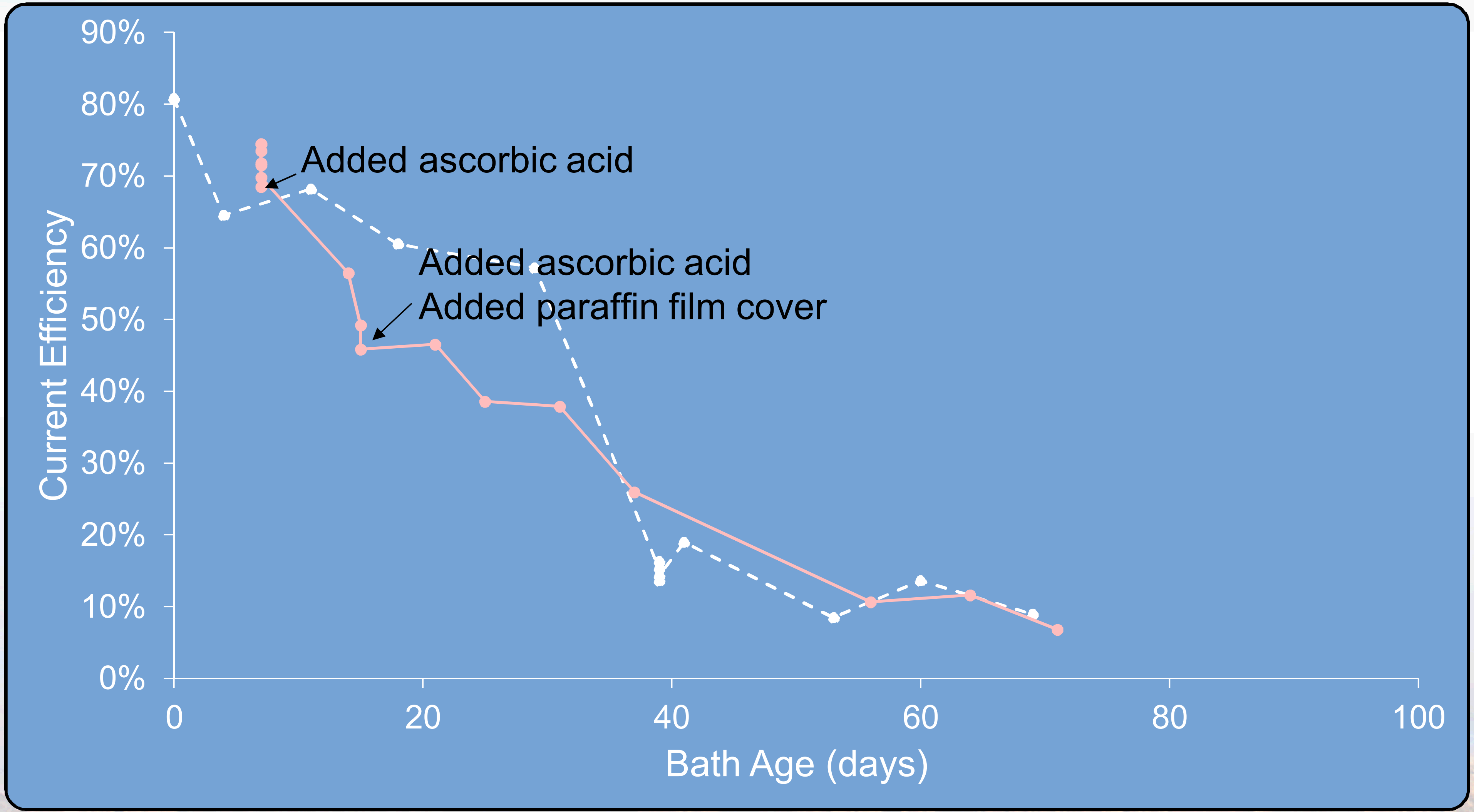
Chemical Additions



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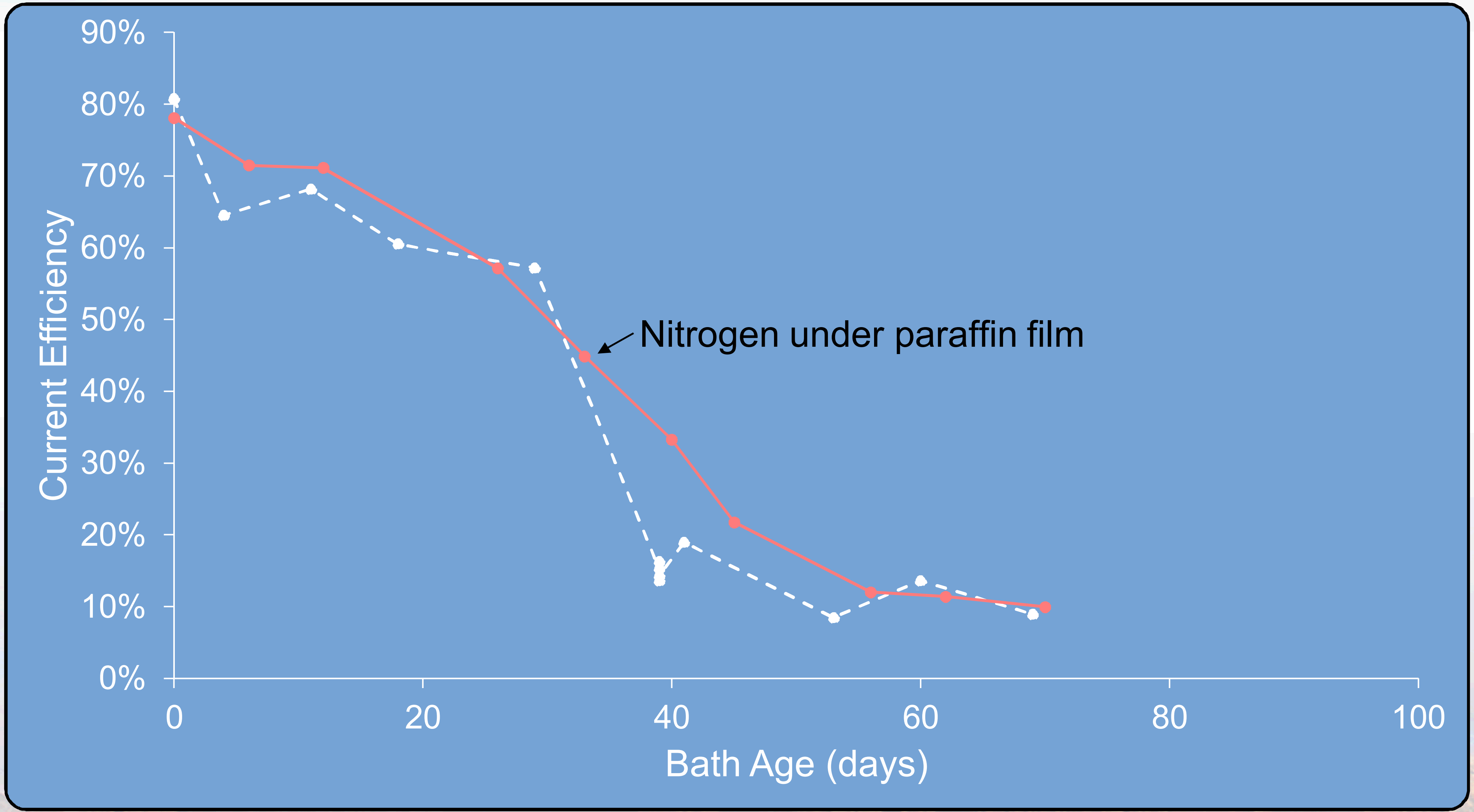
Parafilm Cover



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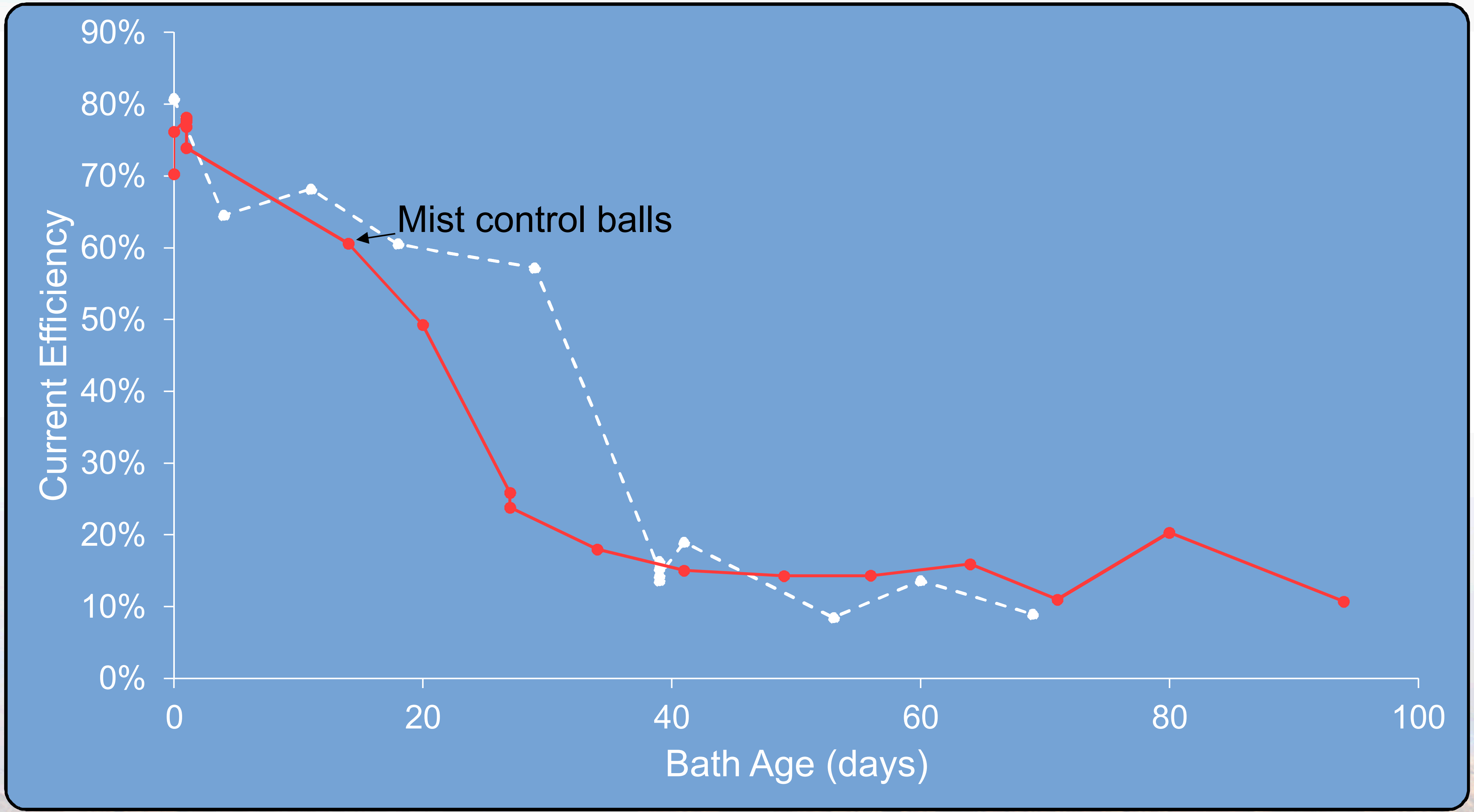
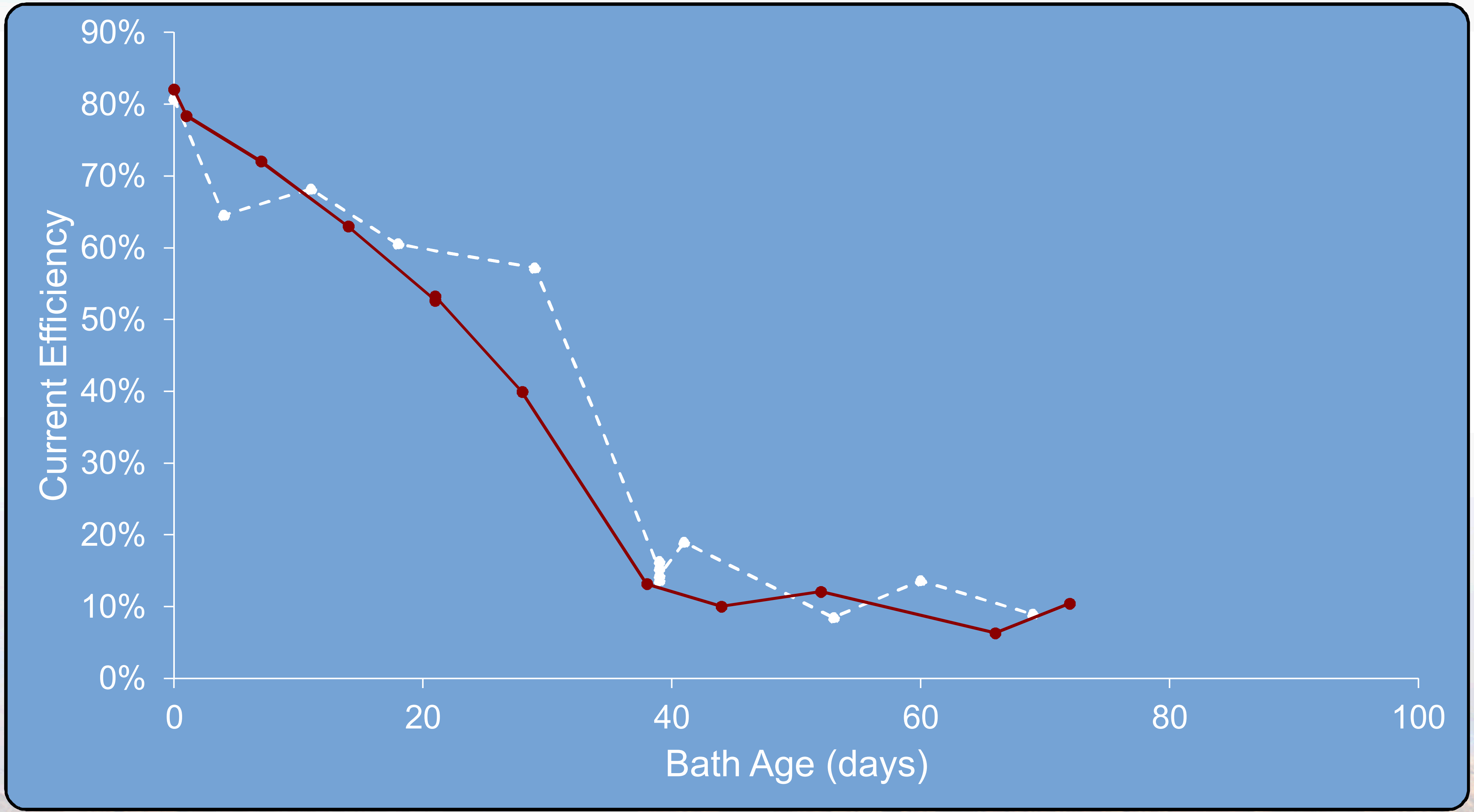
Parafilm Cover

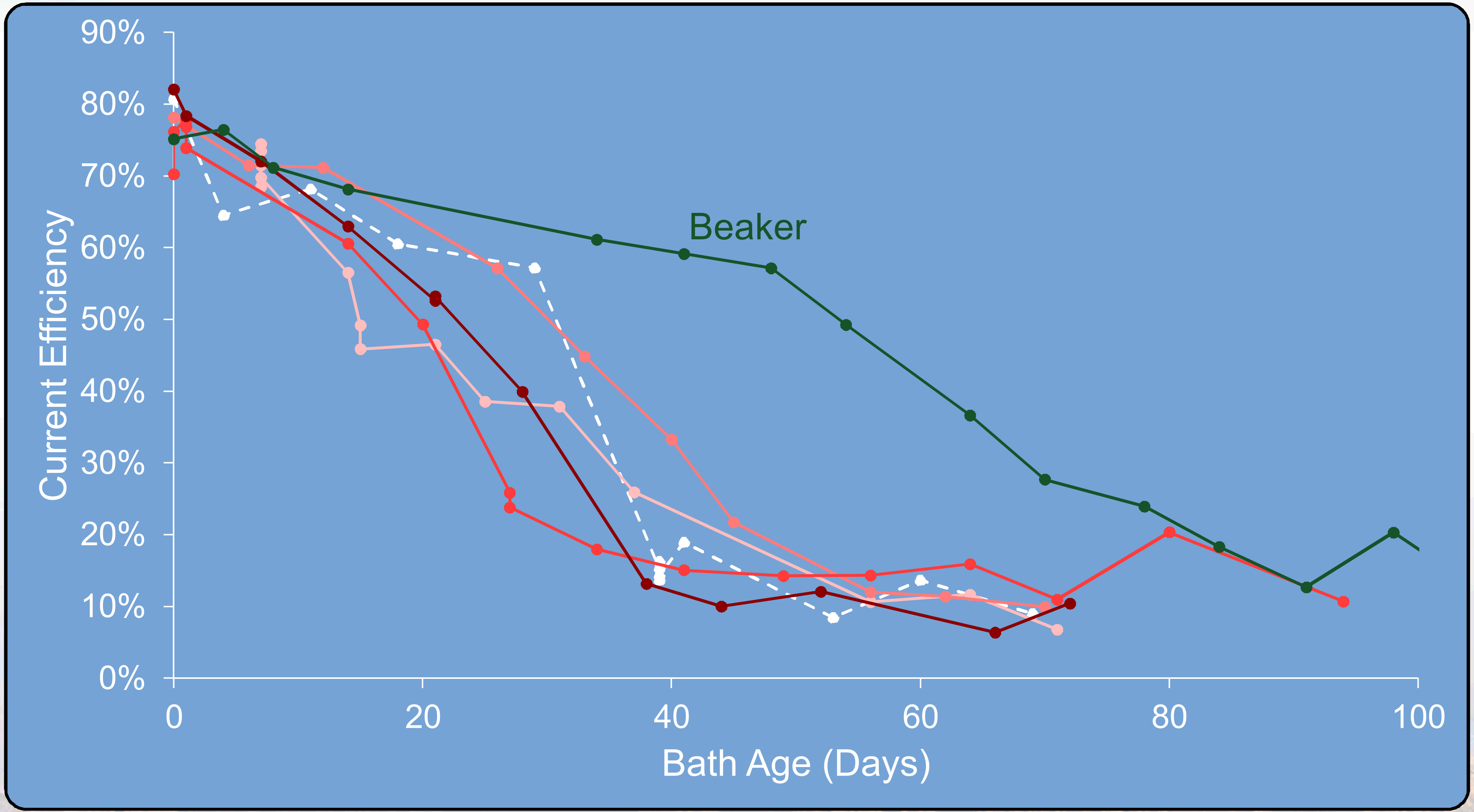


Dry Box



90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Current Efficiency
0
20
40
60
80
100
Bath Age (days)

All together



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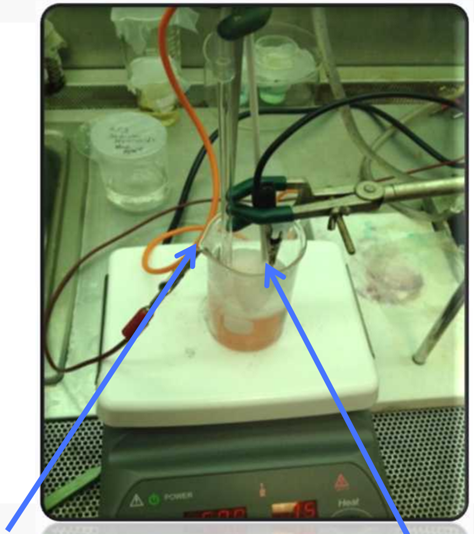
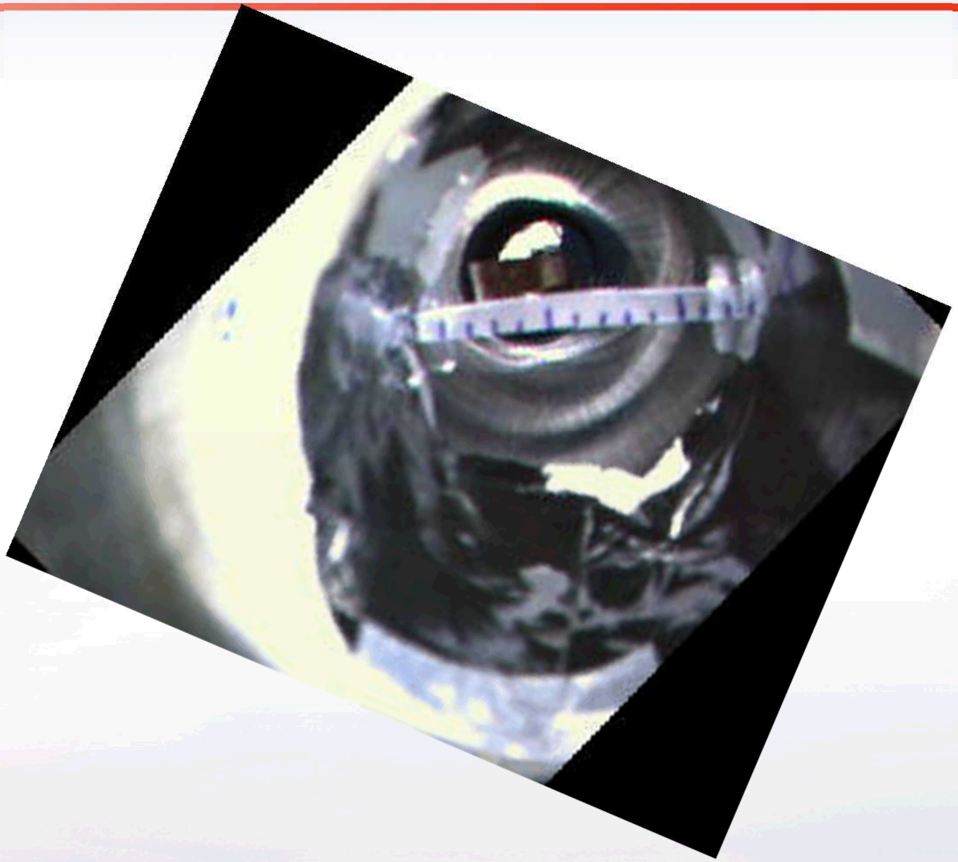
Aging results

Majority Storage	Useful Life (Days)	Amp-hours
Beaker	68	0.25
Hard cover	36	0.14
Hard cover	34	0.36
Paraffin	41	1.01
Paraffin	26	2.27
Dry box	28	2.89

Days	CE
5	70%
15	60%
23	50%
29	40%
35	30%
42	20%
68	10%

Magnetostriction testing





- CoFe plated material is silver color below

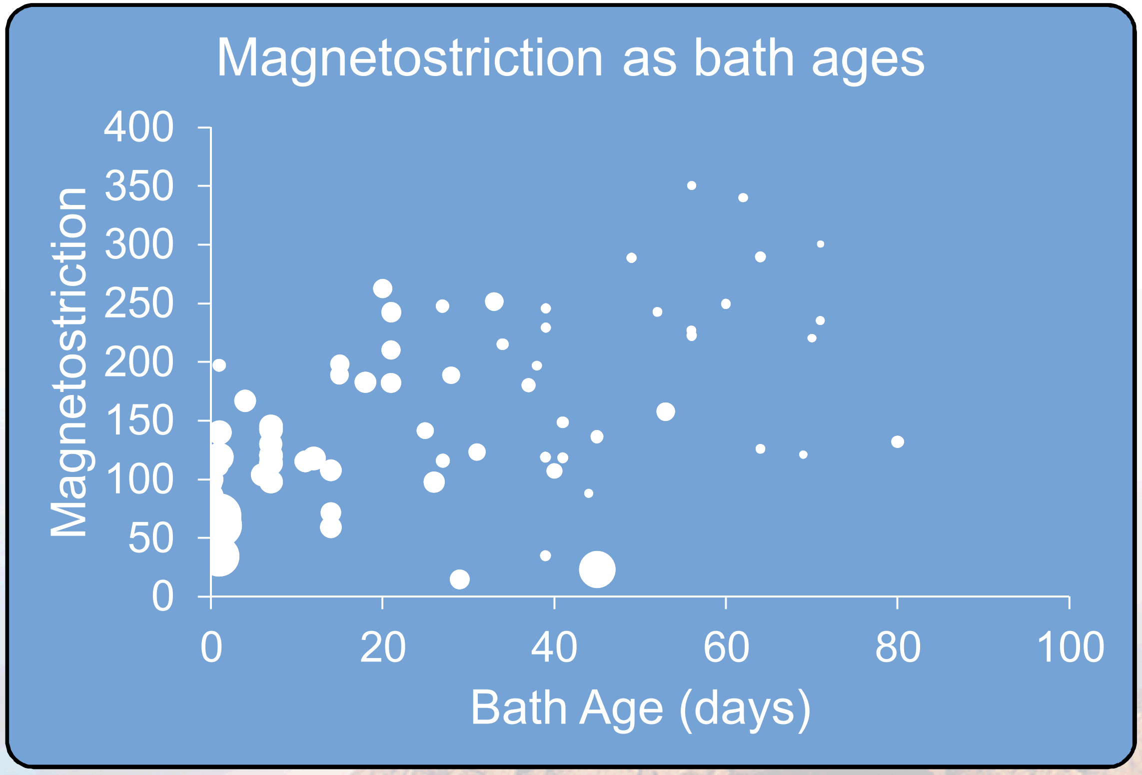
SJC3

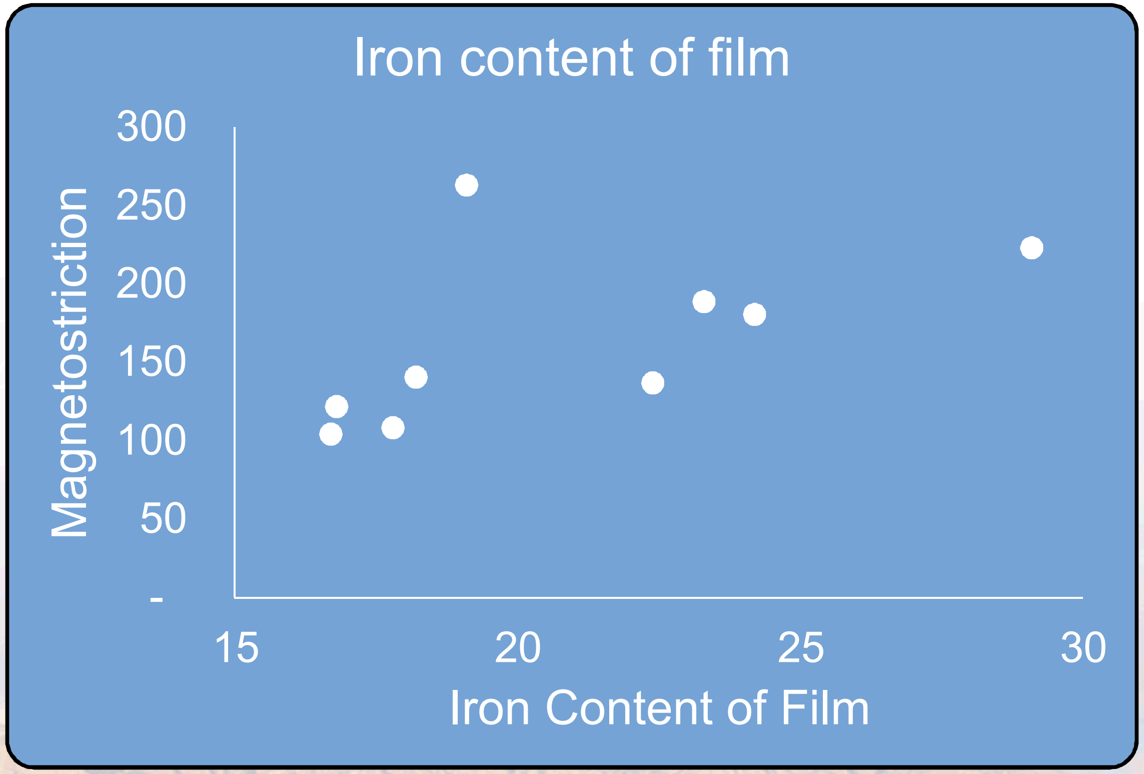
2.5-liter bath magnetostriction results

$$\lambda_{sat} = \frac{2 * (D_{||} + 0.167 * D_{||}) * Es * t_s^{\,2} * (1 + \upsilon_f)}{9 * Ef * L^2 * tf * (1 + \upsilon_s)}$$

Modified du Tremolet de Lacheisserie and Peuzin equation from Hunter et al

$D_{||}$ = Parallel displacement
E = Young's modulus
υ = Poisson's ratio



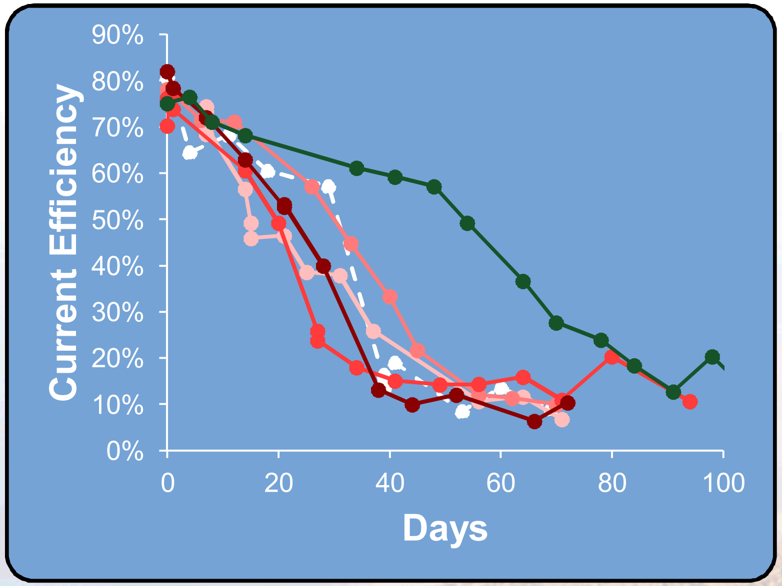


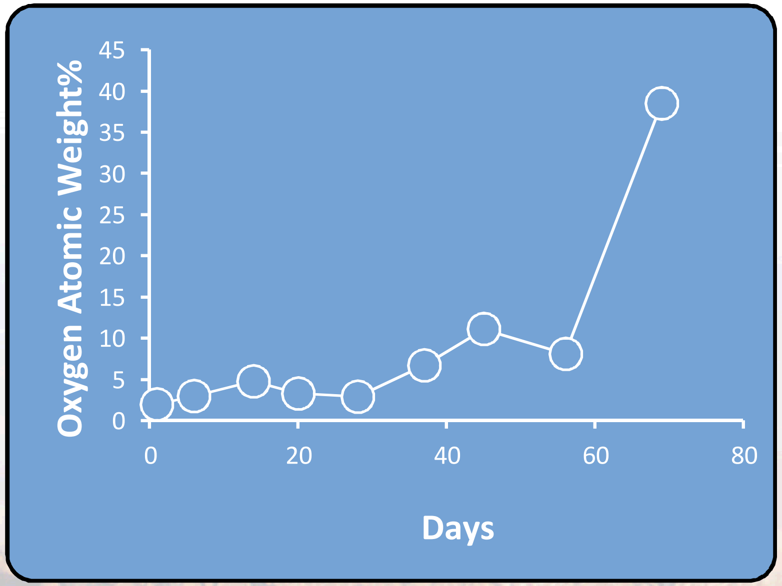
SJC3 Calculations on young's modulus/poisson?

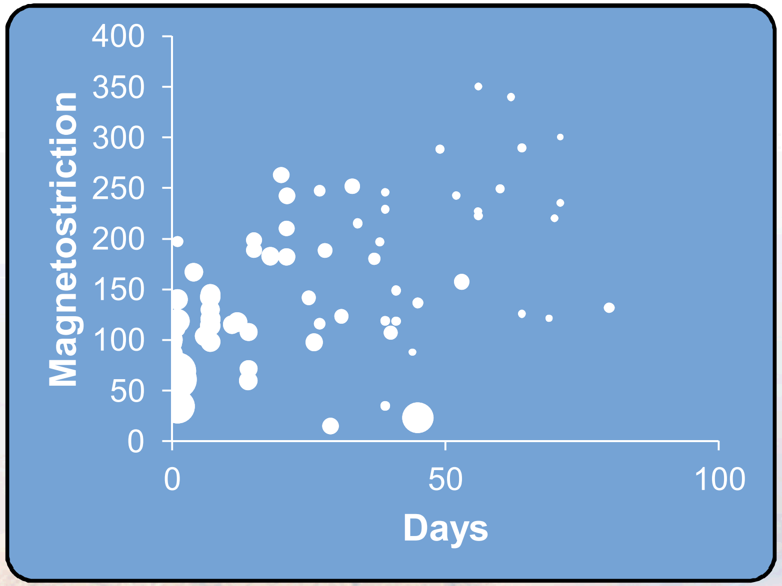
St John, Christopher, 9/21/2016

Conclusions

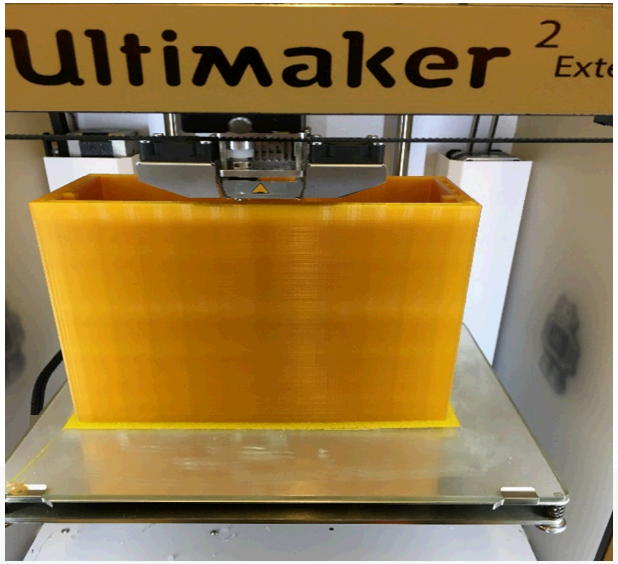
- Wafer-scale chemistry has an average lifespan of approximately one month, compared to beaker scale which lasts approximately two months
- At this point, the current efficiency drops below 30% and oxygen content of the film increases above 10%
- This has an unknown effect on magnetostriction and piezoelectric coefficient







Future Work



Smaller chemistry

Blanket head-space with nitrogen during plating

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What questions do you have?

Acknowledgments: C.L. Arrington[1], J. Pillars[1], E. Langlois[1], P. Finnegan[1], A.E. Hollowell[1], A. Thorpe[1]
(1)Sandia National Laboratories, Albuquerque, NM 87123, USA

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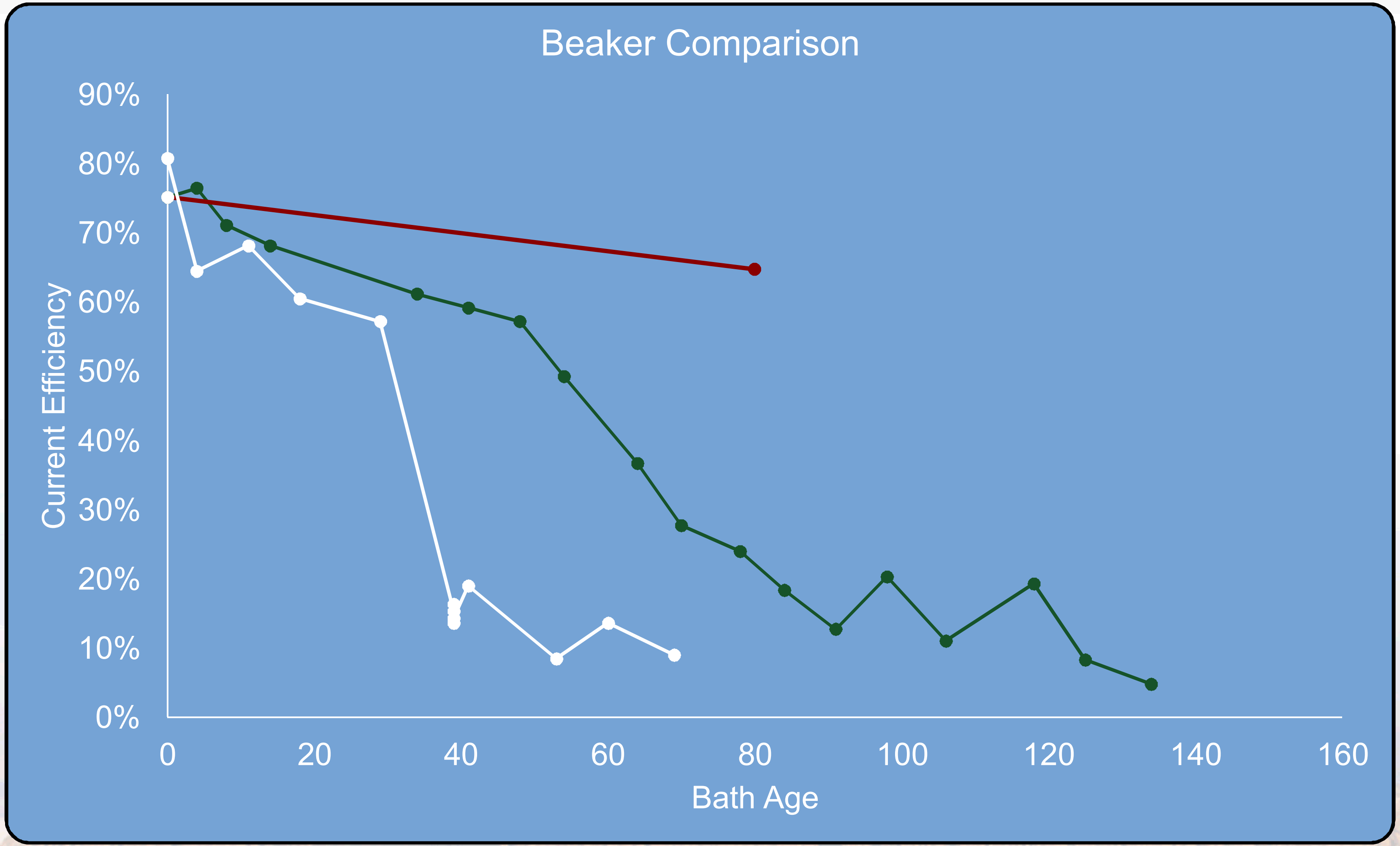
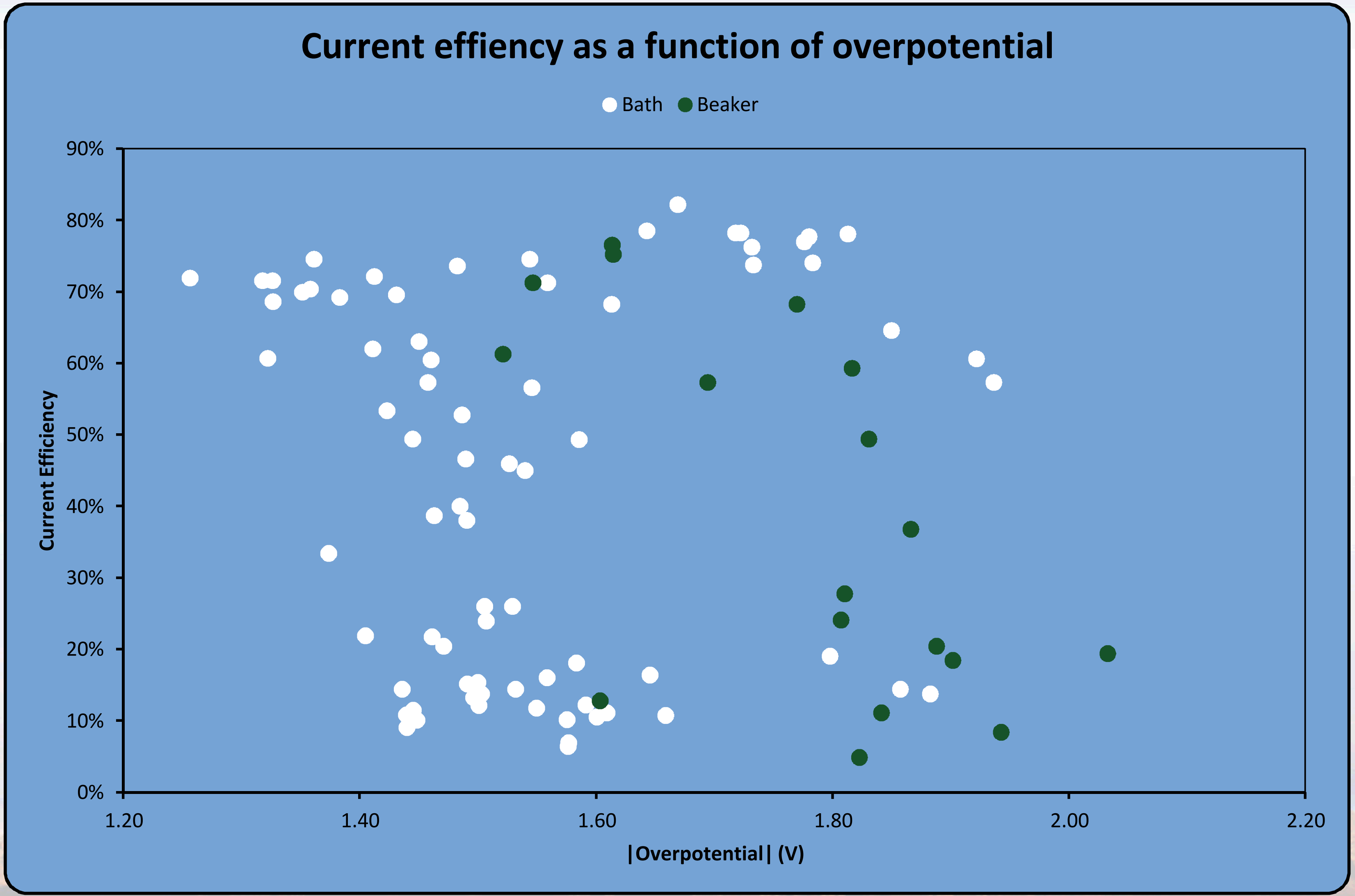
Beaker with no thermal cycling



Comparison between bath and beaker overpotential



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