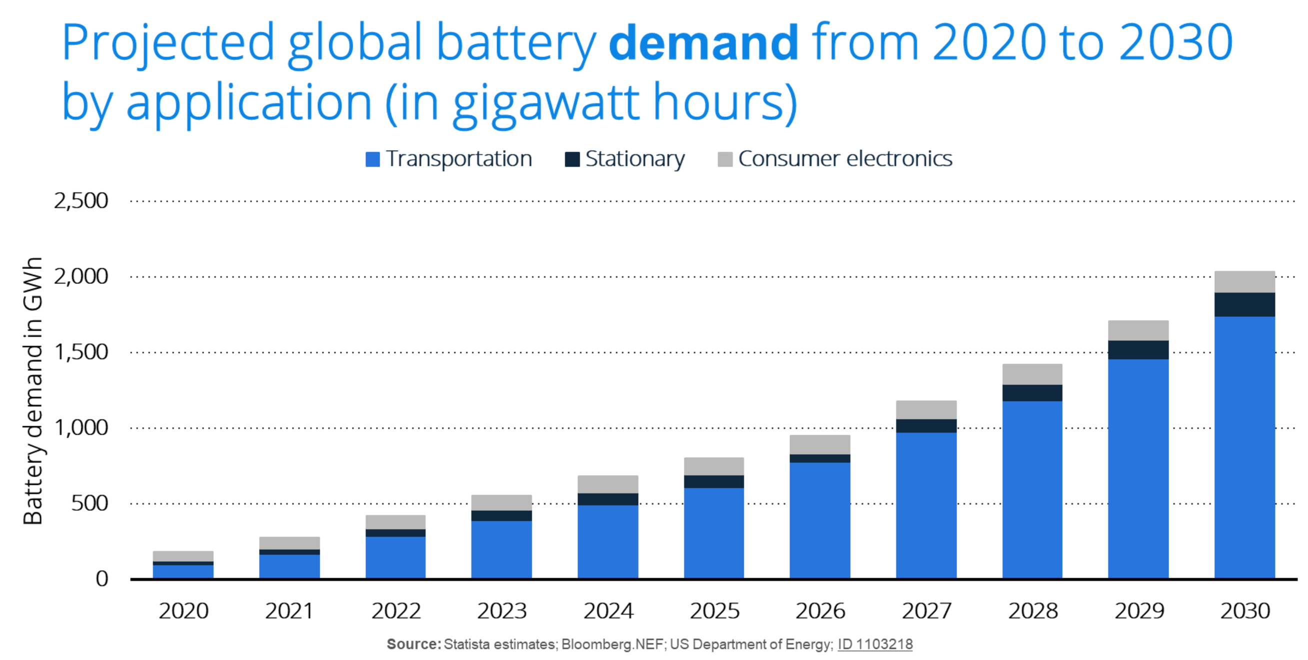
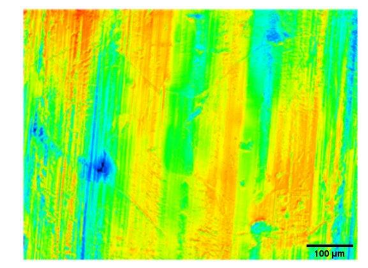
Methodology for Mechanical Pillar Array Indentation of Alkali Metals

Thomas S. Marchese[1], Stephen Harris[2], Katherine L. Jungjohann[3], Brad Boyce[4], Katharine L. Harrison[4], Matthew T. McDowell[1,5]
1. School of Materials Science and Engineering, Georgia Institute of Technology 2. Lawrence Berkeley National Laboratory 3. National Renewable Energy Laboratory 4. Sandia National Laboratories 5. Woodruff School of Mechanical Engineering, Georgia Institute of Technology

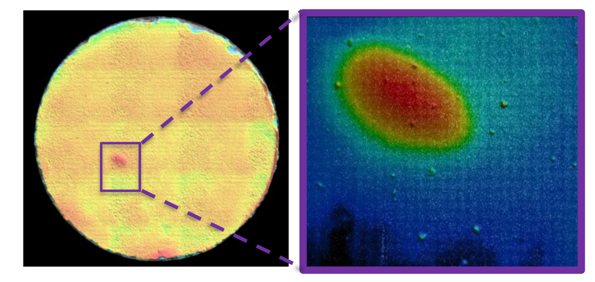
The Need for Mechanical Characterization of Alkali Metals



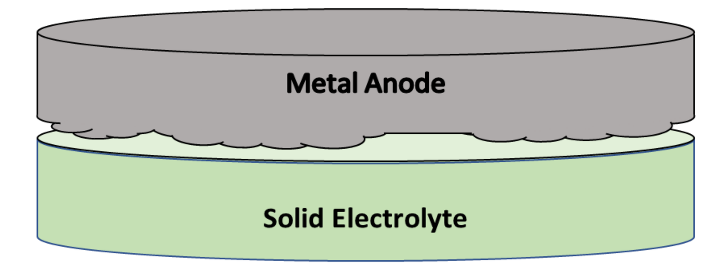
In solid-state batteries, both the anode and solid-electrolyte must create and maintain a conformal solid-solid interface in order to strip and deposit ions during cycling. The surfaces of both components have a roughness which requires consideration.



Untreated lithium foil interferometer measurement
Ra = 0.8 µm Rz = 7.3 µm

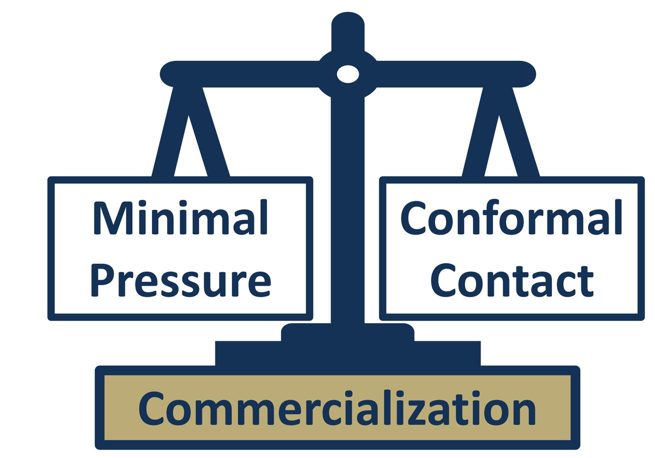


10 mm diameter LAGP solid-state electrolyte ceramic pellet
Ra= 5.8 µm Rz= 131.5 µm



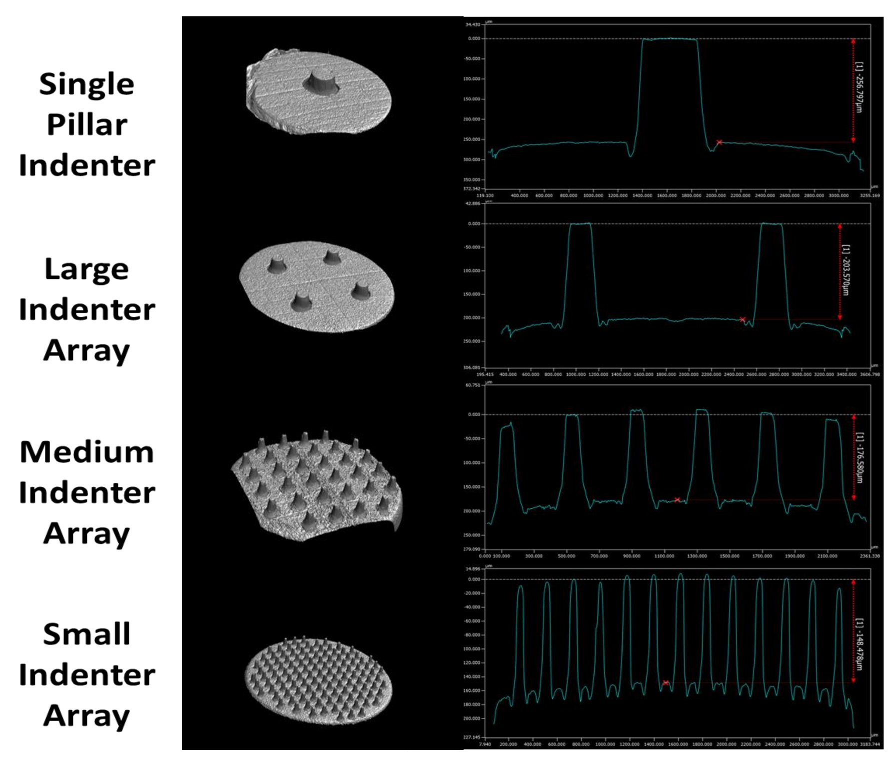
The solid with the lower moduli (the alkali metal anode) will conform under sufficient compressive stress to the surface of the stiffer solid-electrolyte

Mechanical property size dependence of lithium metal from nanoscale to bulk. $\sigma_{y,Li}$ ranges from 1 MPa to 100 MPa

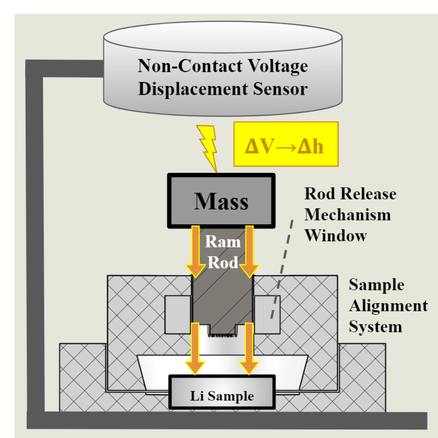


Laser Fabricated Indenter Arrays

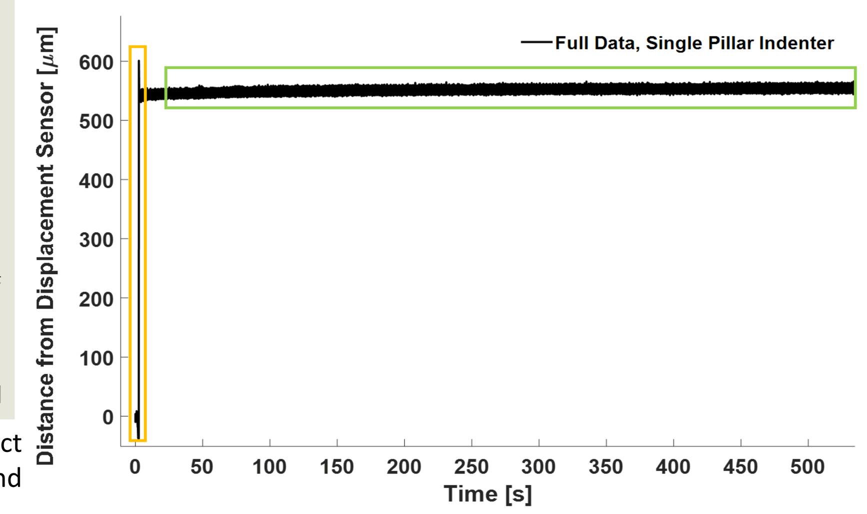
A femtosecond laser etched four stainless steel 316 indenter geometries to investigate any size effect of the material response

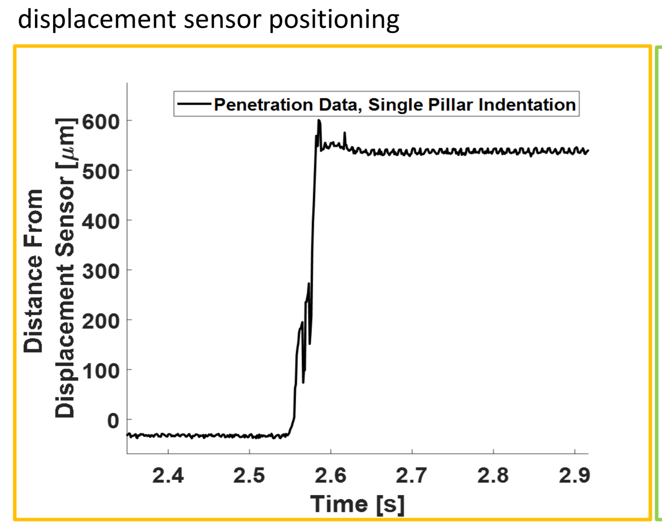


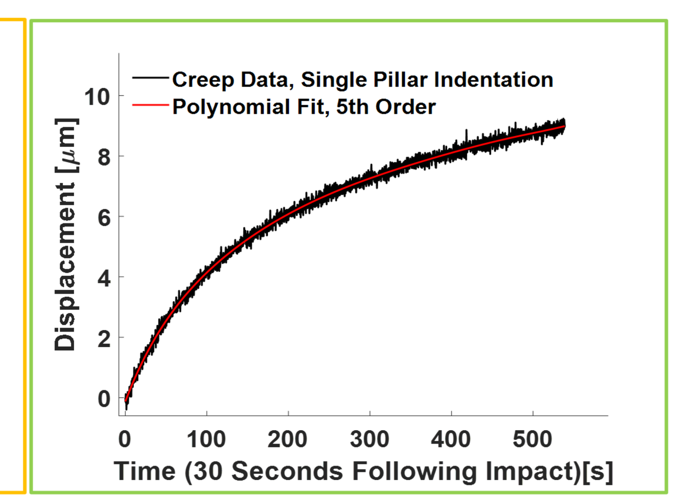
Impact-Indentation Testing of Lithium



Schematic of indenter-array contact introduction holder mechanism and displacement sensor positioning

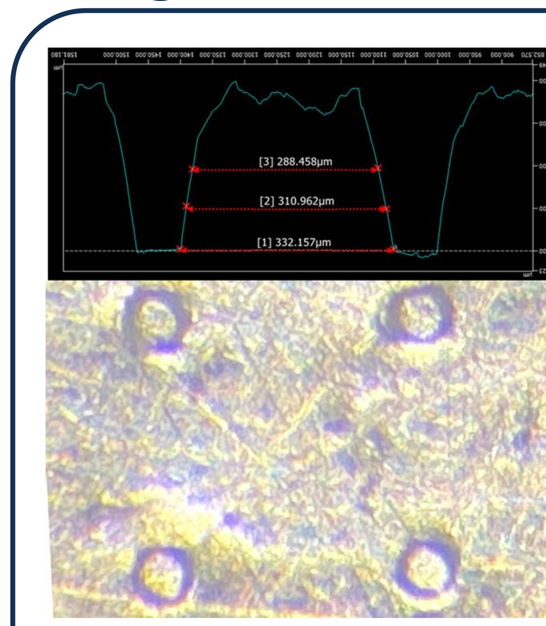




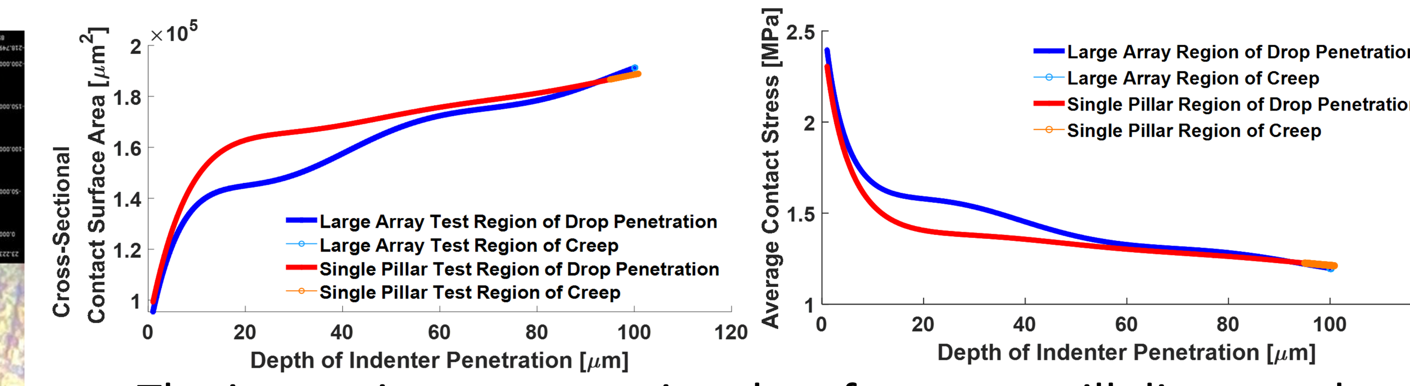


The displacement data recorded by the voltage displacement sensor was segmented into two portions for analysis, Penetration and Creep.

Single Pillar Indenter is comparable to the Large indenter Array

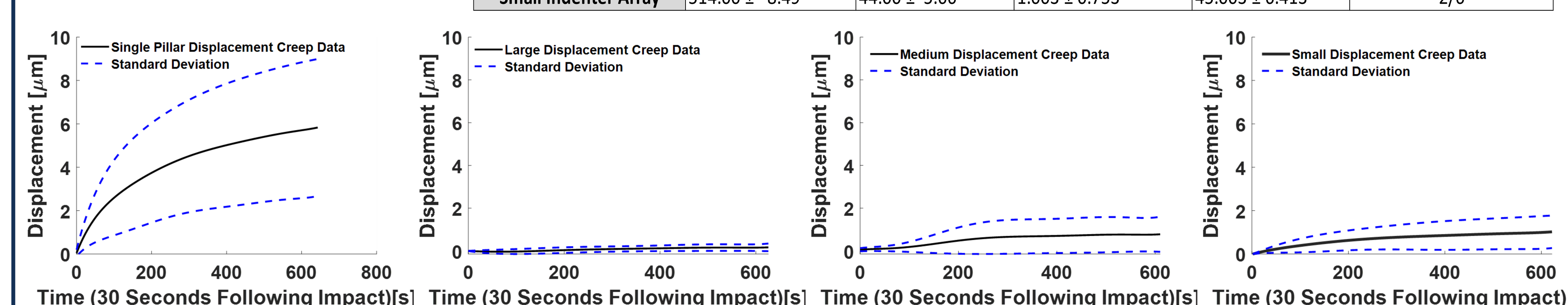


Indentations of lithium foil medium indenter array (bottom) correlated to profilometry scans of the indenters (top) to verify the voltage-displacement data



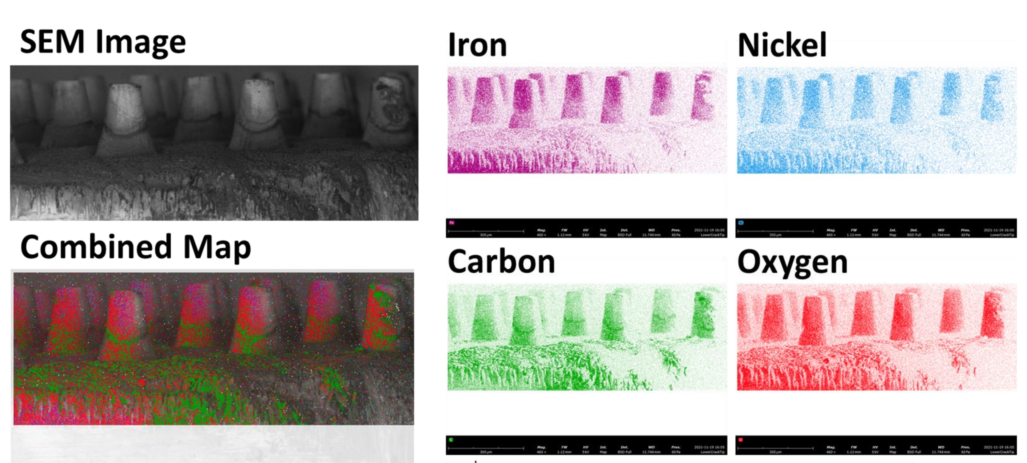
The increasing cross-sectional surface area will disperse the concentration of the load as indenters penetrate the foil further, lowering the average contact stress.

Indenter Geometry	Distance Indenter(s) Dropped into Contact [µm]	Average Penetration [µm]	Average Creep Displacement [µm]	Average Total Displacement [µm]	Number of Tests Accepted in Average/ Total Number of Tests
Single Pillar Indenter	540.96 ± 191.24	94.02 ± 33.82	5.381 ± 2.846	99.401 ± 36.666	5/8
Large Indenter Array	551.68 ± 68.07	99.00 ± 17.81	0.165 ± 0.172	99.165 ± 17.982	3/5
Medium Indenter Array	323.00 ± 75.64	29.17 ± 8.26	0.764 ± 0.803	29.934 ± 9.063	3/6
Small Indenter Array	514.00 ± 8.49	44.00 ± 5.66	1.003 ± 0.753	45.003 ± 6.413	2/6

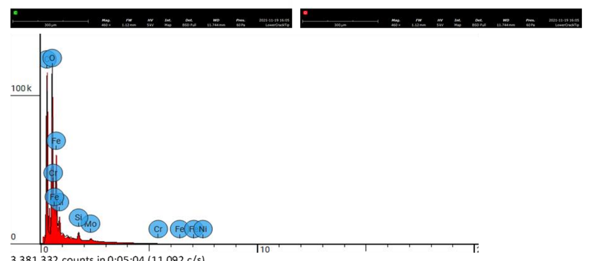


σ =1.22 MPa σ = 1.19 MPa σ = 1.64 MPa σ = 0.654 MPa

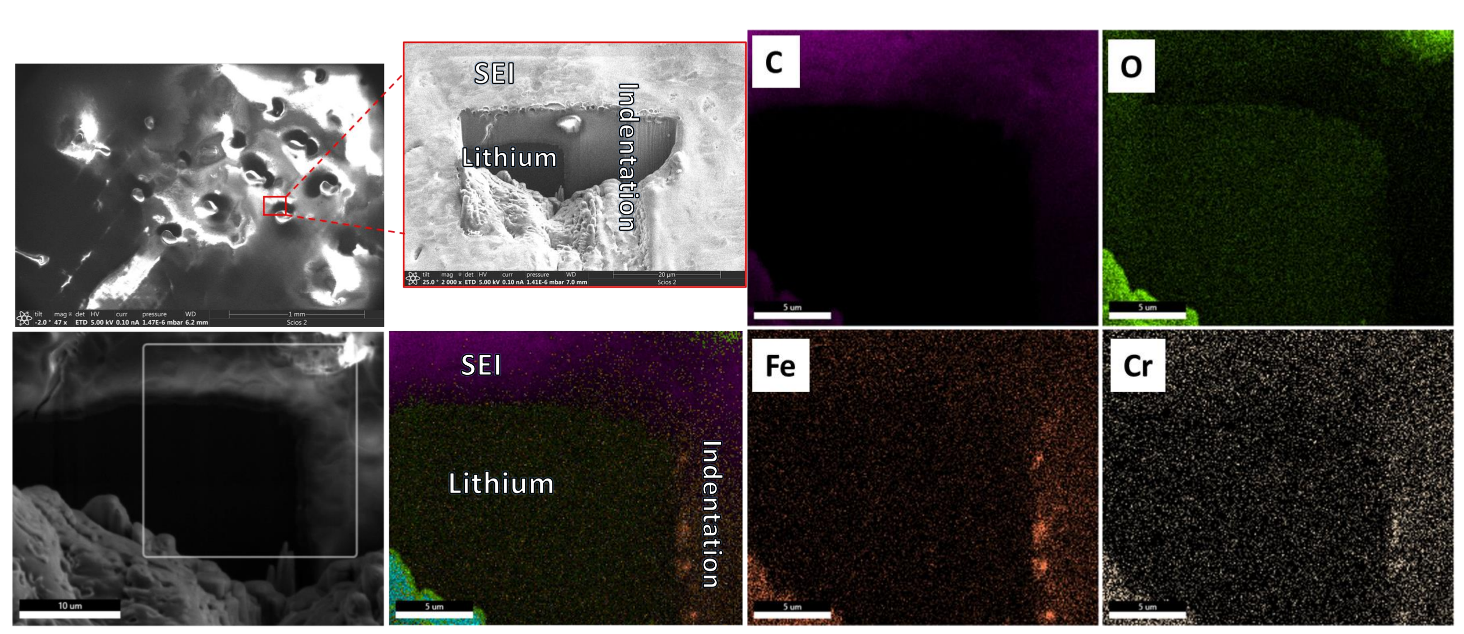
Reactivity of Indenters with Lithium



	Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
	6	C	Carbon	40.701	18.100
	8	O	Oxygen	27.513	16.300
	14	Si	Silicon	1.827	1.900
	24	Cr	Chromium	0.000	0.000
	26	Fe	Iron	25.580	52.900
	28	Ni	Nickel	3.451	7.500
	42	Mo	Molybdenum	0.929	3.300



Post lithium contact, Stainless Steel 316 indenter array low Cr signal SEM/EDS.



Cryo-FIB SEM/EDS 15kV of lithium foil indentations revealed signs of potential reaction with the stainless-steel indenter

Conclusions from Initial Testing

Single pillar indenter was comparable to the large pillar array.

- Showed that Multiple contact points of the same cumulative contact surface area had less penetration depth into lithium foil during long term deformation than a single contact point.
- Lithium preparation is of the utmost importance in creating consistent battery interfaces.
- Femtosecond laser etching is a viable route to creating periodically arranged indenters.
- Mechanical pressure applied to solid-state batteries in between cycling could offer a route for healing micron scale interfacial voids caused by rapid stripping.

Future Recommendations for Array Indentation Testing

- Higher loads are needed to increase reproducibility of the long-term behavior for comparison of the mechanical property size effect of alkali metal thin films.
- Creating or matching a closed form mechanical solution for the high strainrate conditions realized in this experiment.

Acknowledgments

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