Supplementary Information for Automated electron microscopy sample preparation system

David Milsted^{a,*}, Tara P Mishra^{a,*}, Lauren N. Walters^{b,c}, Yuxing Fei^{a,b}, Bernadus Rendy^{a,b}, Pragnay Nevatia^d, Haegyeom Kim^a, Gerbrand Ceder^{a,b}

^a Materials Sciences Division Lawrence Berkeley National Laboratory Berkeley CA USA
 ^b Department of Materials Science and Engineering University of California Berkeley Berkeley CA USA
 ^c Bakar Institute of Digital Materials for the Planet University of California Berkeley Berkeley CA USA
 ^d Department of Chemical Engineering UC Berkeley Berkeley CA USA
 * These authors contributed equally.



Supplementary Figure S1: Control panel chassis for the EMSBot.



Supplementary Figure S2: Control panel diagram for the EMSBot showing electrical circuit diagrams.



Supplementary Figure S3: EMSBot webpage based GUI interface.

Machine Test	Terminal Output > Socket.10 connected > HTTP request: server_test_connectivity called > Resonance: Pion called to 142.251.214.142. It
Communication tests 3D-Printer: test connectivity Send "MACSTAT" Send "ping"	<pre>was successful PING 142.251.214.142 (142.251.214.142): 56 data bytes 64 bytes from 142.251.214.142): icomp_seq=0 tur56 time=44.626 ms 64 bytes from 142.251.214.142: icomp_seq=1 ttl=56 time=42.634 ms — 142.251.214.142 ping statistics — 2 packets transmitted, 2 packets received, 0.0% packet loss round-trip min/avg/max/stddev = 42.034/43.330/44.626/1.296 ms</pre>
Manual use and maintenance Extend bed Uncover SEM stage Cover SEM grid cover Close TEM grid cover Control panel: send "standby" Control panel: send "shutdown"	
Manual Control	
▲ ▲ IMPORTANT ▲ ▲ ▲ Misusing these functions might break machine parts! Please read the instructions for each manual control module before using them!	
3DP - Direct Control	

Supplementary Figure S4: Interface and controls during machine test. Terminal interface with the control messages and feedback from EMSBot.

Preparation parameters Tray Positions Exposure Voltage: 1000 1000 Iminum: 1000, Maximum: 2000 Container height: (2) 39 Values in mm. Vertical shift: (1) 18 (2) Values in mm. Lowset: 25.0, Highest: 25.0 Exposure Voltage: 10 Values in mm. Lowset: 25.0, Highest: 25.0	Tray Positions
5000 Minimum: 0, Maximum: 9999 5000 Origin and Destination: Minimum: 0, Maximum: 9999 Origin: A1 Tot Tot Prepare Sample Destination: Tet	

Supplementary Figure S5: Sample preparation screen for EMSBot for (left) SEM and (right) TEM.



Supplementary Figure S6: Control flow diagram for the electron microscopy sample preparation using EMSBot.



Supplementary Figure S7: The distribution of particle size when the SEM grid is at distance of -16 mm and -18 mm. The total number of particles analyzed for 580 and 311 particles were analyzed for the -16 mm and -18 mm from the top of the crucible respectively. (Inset) Magnified histogram of the distribution of the particles > 25 μ m.



Supplementary Figure S8: Statistical analysis of particle sizes for ball-milled precursor samples of Na₄P₂O₇ and SnO₂, prepared using both the EMSBot and manual methods.



Supplementary Figure S9: Comparison of SEM images of samples prepared using EMSBot (A) and manual methods (B), based on wet spin mixing of $Na_4P_2O_7$ and SnO_2 precursors. The example images are shown for medium sized particles (8-50 µm diameter). Images on the left are the SEM with overlaid red circles demonstrating where the automated SEM program identified a single particle, as well as the particle's identification number. Images are chosen to be as representative as possible. (C) Statistical analysis of particle sizes, for SEM samples prepared using the EMSBot and manual methods.



Supplementary Figure S10: Comparison of SEM sample prepared (A) manually vs (B) using EMSBot.





Supplementary Figure S11: SEM micrograph for the $Na_6W_{12}O_{39}$ sample prepared using EMSBot and the respective Na, O, and W EDS compositional maps along with the total EDS spectrum.





Supplementary Figure S12: SEM micrograph for the $Ni(OH)_2$ sample prepared using EMSBot and the respective Ni and O EDS compositional maps along with the total EDS spectrum.





Energy (keV)

Supplementary Figure S13: SEM micrograph for the $NH_4H_2PO_4$ sample prepared using EMSBot and the respective N, P, and O EDS compositional maps along with the total EDS spectrum.

Ti



Supplementary Figure S14: SEM micrograph for the Ti sample prepared using EMSBot and the respective Ti EDS compositional maps along with the total EDS spectrum.



Supplementary Figure S15: SEM micrograph for the Na_2CO_3 sample prepared using EMSBot and the respective C, Na, and O EDS compositional maps along with the total EDS spectrum.





Supplementary Figure S16: SEM micrograph for the mixture of Na_2CO_3 and Ti samples prepared using EMSBot and the respective C, O, Na, and Ti EDS compositional maps along with the total EDS spectrum.



Supplementary Figure S17: SEM micrograph for the SiO_2 sample prepared using EMSBot and the respective Si and O EDS compositional maps along with the total EDS spectrum.





Supplementary Figure S18: SEM micrograph for the KCI sample prepared using EMSBot and the respective K and CI EDS compositional maps along with the total EDS spectrum.



Supplementary Figure S19: SEM micrograph for the mixture of SiO_2 , Ti, and KCI samples prepared using EMSBot and the respective C, CI, O, K, Si, and Ti EDS compositional maps along with the total EDS spectrum.