

Supporting Information

Revealing Interfacial Failure Mechanism of Silicon Based All Solid State Batteries via Cryogenic Electron Microscopy

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Supplementary Figures

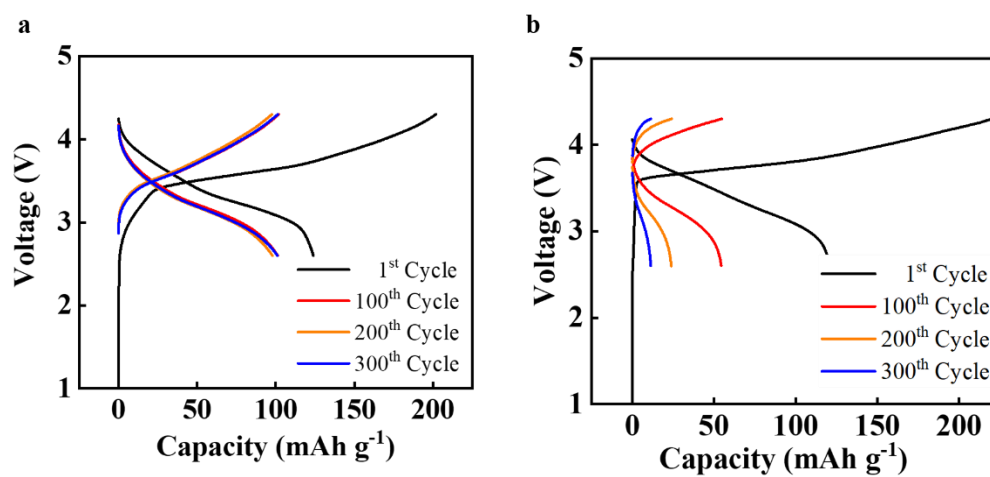


Figure S1. The voltage profiles of the 1st, 100th, 200th and 300th cycles for (a) Si|LSPSC|NMC811 ASSB and (b) Si|LGPS|NMC811 ASSB.

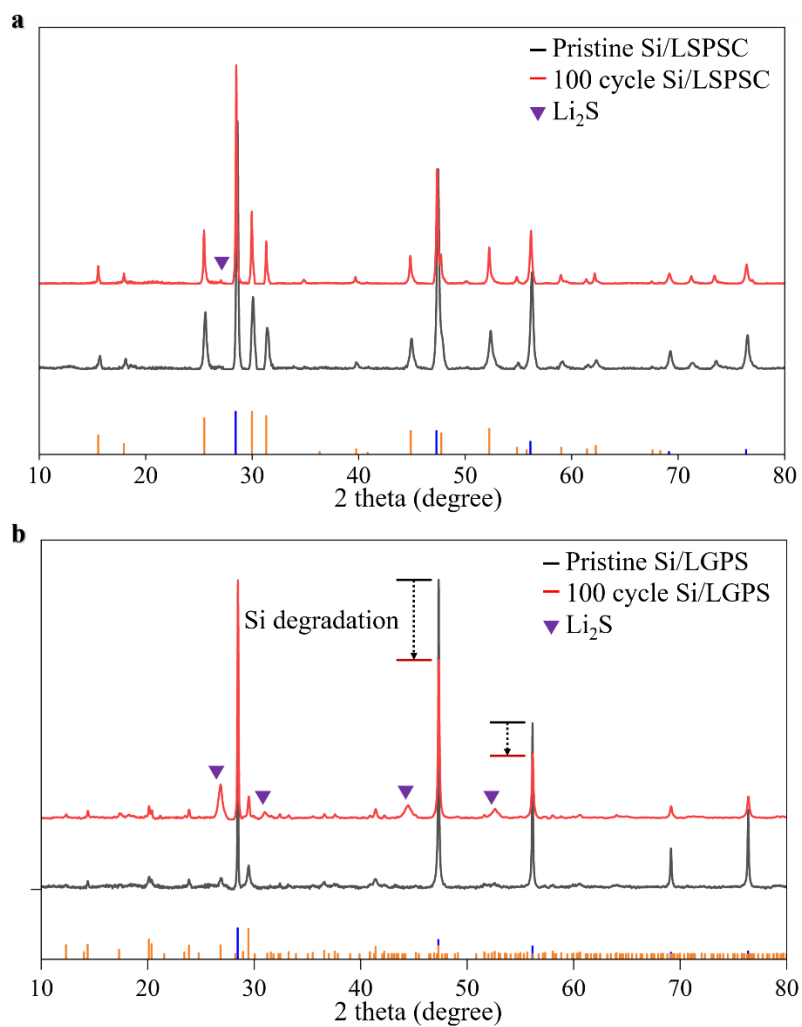


Figure S2. XRD characterization about the Si/electrolyte interface of pristine state and fully charged state at 90 mA/g at 25 °C after 100 cycles cycling. (a) XRD of the Si/LSPSC interface before and after cycling. (b) XRD of the Si/LGPS interface before and after cycling.

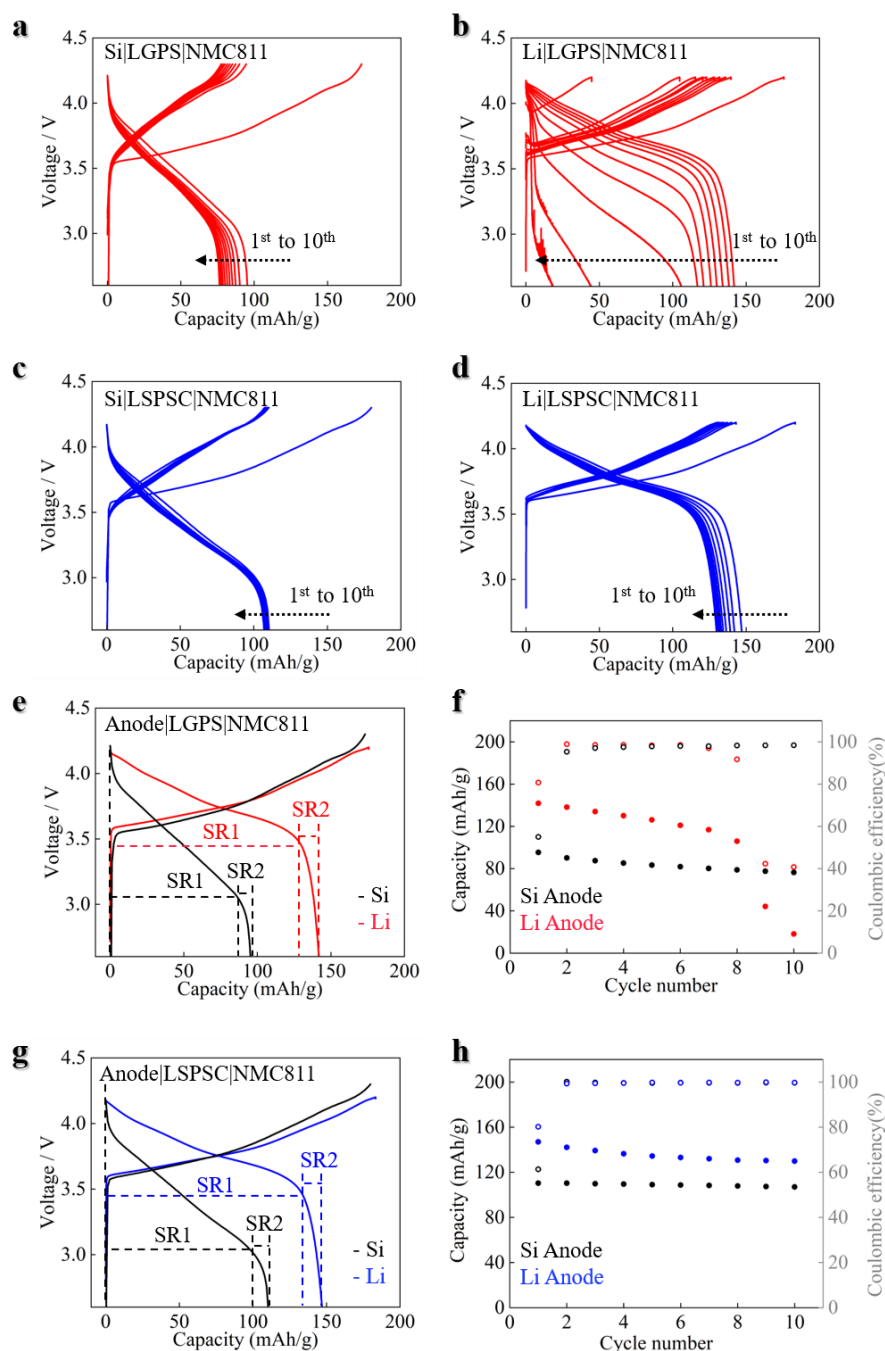


Figure S3. Electrochemical measurements of the Si|sulfide|NMC811 ASSBs at 90 mA/g at 25 °C and the Li|sulfide|NMC811 ASSBs at 20 mA/g at 25 °C: (a) Voltage-capacity profile of the Si|LGPS|NMC811 cell. (b) Voltage-capacity profile of the Li|LGPS|NMC811 cell. (c) The 1st cycle voltage-capacity profile with Si and Li negative electrodes in LGPS based ASSBs. (d) The capacity changes with cycle numbers of the ASSBs based on Si and Li negative electrodes with LGPS SSE. (e) Voltage-capacity profile of the Si|LSPSC|NMC811 cell. (f) Voltage-capacity profile of the Li|LSPSC|NMC811 cell. (g) The 1st cycle voltage-capacity profile with Si negative electrode and Li negative electrodes in LSPSC based ASSBs. (h) The capacity changes with cycle numbers of the ASSBs based on Si and Li negative electrodes in LSPSC SSE.

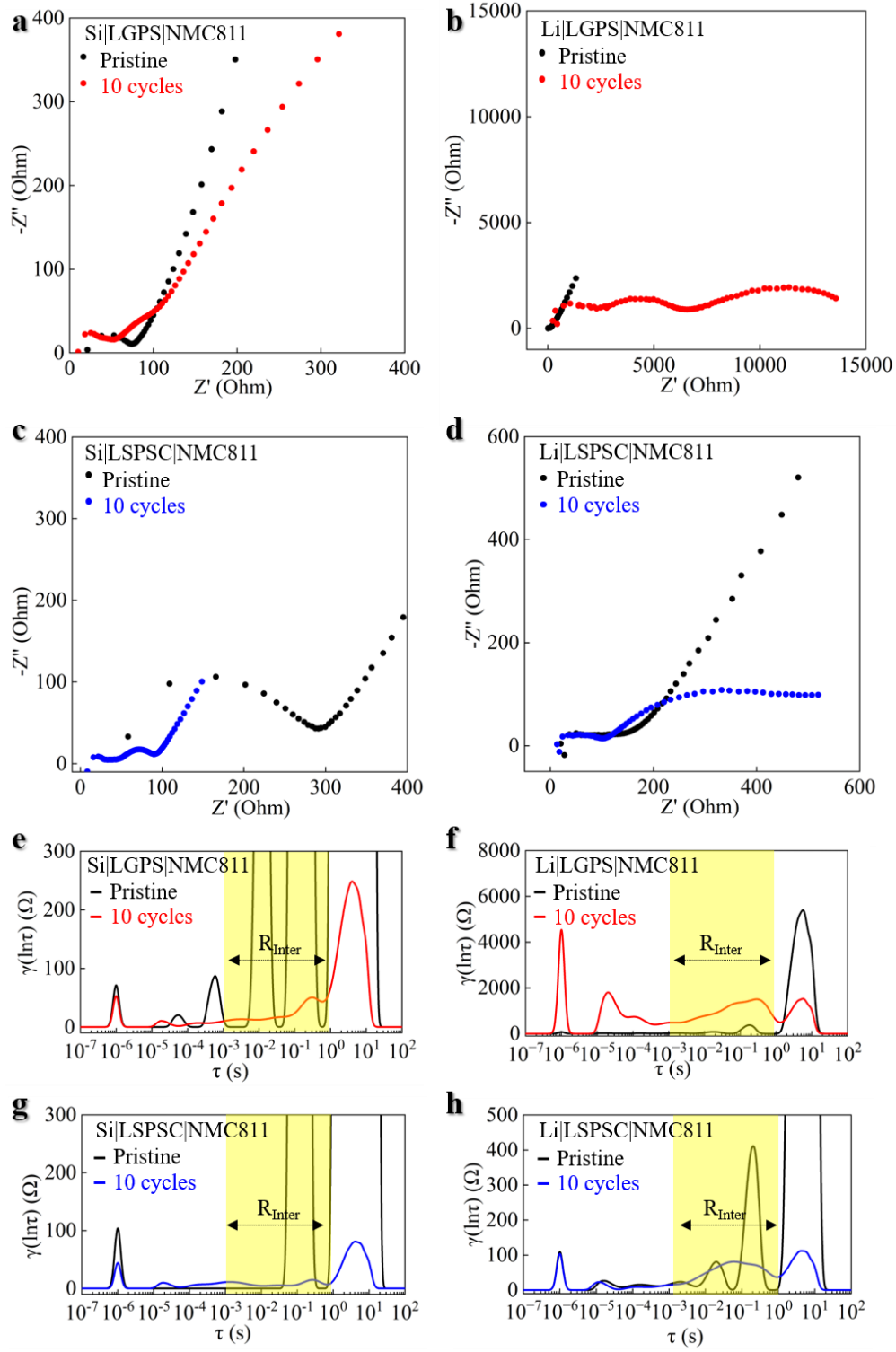


Figure S4. Impedance analysis of the Si|sulfide|NMC811 ASSB at fully charged state after 10 cycles and the Li|sulfide|NMC811 ASSB at fully charged state after 10 cycles. (a) EIS of the Si|LGPS|NMC811 cell. (b) EIS of the Li|LGPS|NMC811 cell. (c) EIS of the Si|LSPSC|NMC811 cell. (d) EIS of the Li|LSPSC|NMC811 cell. (e) DRT analysis of the Si|LGPS|NMC811 cell. (f) DRT analysis of the Li|LGPS|NMC811 cell. (g) DRT analysis of the Si|LSPSC|NMC811 cell. (h) DRT analysis of the Li|LSPSC|NMC811 cell.

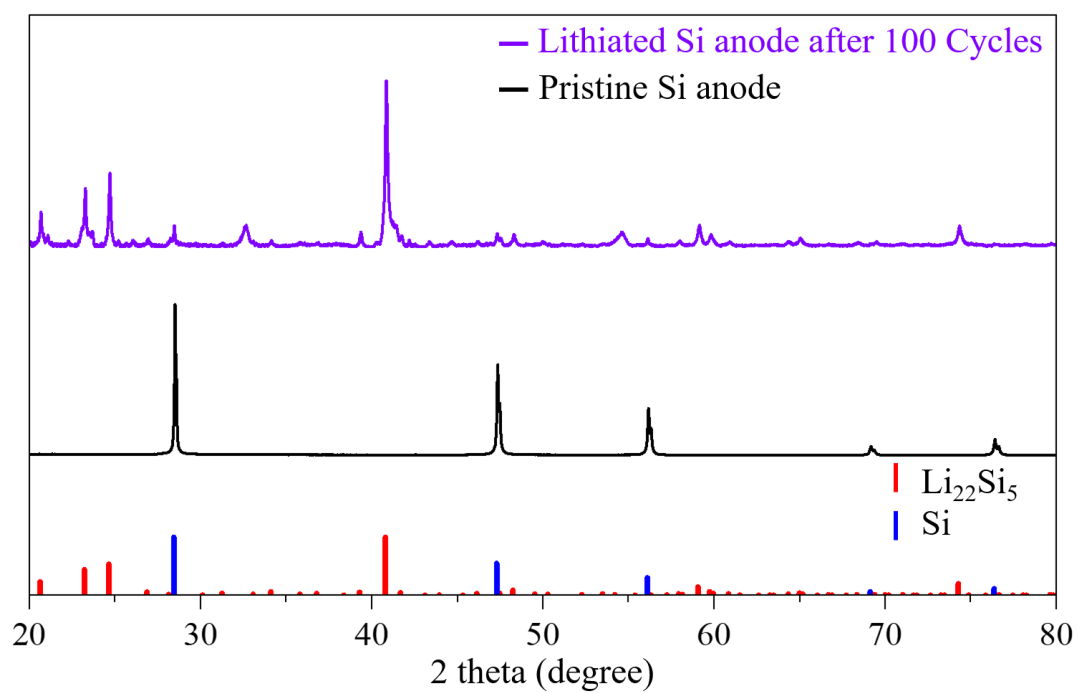


Figure S5. XRD characterization of the pristine Si negative electrode and lithiated Si negative electrode in the Si|LSPSC|NMC811 ASSB at 90 mA/g at 25 °C.

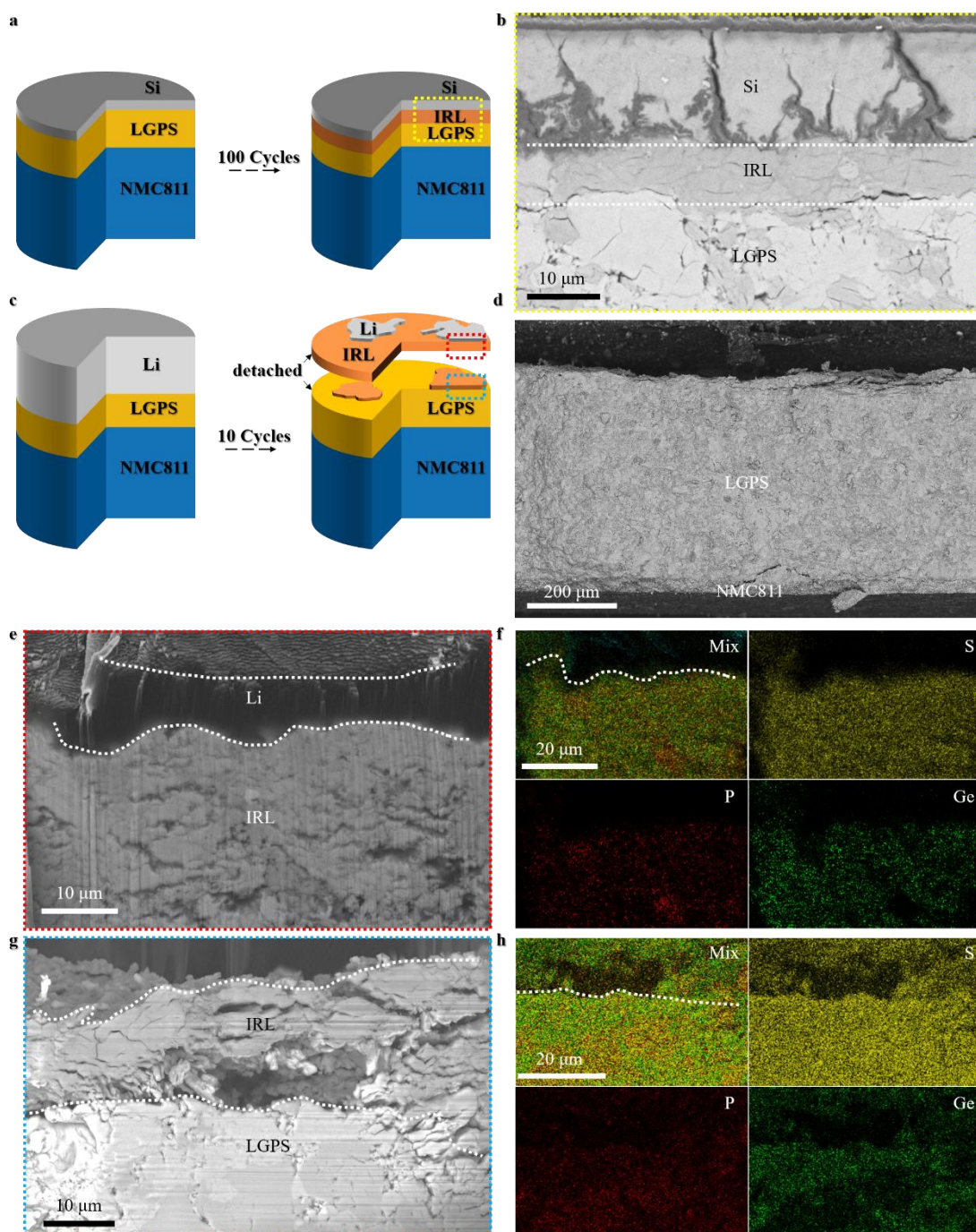


Figure S6. Characterization of the interface of negative electrode/electrolyte in Si electrode at fully charged state after 100 cycles and Li electrode at fully charged state after 10 cycles, respectively. (a) Schematic diagram of the Si|LGPS|NMC811 cell before and after cycling. (b) CBS image of the Si/LGPS interface. (c) Schematic diagram of the Li|LGPS|NMC811 cell before and after cycling. (d) CBS image about the cross section of the entire cell. (e) CBS image of the Li/IRL interface. (f) EDS mapping the Li/IRL interface. (g) CBS image of the IRL/LGPS interface. (h) EDS mapping the IRL/LGPS interface.

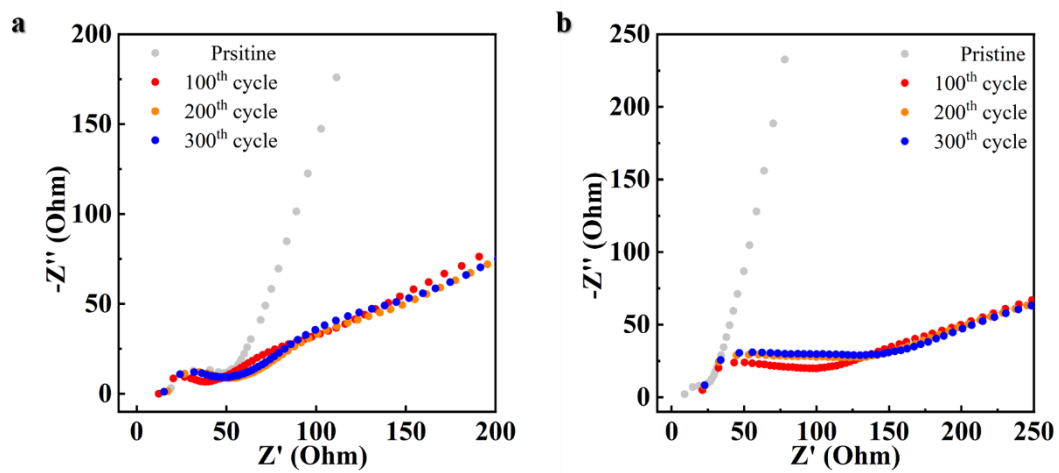


Figure S7. Electrochemical impedance spectra of the Si|Sulfide|NMC811 full cell at different cycles of (a) Si|LSPSC|NMC811 ASSB and (b) Si|LGPS|NMC811 ASSB.

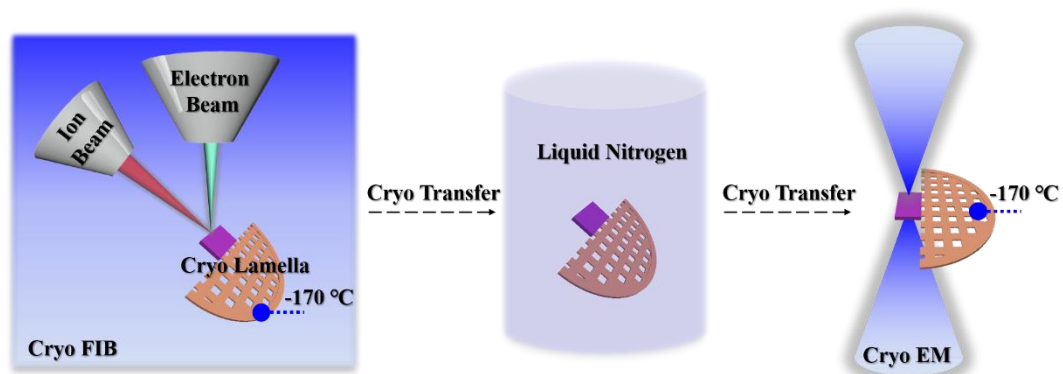


Figure S8. Preparation, transfer, and characterization processes of the sulfide-based ASSB interfaces.

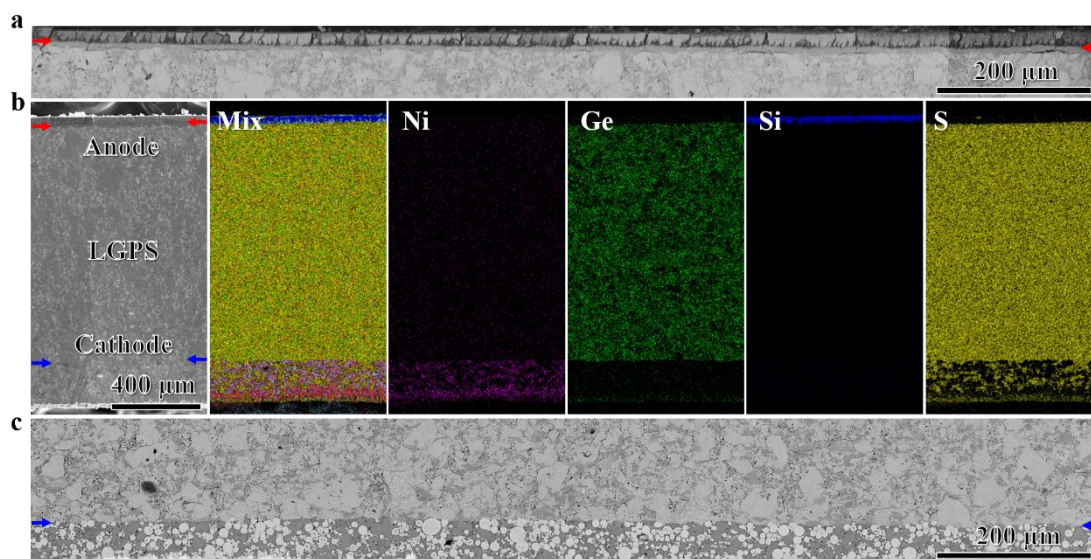


Figure S9. SEM images and energy dispersive spectroscopy (EDS) mapping revealing the battery structure of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Central backscattered electron (CBS) image of the Si/LGPS interface, and (b) Secondary electron (SE) image and the corresponding EDS mapping. (c) CBS image of the LGPS/NMC811 interface.

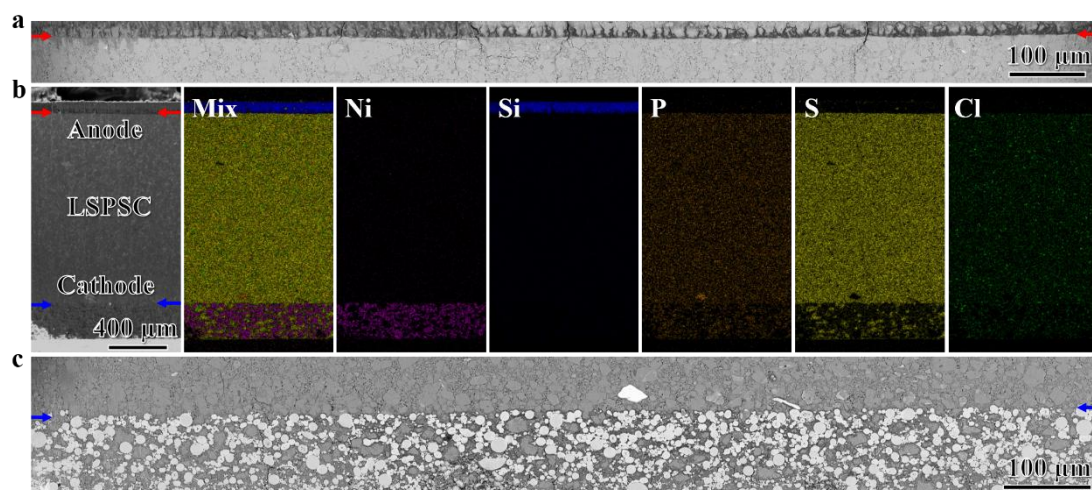


Figure S10. SEM images and EDS mapping revealing the battery structure of the Si|LSPSC|NMC811 ASSB at fully charged state after 100 cycles. (a) CBS image of the Si/LSPSC interface. (b) SE image and the corresponding EDS mapping. (c) CBS image of the LSPSC/NMC811 interface.

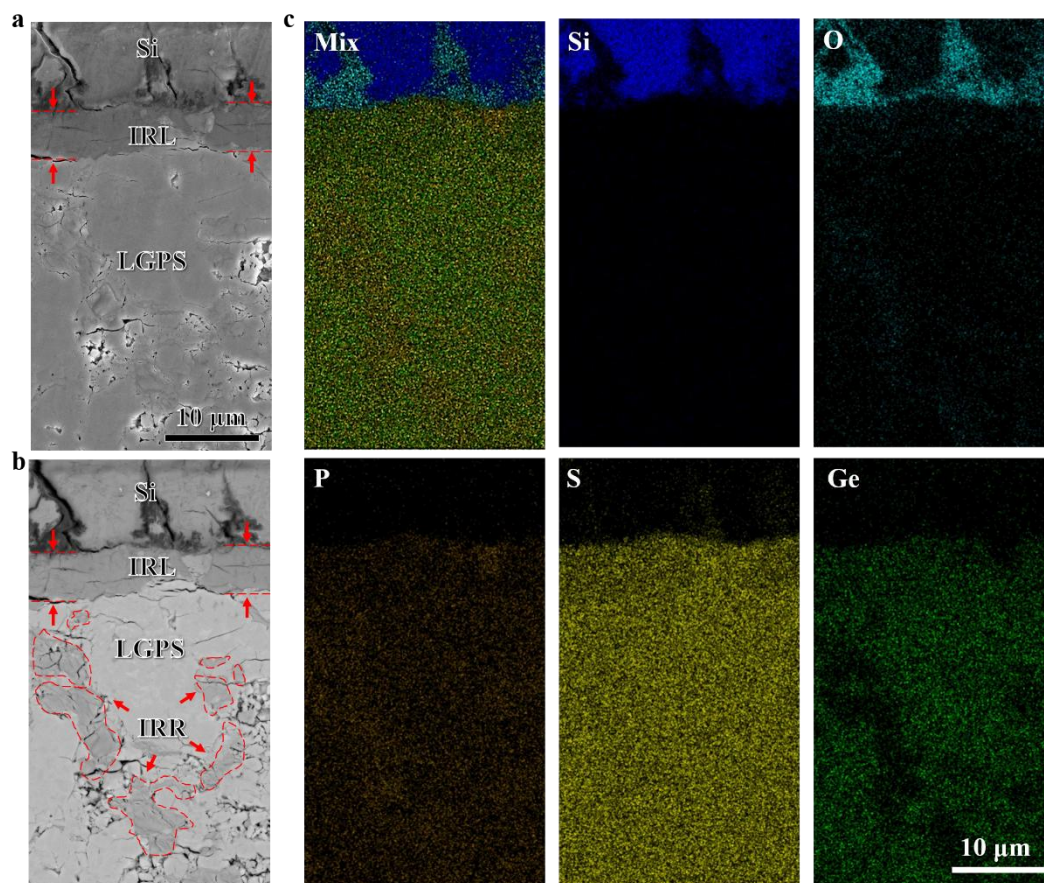


Figure S11. Cryo-SEM images revealing the cross-section of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) SE image of the Si/LGPS interface, and (b) CBS image of Si/LGPS interface and (c) the corresponding elemental mapping.

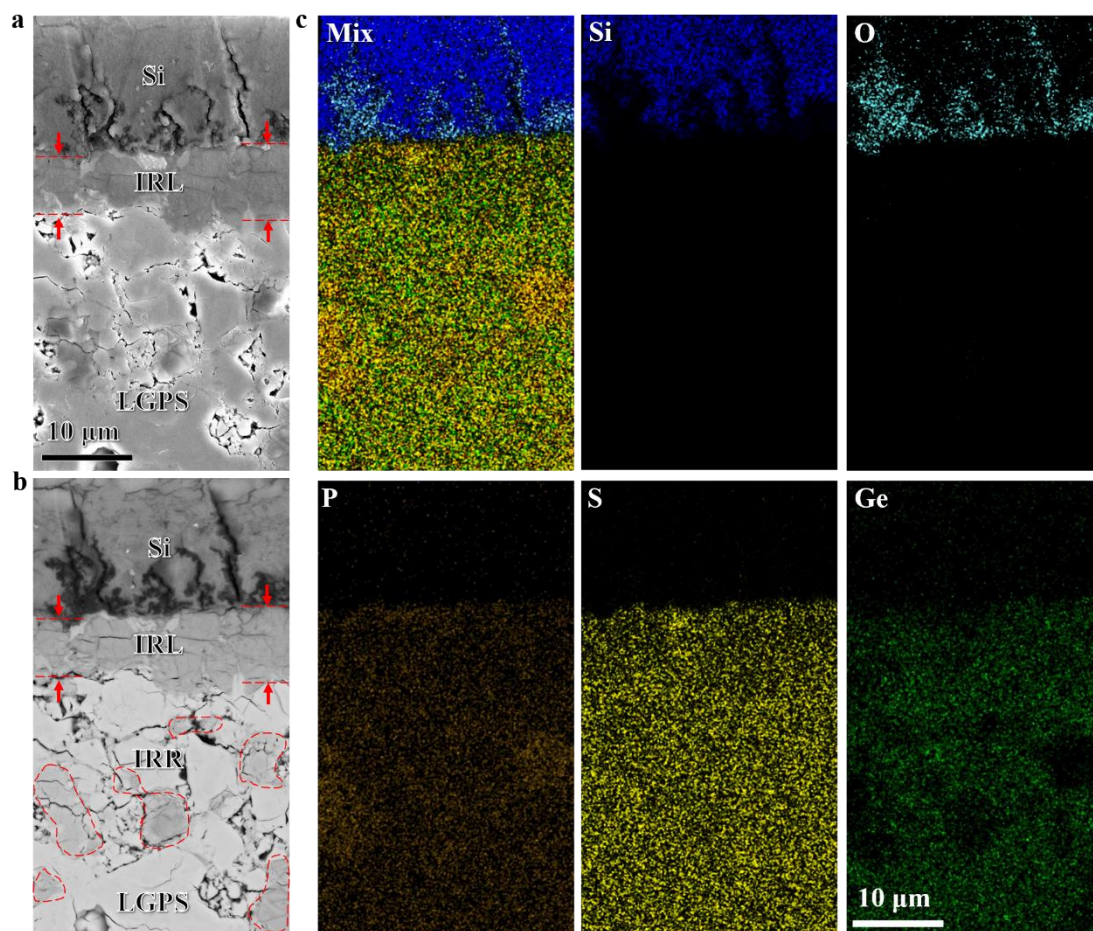


Figure S12. Another set of Cryo-SEM images revealing the cross-section of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) SE image of the Si/LGPS interface, and (b) CBS image of Si/LGPS interface and (c) the corresponding elemental mapping.

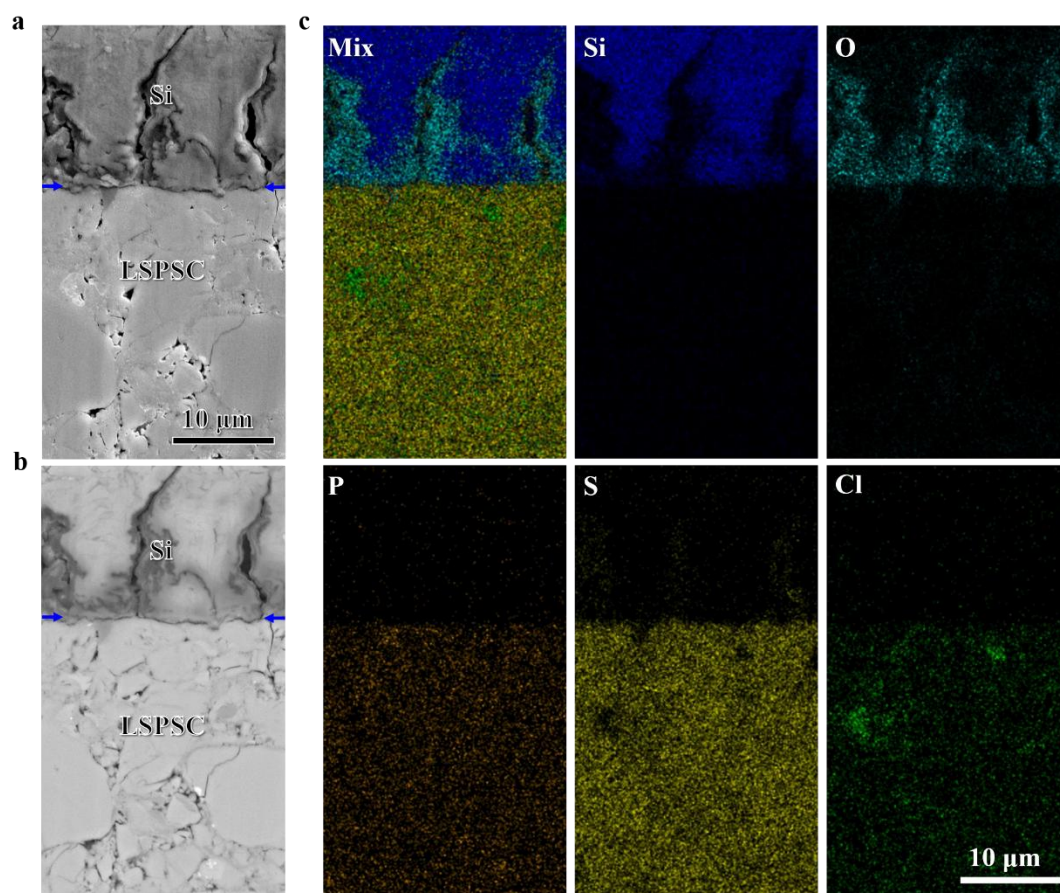


Figure S13. Cryo-SEM images revealing the cross-section of the Si|LSPSC|NMC811 ASSB at fully charged state after 100 cycles. (a) SE image of the Si/LSPSC interface, and (b) CBS image of Si/LSPSC interface and (c) the corresponding elemental mapping.

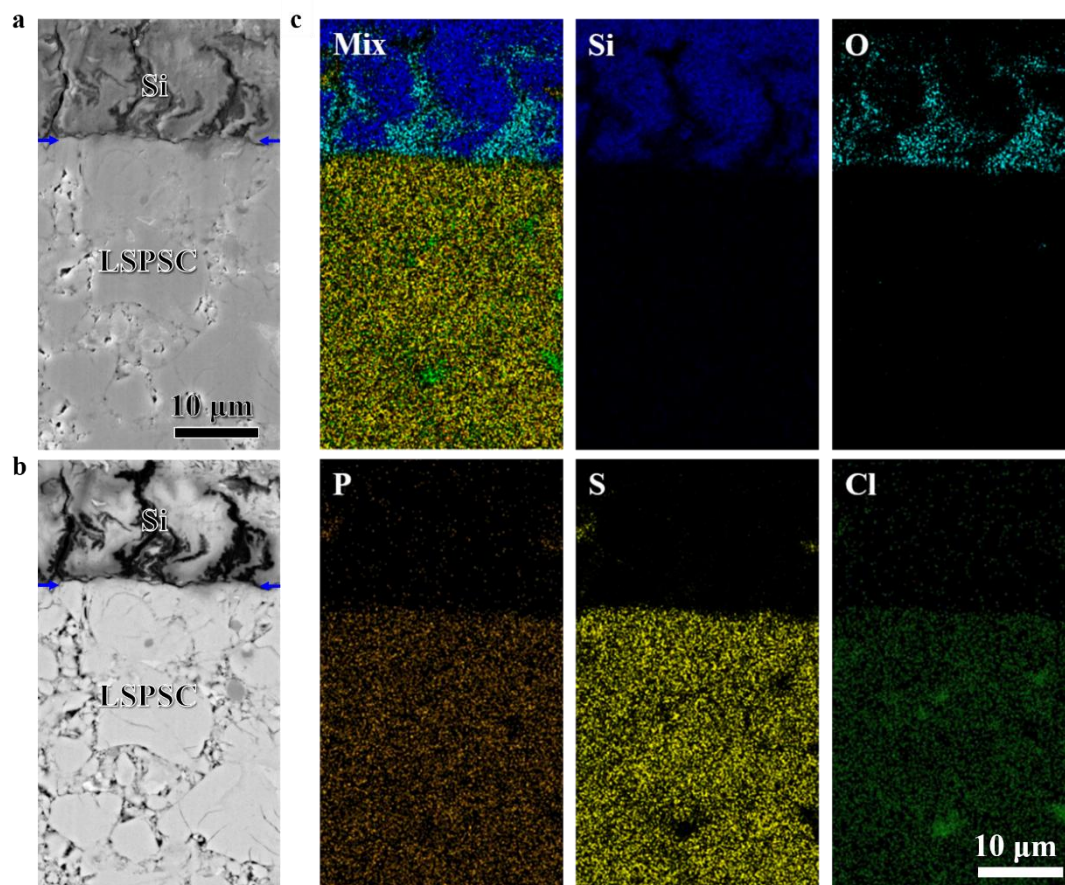


Figure S14. Another set of Cryo-SEM images revealing the cross-section of the Si|LSPSC|NMC811 ASSB at fully charged state after 100 cycles. (a) SE image of the Si/LSPSC interface, and (b) CBS image of Si/LSPSC interface and (c) the corresponding elemental mapping.

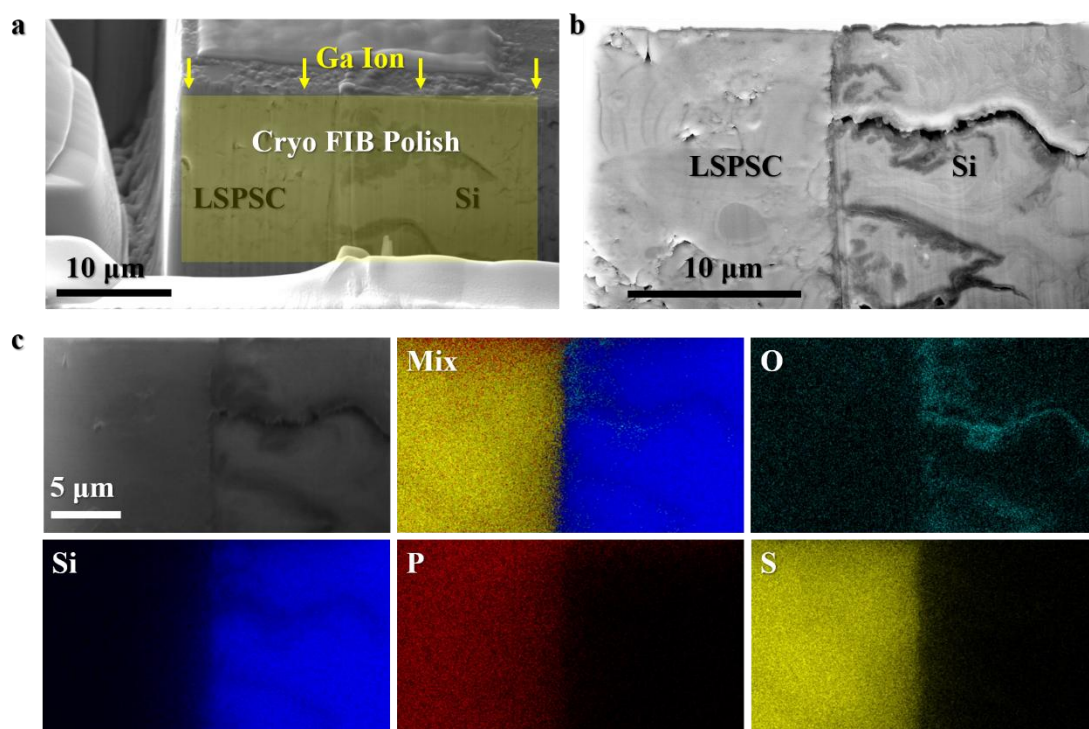


Figure S15. Cryo FIB-SEM characterization of interfaces buried within the battery. (a) Cryo-FIB processed cross-section of the interior of the Si|LSPSC|NMC811 ASSB at fully charged state after 100 cycles. (b) CBS imaging of the Si/LSPSC interface. (c) EDS mapping of the Si/LSPSC interface.

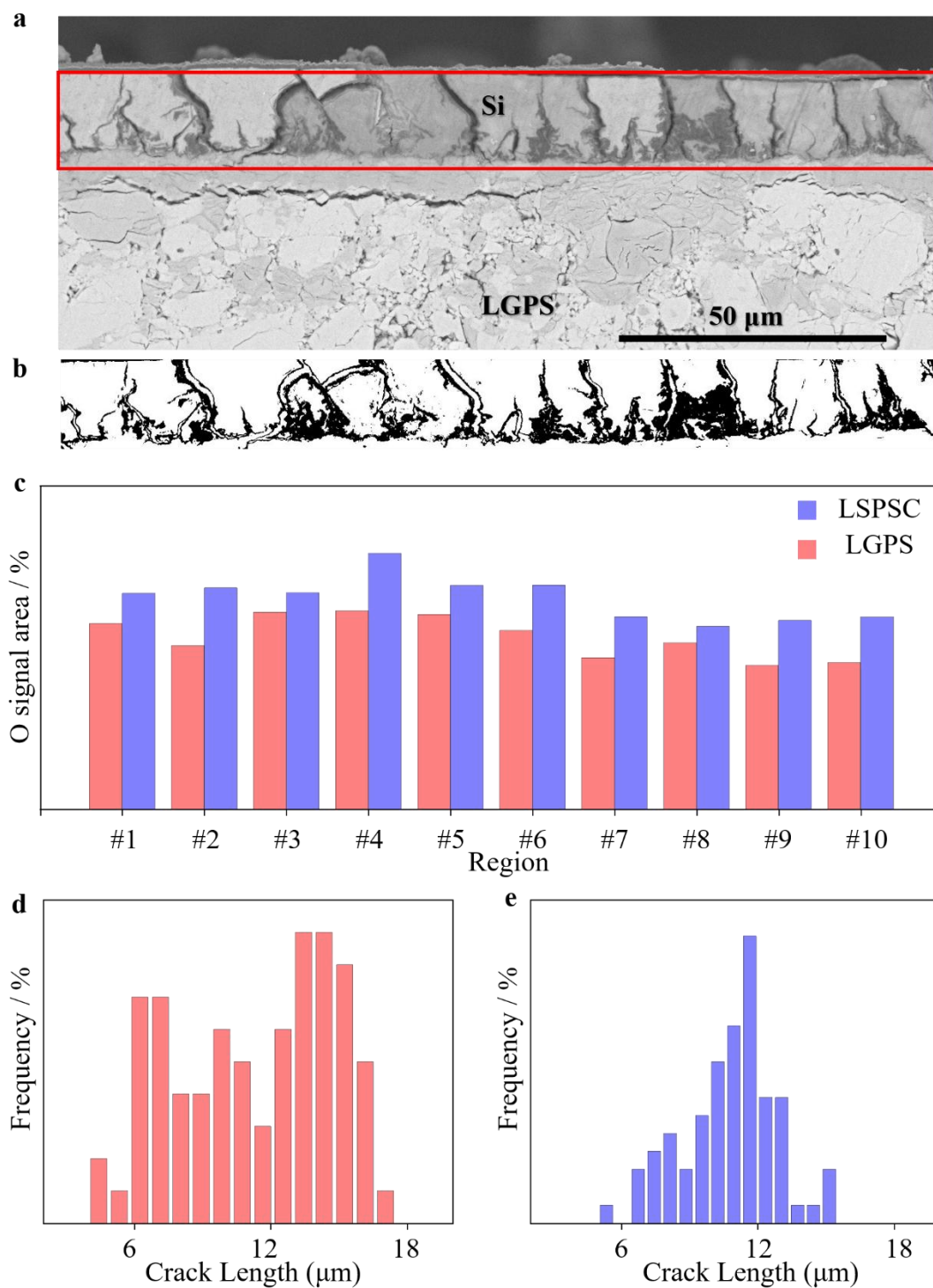


Figure S16. The length of cracks and the distribution state of oxygen elements in the Si negative electrode. (a) SEM image of the Si/sulfide interface. (b) Using ImageJ to analyze the presence of oxygen and cracks in the negative electrode. (c) Area distribution of the oxygen signal in the EDS mapping of the Si/sulfide interface. Frequency distribution of crack depth in (d) Si/LGPS and (e) Si/LSPSC.

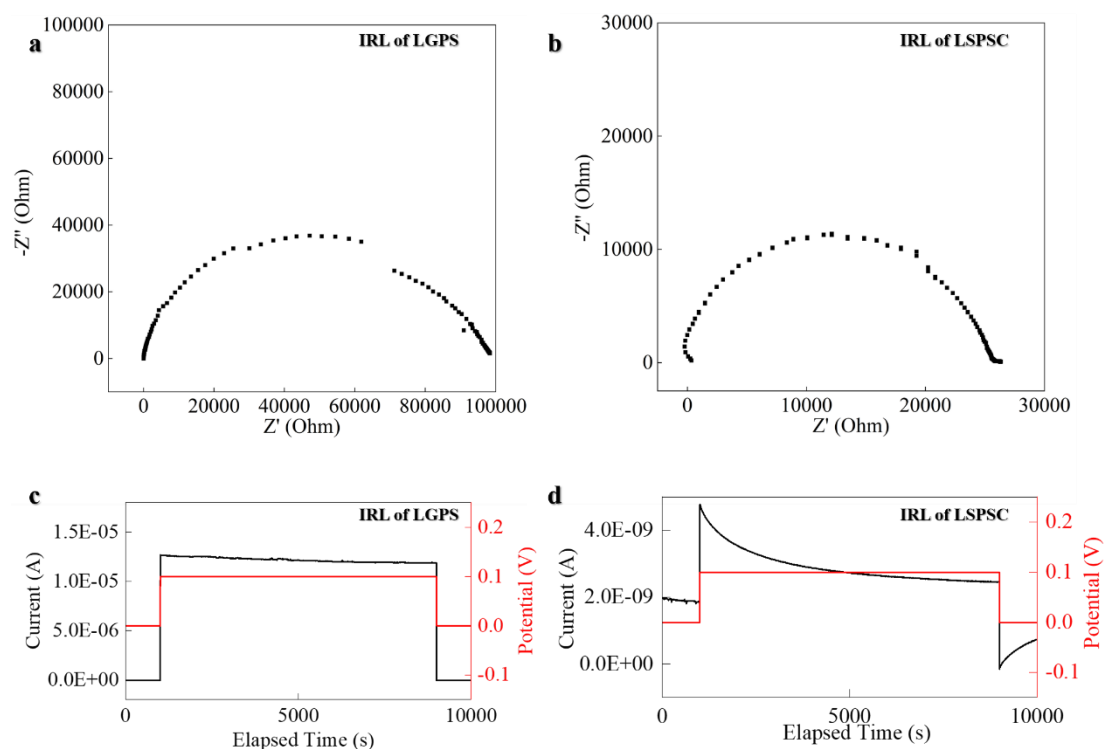


Figure S17. Testing of the ionic conductivity and electronic conductivity of the IRL. (a) The ionic conductivity testing of the IRL about the Si/LGPS interface. (b) The ionic conductivity testing of the IRL about the Si/LSPSC interface. (c) The electronic conductivity testing of the IRL about the Si/LGPS interface. (d) The electronic conductivity testing of the IRL about the Si/LSPSC interface.

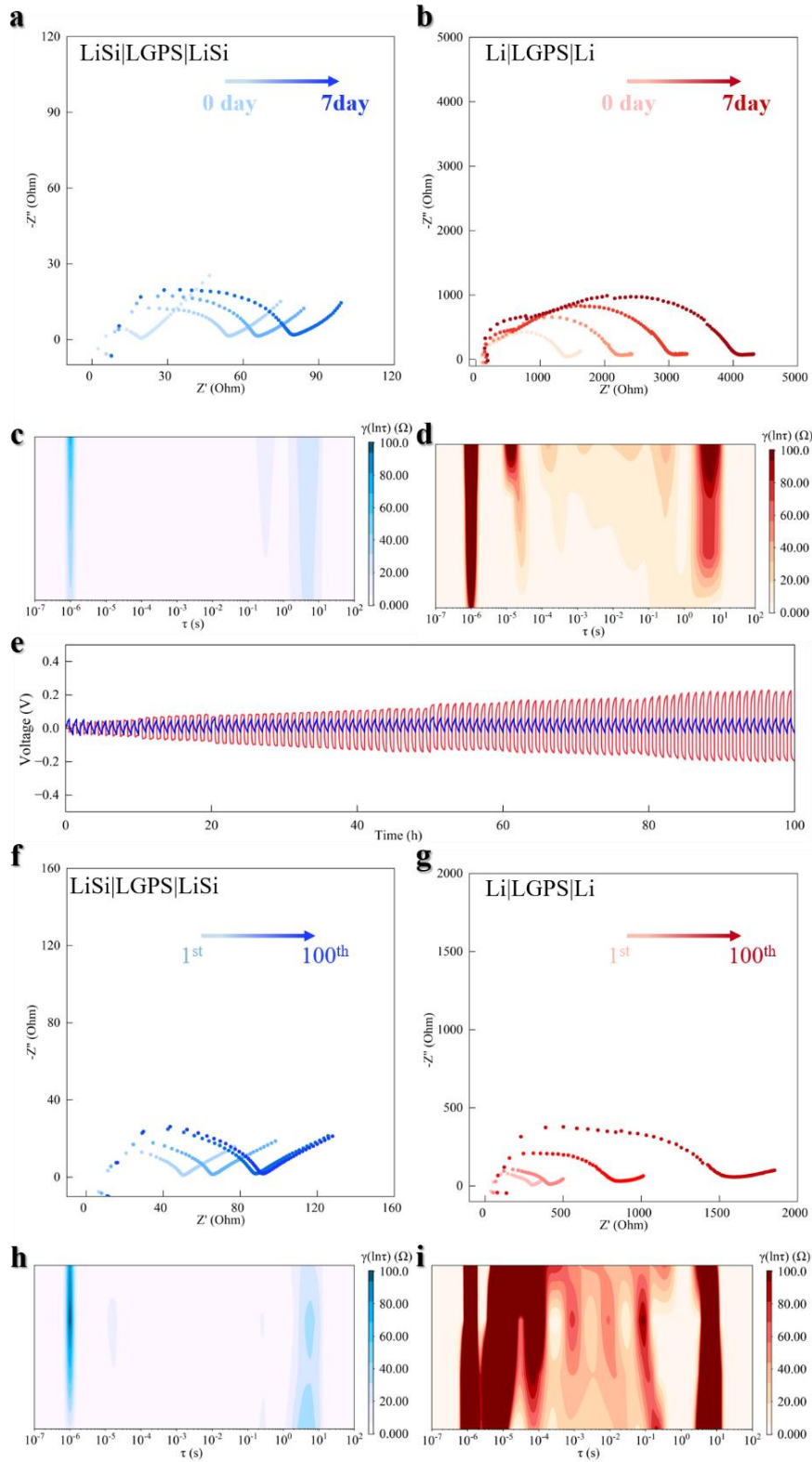


Figure S18. Electrochemical tests of ASSBs assembled with Li and LiSi negative electrodes. (a) Impedance variation of the LiSi|LGPS|LiSi ASSB under static conditions. (b) Impedance variation of the Li|LGPS|Li ASSB under static conditions. (c) DRT analysis corresponding to the impedance tests of the LiSi|LGPS|LiSi cell under static conditions. (d) DRT analysis corresponding to the impedance tests of the Li|LGPS|Li

cell under static conditions. (e) Long-term Li plating/stripping of the LiSi|LGPS|LiSi and Li|LGPS|Li symmetric cells, with a current density of 0.1 mA/cm^2 and an area capacity of 0.05 mAh/cm^2 . (f) Impedance tests of the LiSi|LGPS|LiSi cell at different operating times. (g) Impedance tests of the Li|LGPS|Li cell at different operating times. (h) DRT analysis corresponding to the impedance tests of the LiSi|LGPS|LiSi cell. (i) DRT analysis corresponding to the impedance tests of the Li|LGPS|Li cell.

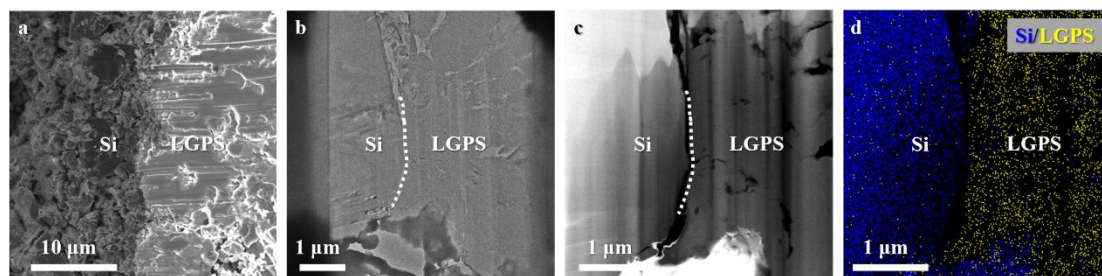


Figure S19. Characterization of the pristine Si/LGPS interface before electrochemical cycling. (a) A cryo-SEM imaging of the Si/LGPS pristine interface. (b) Bright-field image of the Cryo-FIB processed sample transferred to Cryo-TEM. (c) HAADF image corresponding to (b). (d) EDS mapping corresponding to (b) and (c).

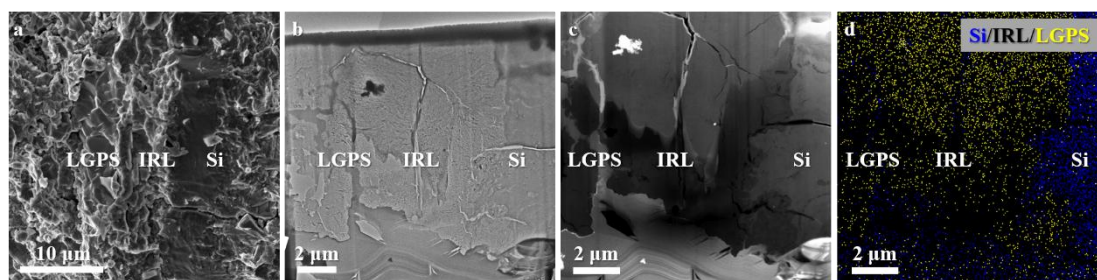


Figure S20. Characterization of the IRL about the Si/LGPS interface at fully charged state after 100 cycles. (a) A cryo-SEM image. (b) Bright-field image of the Cryo-FIB processed sample transferred to Cryo-TEM. (c) HAADF image corresponding to (b). (d) EDS mapping corresponding to (b) and (c).

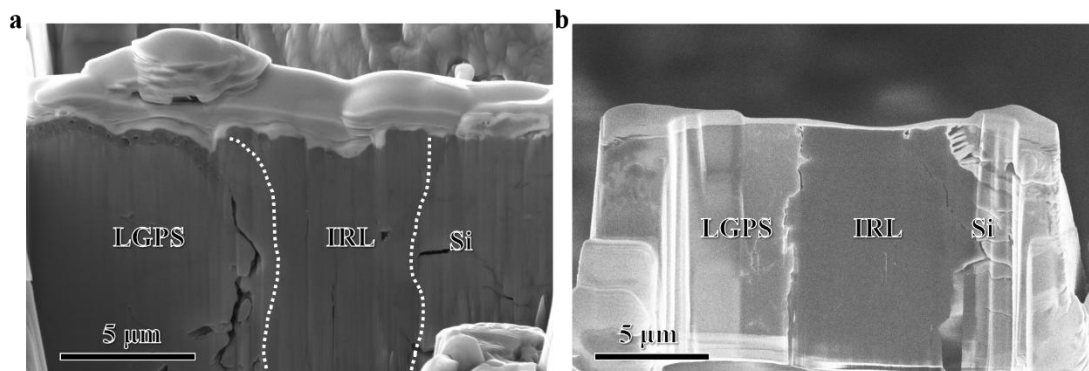


Figure S21. Characterization of the Si/LGPS lamellar at fully charged state after 100 cycles. (a) SE image of the Si/LGPS interface after 100 cycles. (b) The lamellar of the Si/LGPS interface after FIB thinning.

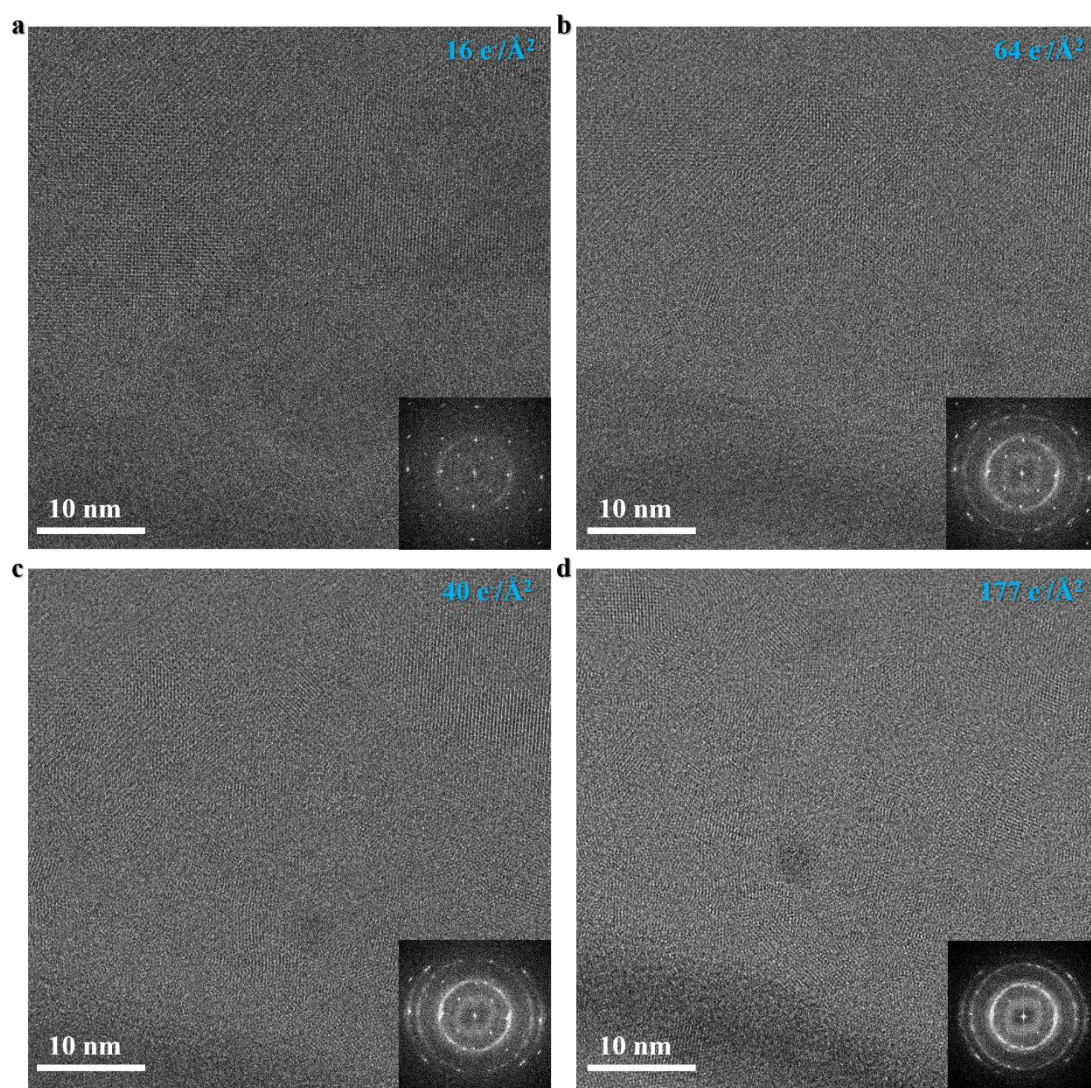


Figure S22. Dose damage test of the LGPS electrolyte. Cryo-HRTEM imaging and its corresponding FFT at: (a) 16 e-/Å²; (b) 40e-/Å²; (c) 64 e-/Å². (d) 177 e-/Å².

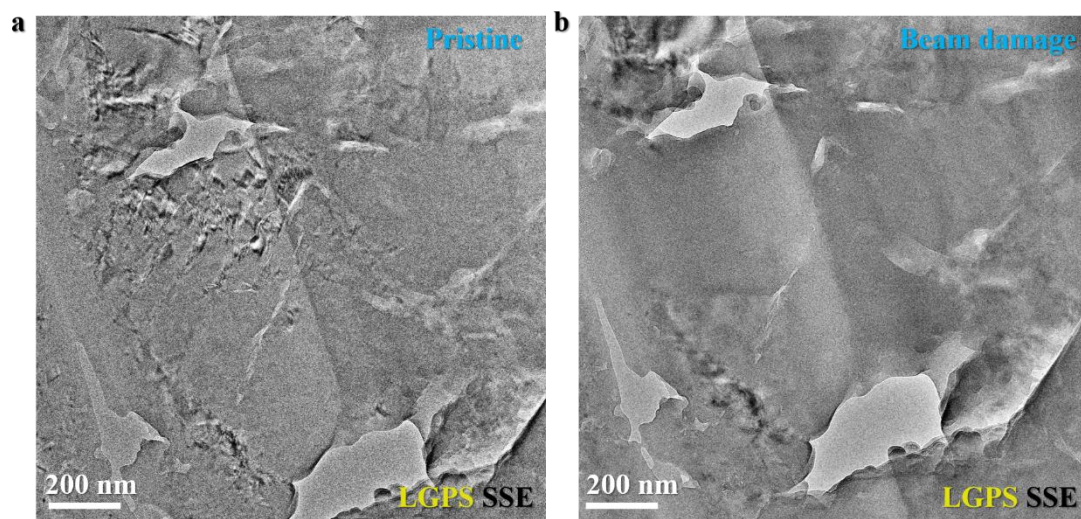


Figure S23. Dose damage test of the LGPS electrolyte. (a) The BF image of the pristine LGPS. (b) The BF image after electron beam irradiation damage.

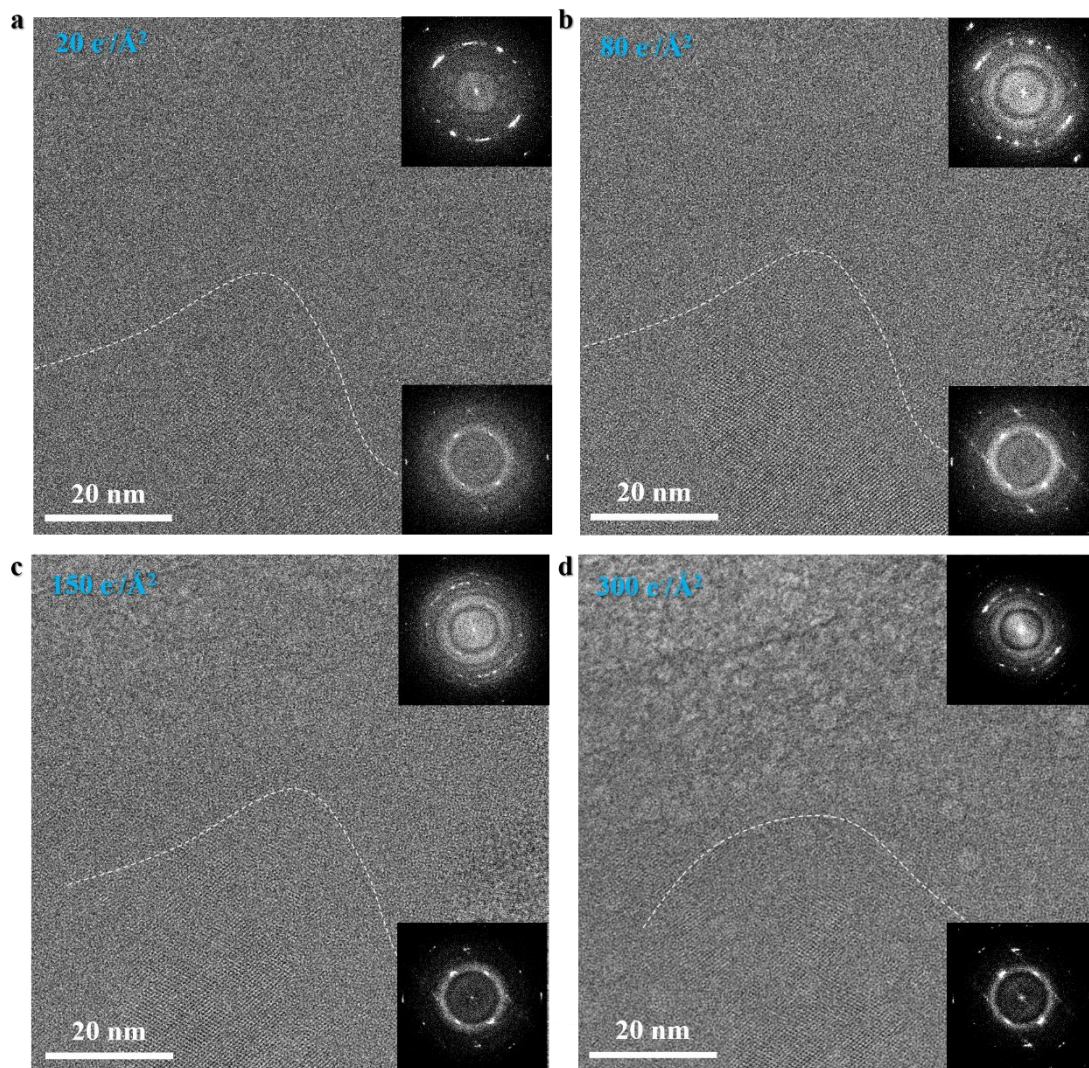


Figure S24. Dose damage test of the IRL of the Si/LGPS interface. Cryo-HRTEM imaging and its corresponding FFT at: (a) 20 $\text{e}^-/\text{\AA}^2$; (b) 80 $\text{e}^-/\text{\AA}^2$; (c) 150 $\text{e}^-/\text{\AA}^2$; (d) 300 $\text{e}^-/\text{\AA}^2$.

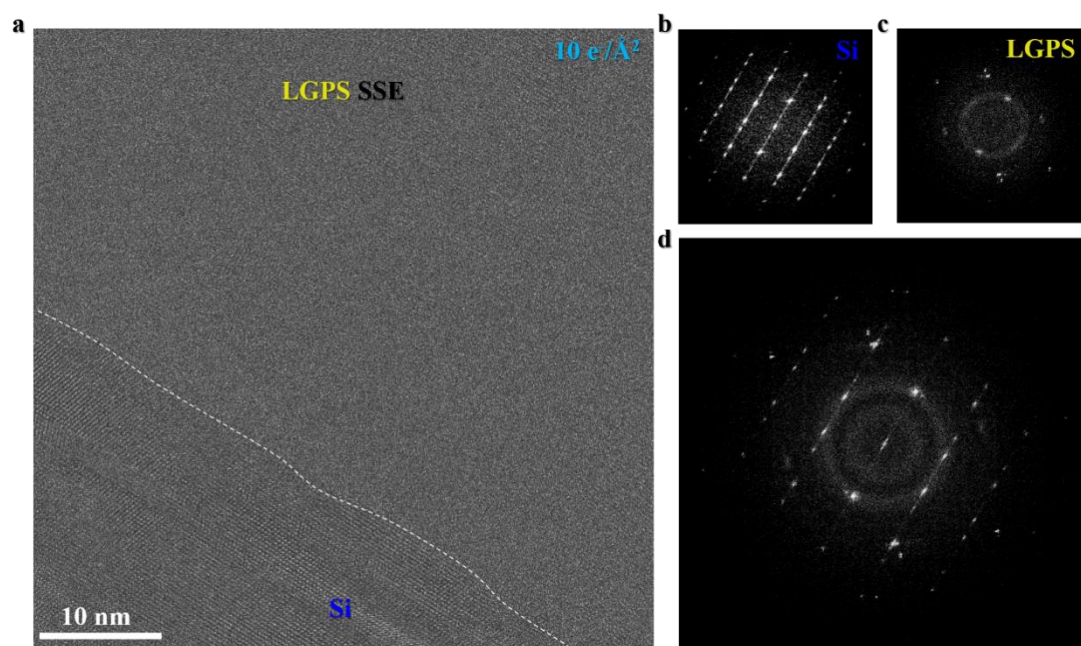


Figure S25. Cryo low-dose HRTEM imaging of the pristine Si/LGPS interface before electrochemical cycling. (a) HRTEM image. (b) FFT of the Si negative electrode. (c) FFT of the LGPS electrolyte. (d) FFT of the entire Si/LGPS interface.

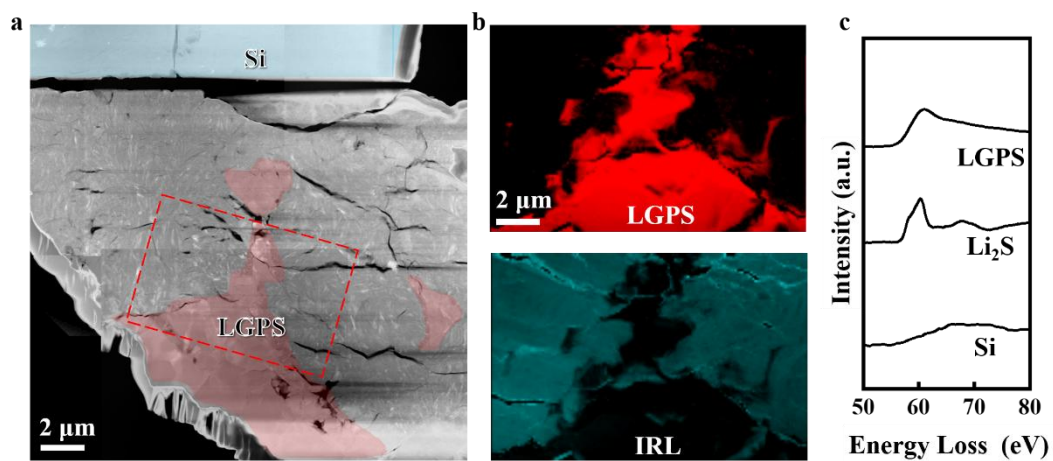


Figure S26. Cryo-STEM characterization of the Si/LGPS interface at fully charged state after 100 cycles. (a) Cryo-STEM image of the Si/LGPS interface after 100 cycles, and (b) Li-K edge mapping. (c) Reference Li K-edge EELS spectra obtained from standard LSPSC, Li₂S, μ -Si.

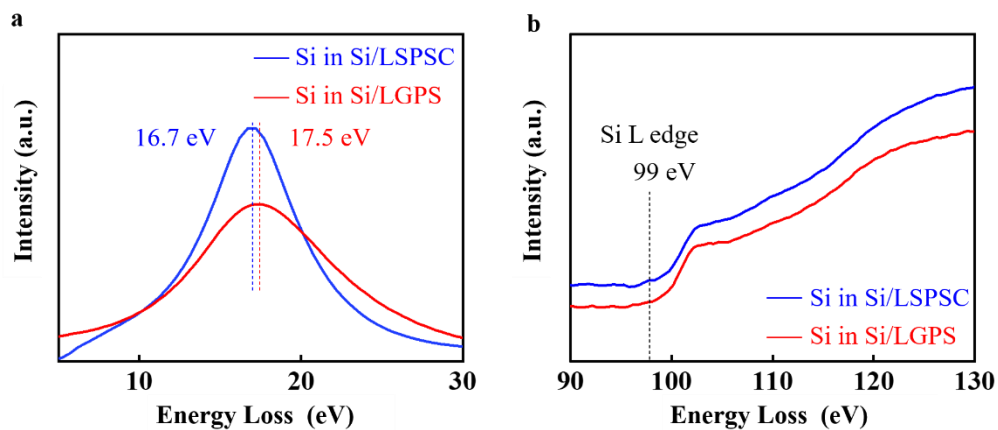


Figure S27. Cryo-STEM EELS characterization of the Si/sulfide interface at fully charged state after 100 cycles. (a) Cryo-STEM EELS revealing the plasmon signals of Si negative electrode from Si/Sulfide interface after 100 cycles. (b) STEM-EELS revealing Si-L edge signals of Si negative electrode after cycle.

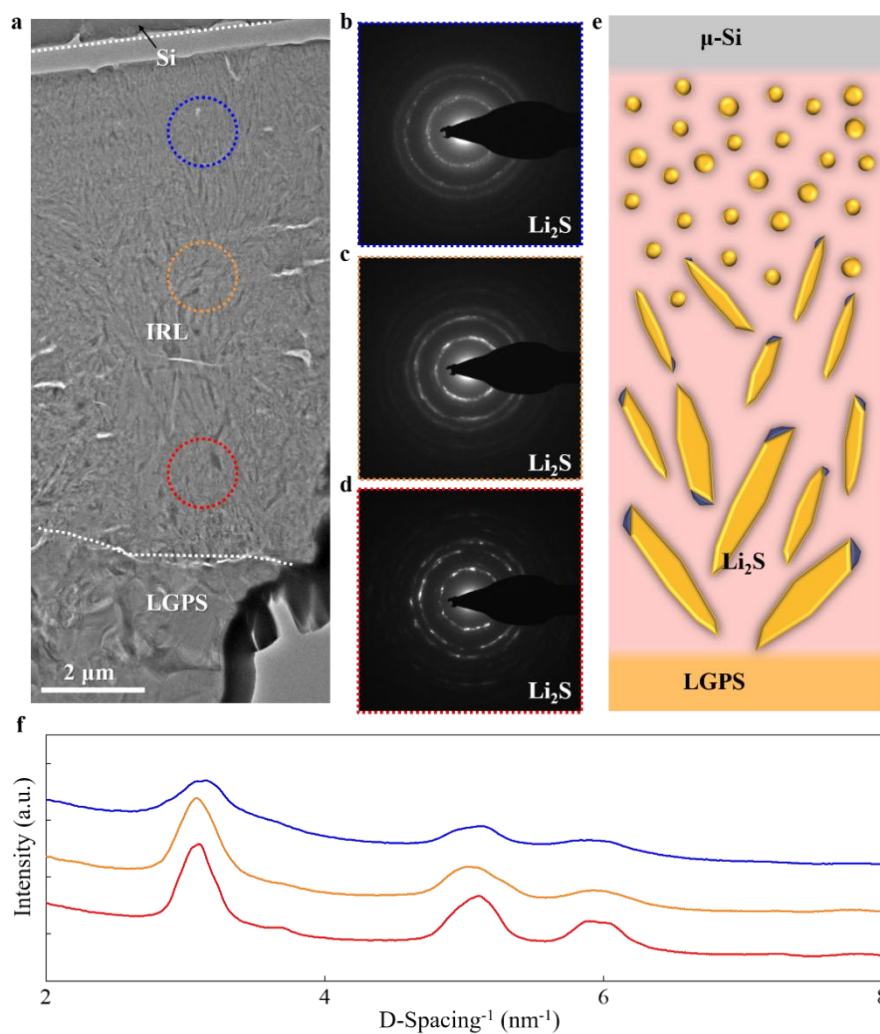


Figure S28. Characterization of the Si/LGPS interface at fully charged state after 100 cycles. (a) Bright field (BF) image of the Si/LGPS interface. (b-d) The electron diffraction pattern evolution of the interphase. (e) Schematic of the Li₂S shape and distribution. (f) Diffraction integral diagram about the IRL of the Si/LGPS interface.

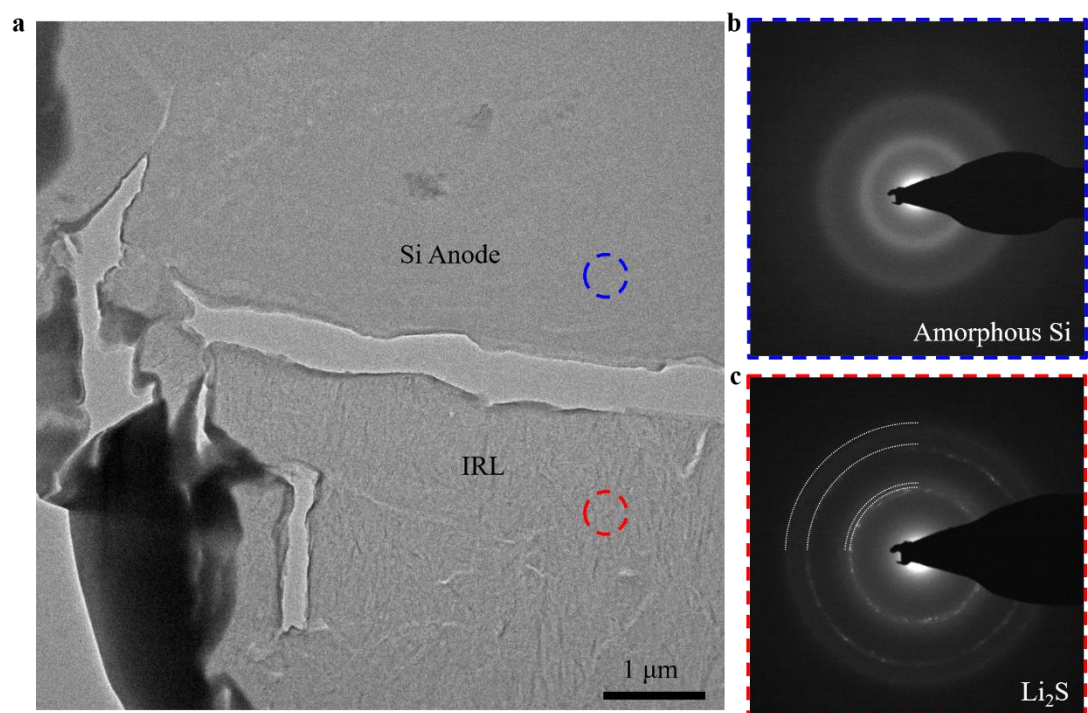


Figure S29. Characterization of the Si/LGPS interface at fully charged state after 100 cycles. (a) TEM image and diffraction analysis of (b) the IRL from the Si/LGPS interface after 100 cycles, and (c) the Si negative electrode.

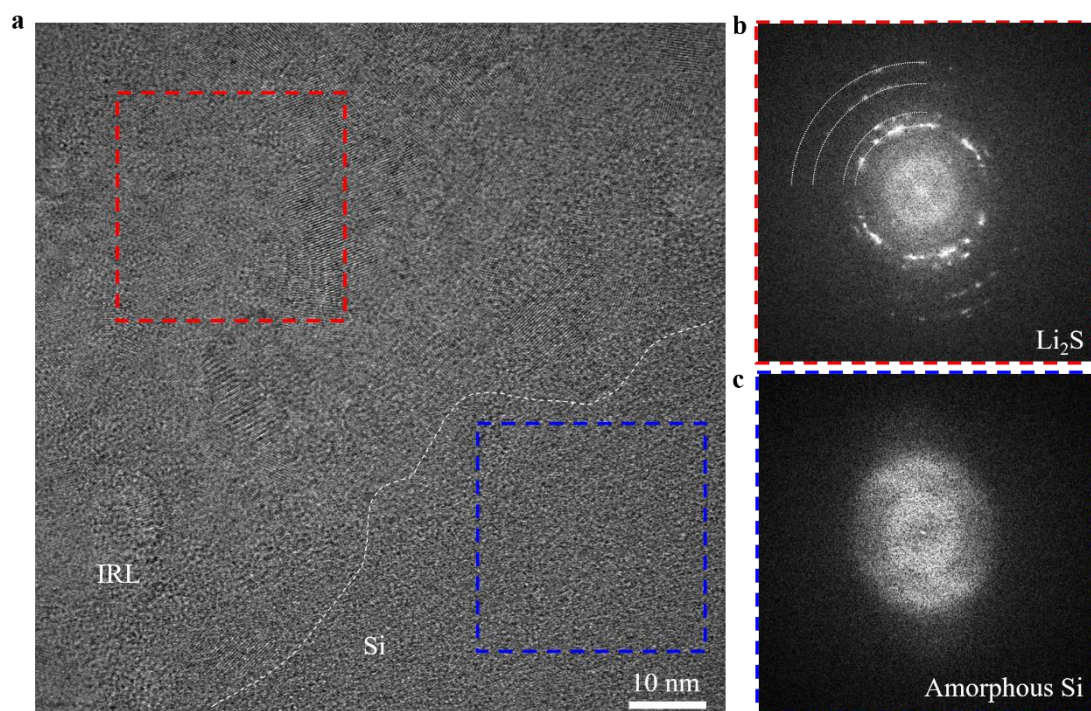


Figure S30. Cryo-HRTEM revealing the Si/LGPS interface of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) High-resolution transmission electron microscopy (HRTEM) image of the IRL from the Si/LGPS interface after 100 cycles, and (b, c) the corresponding FFT.

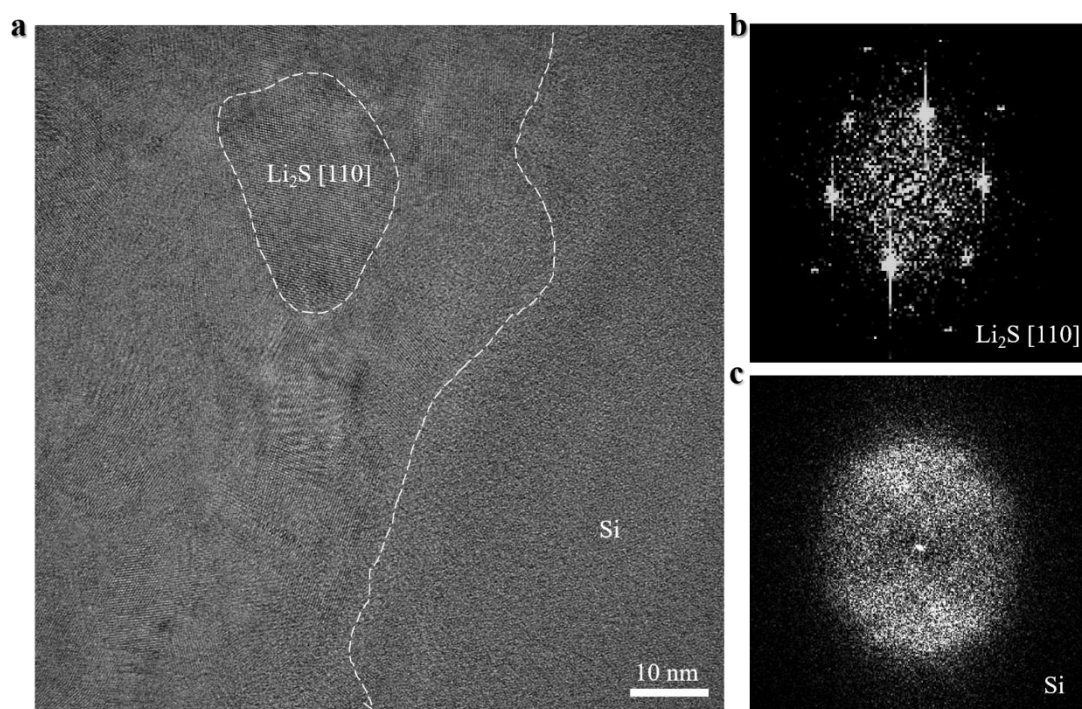


Figure S31. Cryo-HRTEM revealing the Si/LGPS interface of the Si|LGPS|NMC811 ASSB at fully charged state after 30 cycles. (a) Cryo-HRTEM image of the IRL in the Si/LGPS interface. (b) FFT of Li_2S in the IRL. (c) FFT of Si at the interface.

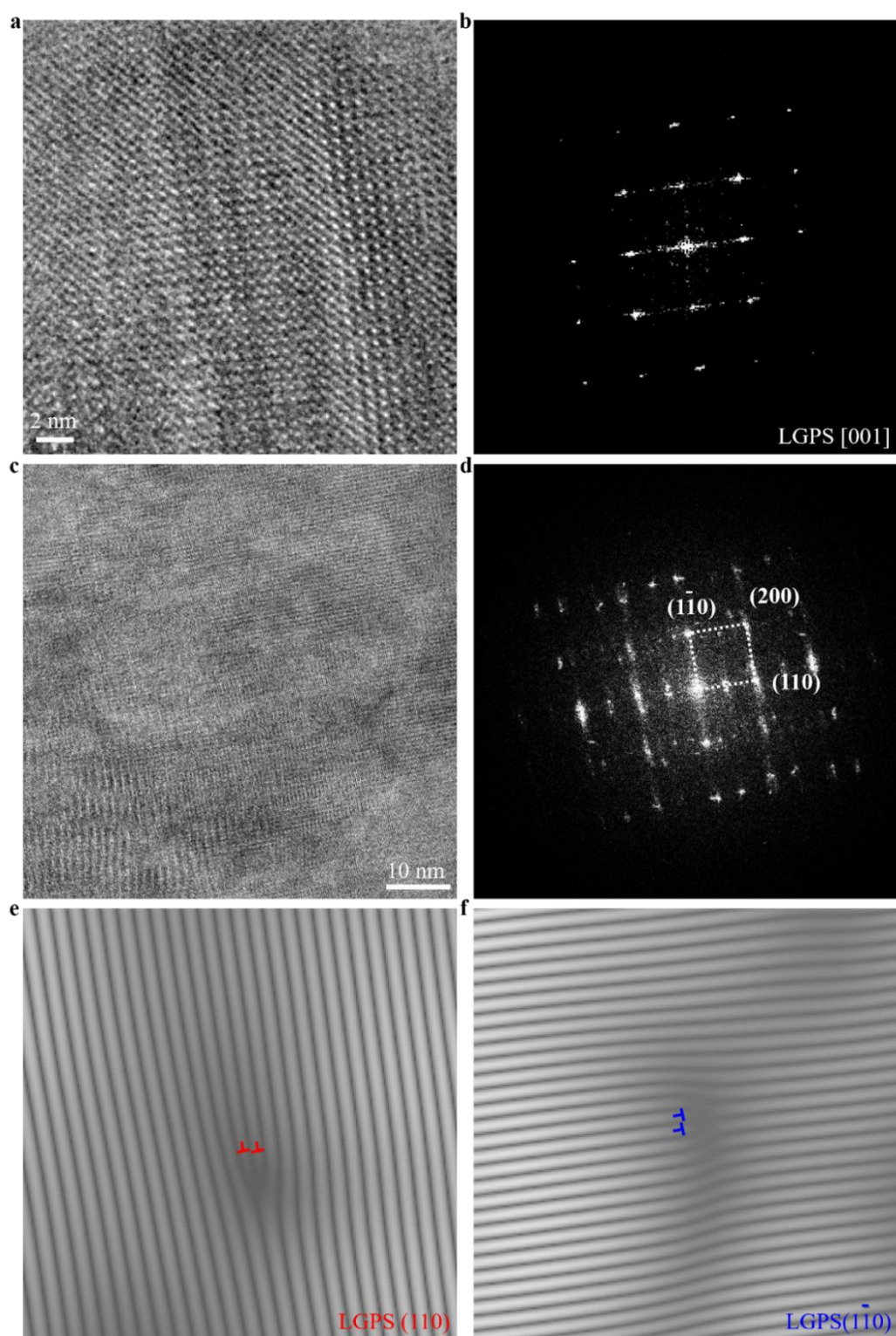


Figure S32. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image revealing the lattice distortion of in the non-reacted LGPS, and (b) the corresponding FFT. (c) Cryo-HRTEM revealing the lattice distortion in the non-reacted LGPS, and (d) the corresponding FFT. The inverse fast Fourier transform images of (e) (110) planes and (f) ($1\bar{1}1$) planes revealing the edge dislocations in the non-reacted LGPS.

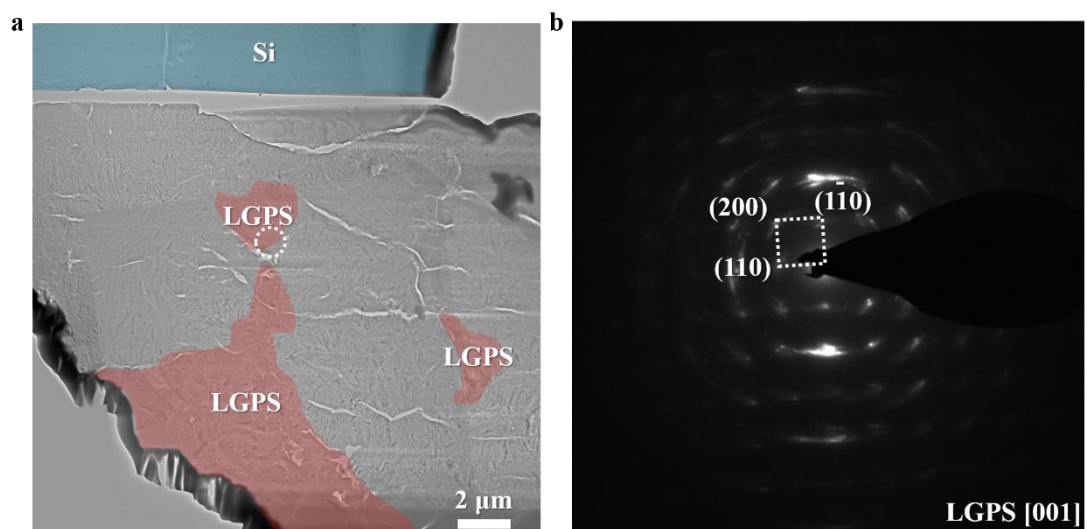


Figure S33. Characterization of the IRL about the Si/LGPS interface at fully charged state after 100 cycles. (a) The IRL from the Si/LGPS interface after 100 cycles, and (b) the corresponding electron diffraction from the dash circle.

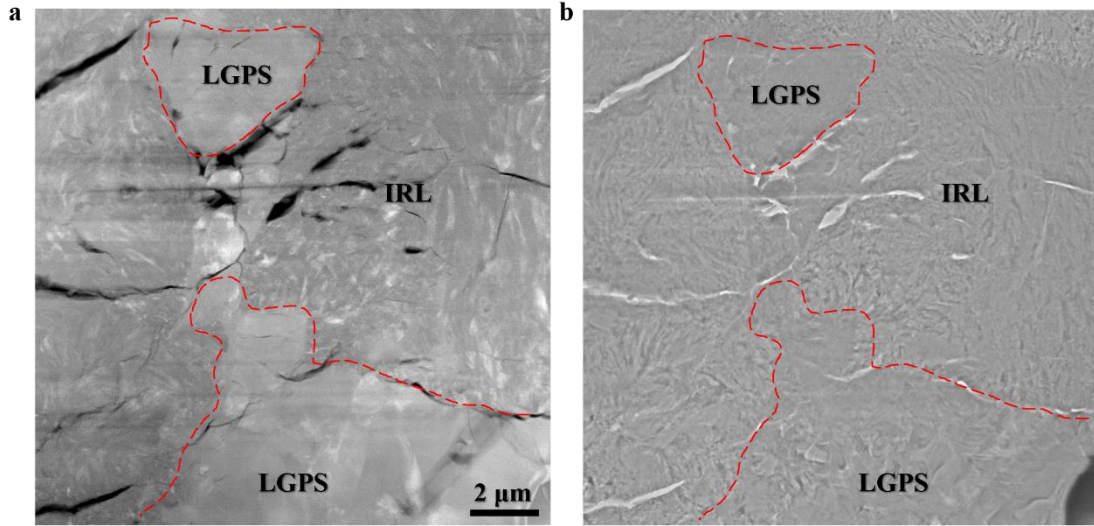


Figure S34. Characterization of the Si/LGPS interface at fully charged state after 100 cycles. (a) Annular dark field image of the Si/LGPS interface after 100 cycles. (Adopted from Figure S11) (b) Bright field image of the Si/LGPS interface after 100 cycles (Adopted from Figure 3a).

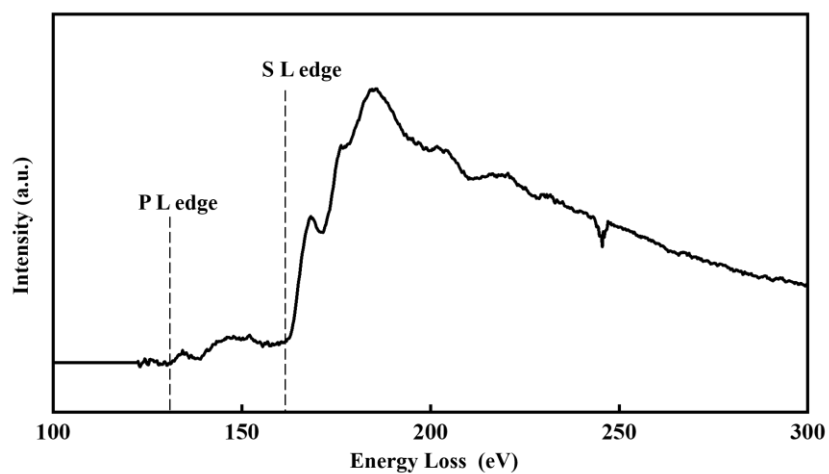


Figure S35. Cryo-STEM EELS of the IRL from the Si/LGPS interface at fully charged state after 100 cycles of the Si|LGPS|NMC811 ASSB.

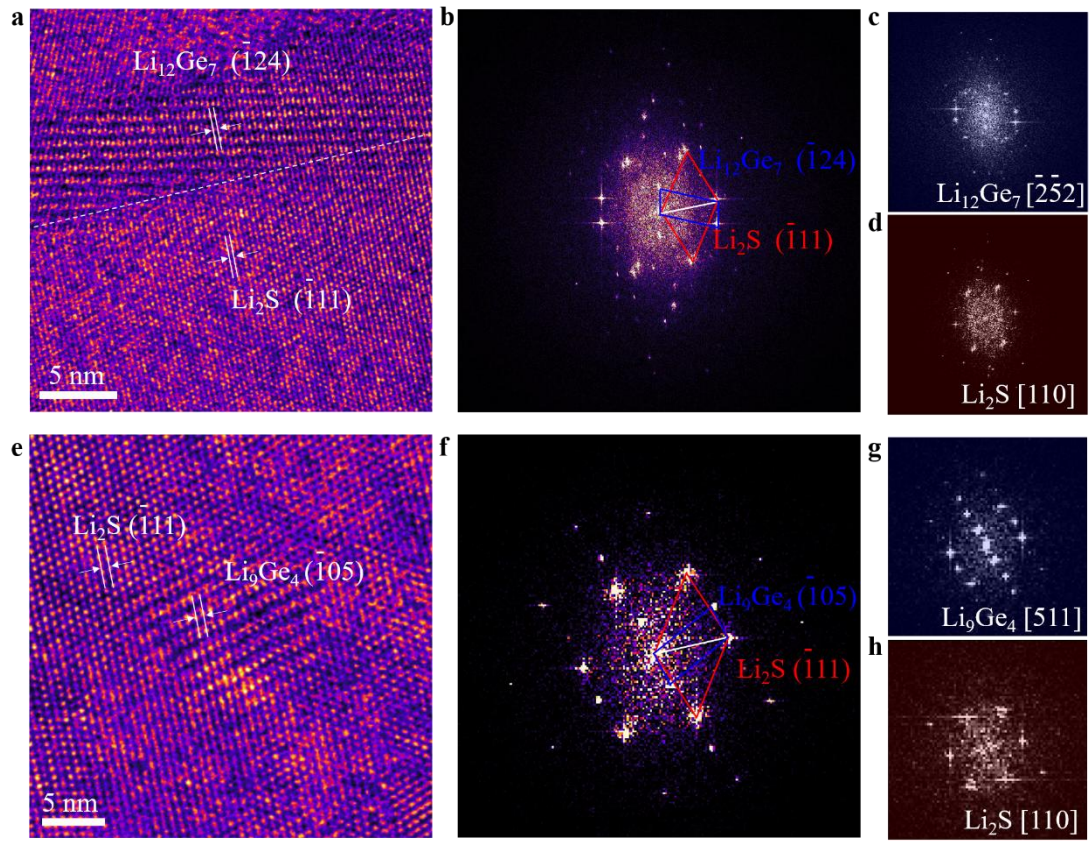


Figure S36. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image of the $\text{Li}_{12}\text{Ge}_7/\text{Li}_2\text{S}$ interface, and (b) the corresponding FFT of the entire region. (c) FFT of $\text{Li}_{12}\text{Ge}_7$ $[\bar{2}\bar{5}2]$, and (d) Li_2S $[110]$. (e) Cryo-HRTEM image of the $\text{Li}_9\text{Ge}_4/\text{Li}_2\text{S}$ interface, and (f) the corresponding FFT of the entire region. (g) FFT of Li_9Ge_4 $[511]$, and (h) FFT of Li_2S $[110]$.

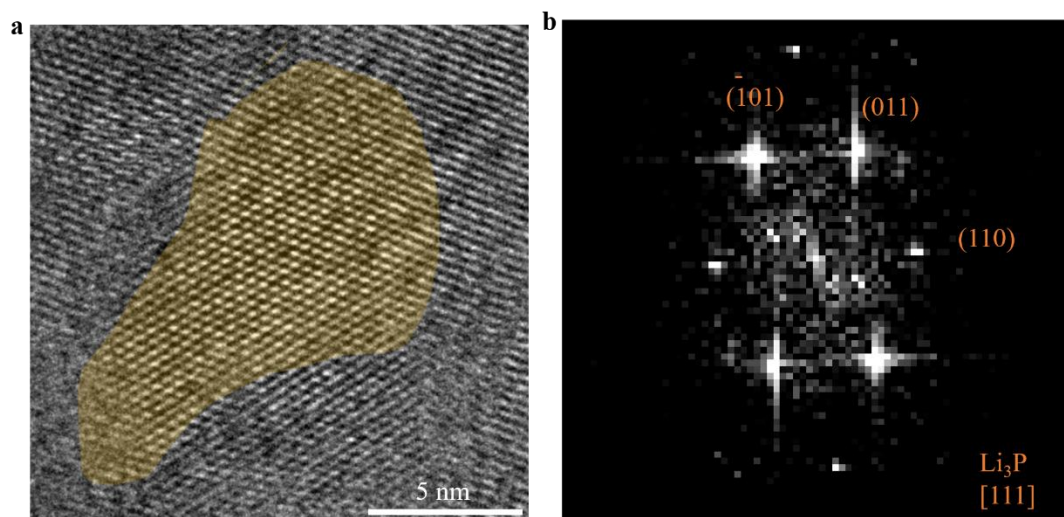


Figure S37. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image of Li₃P in the IRL from the Si/LGPS interface after 100 cycles, and (b) the corresponding FFT.

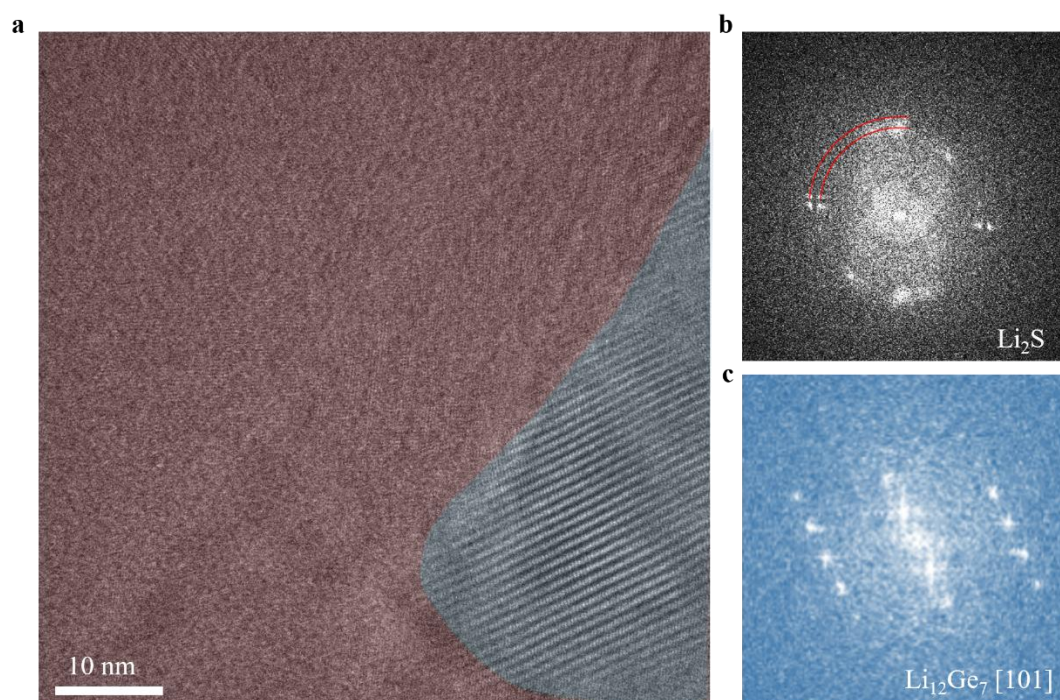


Figure S38. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image of Li-Ge and Li₂S in the IRL form the Si/LGPS interface after 100 cycles, and the corresponding FFT (b, c).

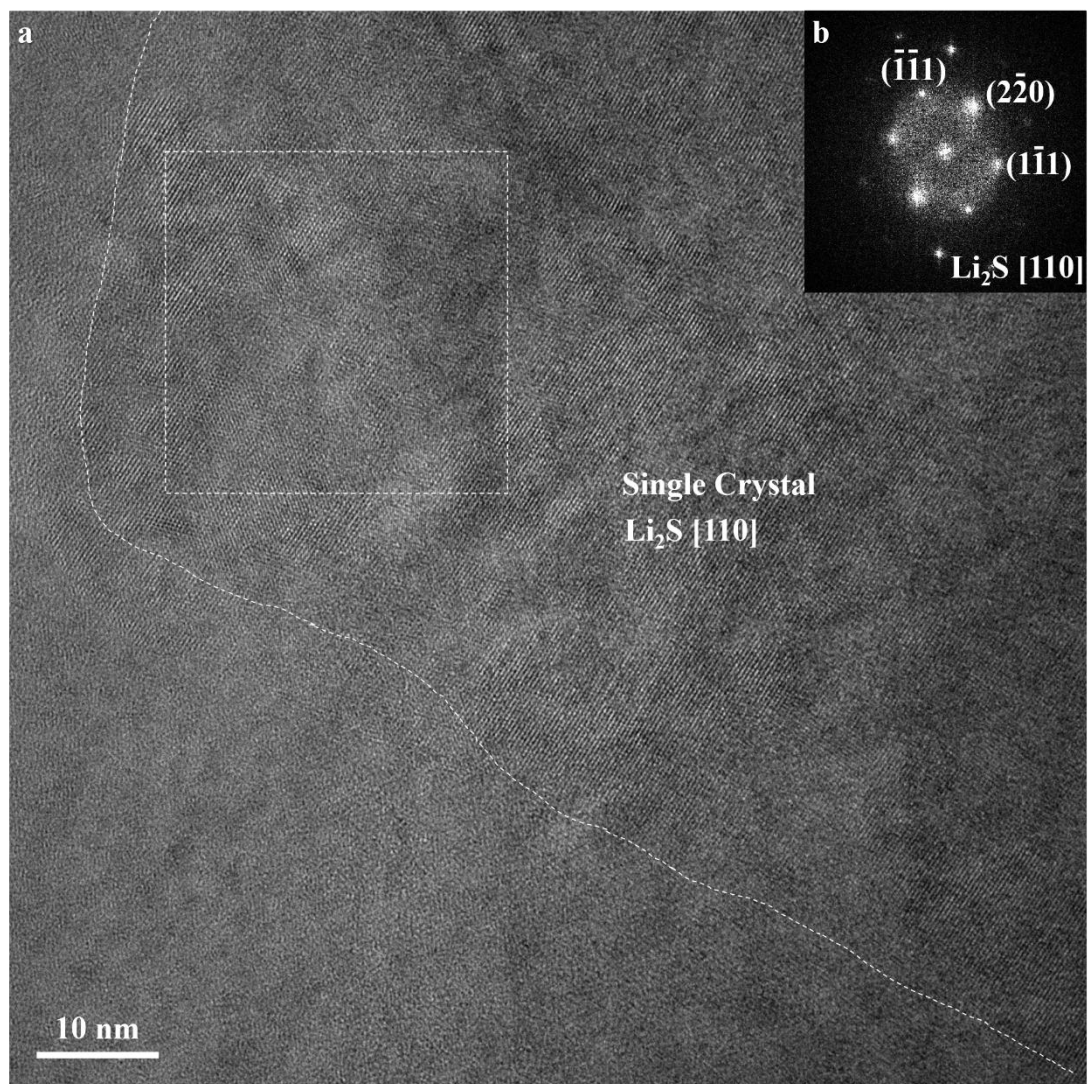


Figure S39. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image, and (b) the corresponding FFT of Li₂S from the IRL of the Si/LGPS interface.

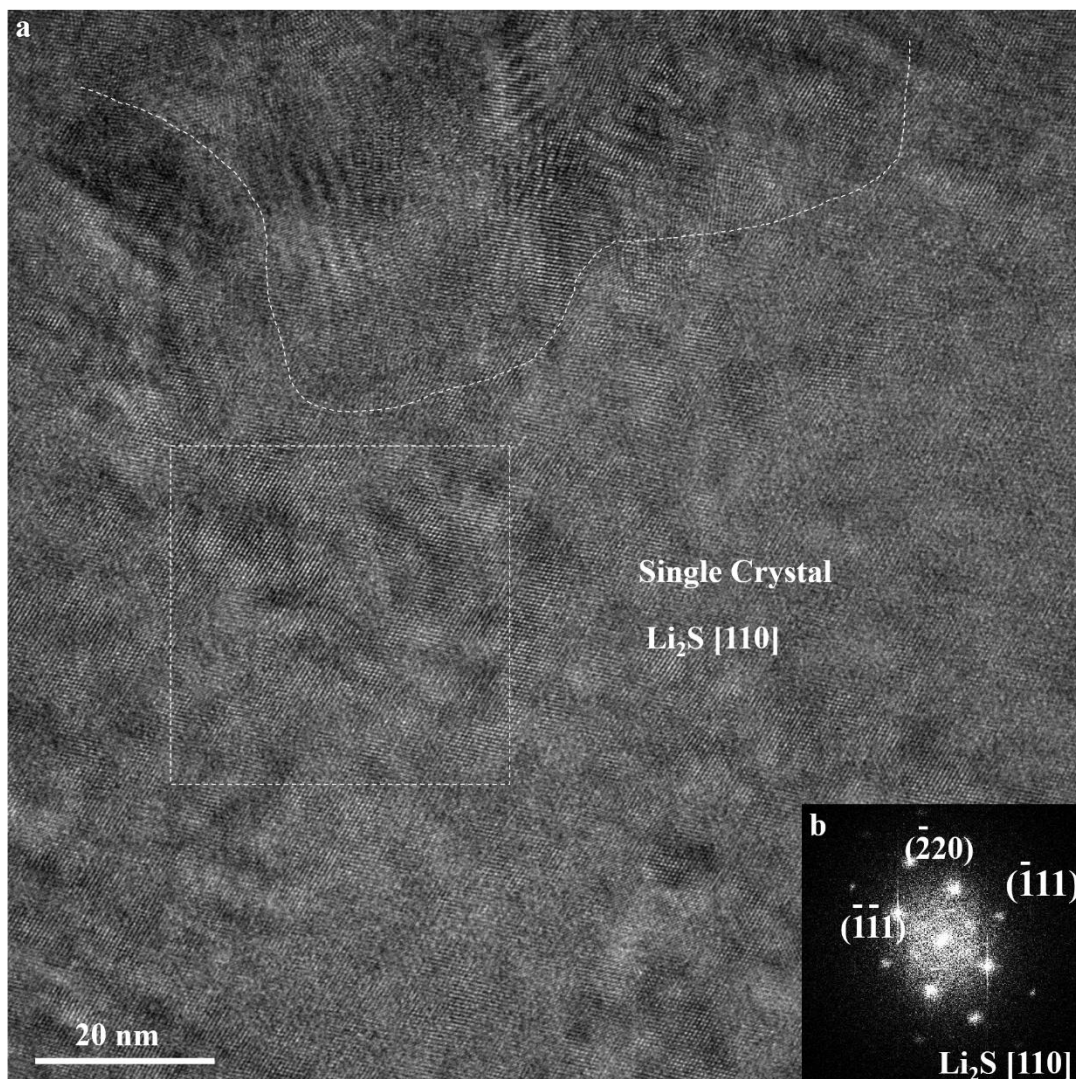


Figure S40. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image, and (b) the corresponding FFT of Li₂S from the IRL of the Si/LGPS interface.

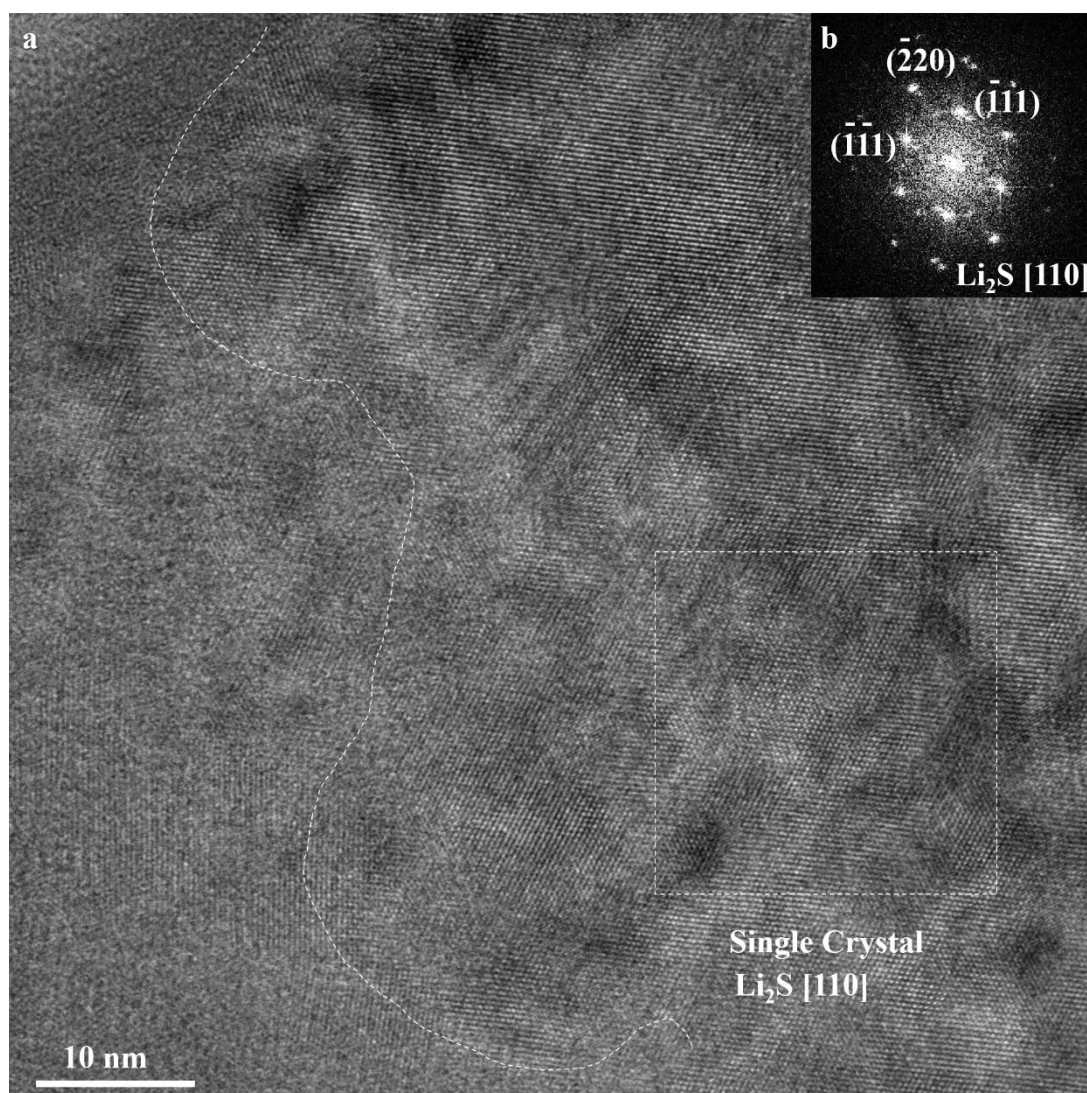


Figure S41. Cryo-HRTEM revealing the IRL of the Si|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Cryo-HRTEM image, and (b) the corresponding FFT of Li₂S from the IRL of the Si/LGPS interface.

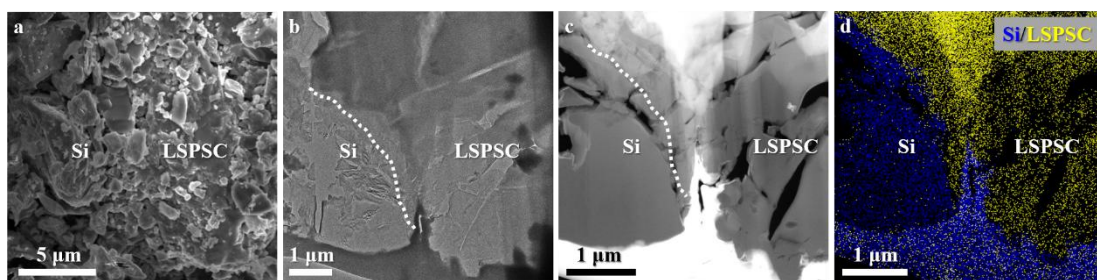


Figure S42. Characterization of the pristine Si/LSPSC interface before electrochemical cycling. (a) A cryo-SEM image of the Si/LSPSC original interface. (b) Bright-field image of the Cryo-FIB processed sample transferred to Cryo-TEM. (c) HAADF image corresponding to (b). (d) EDS mapping corresponding to (b) and (c).

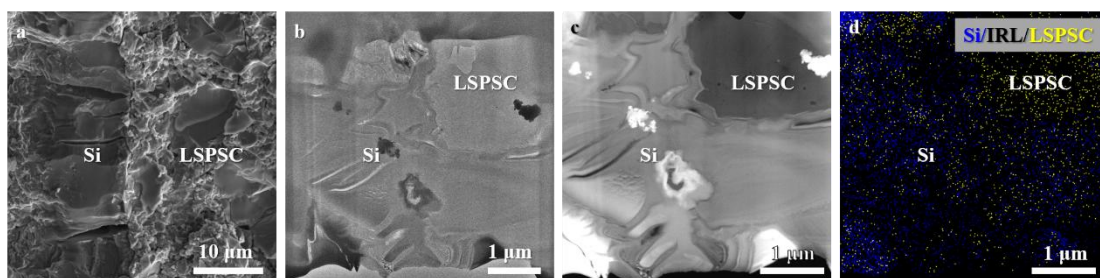


Figure S43. Characterization of the IRL about the Si/LSPSC interface at fully charged state after 100 cycles. (a) A cryo-SEM image. (b) Bright-field image of the Cryo-FIB processed sample transferred to Cryo-TEM. (c) HAADF image corresponding to (b). (d) EDS mapping corresponding to (b) and (c).

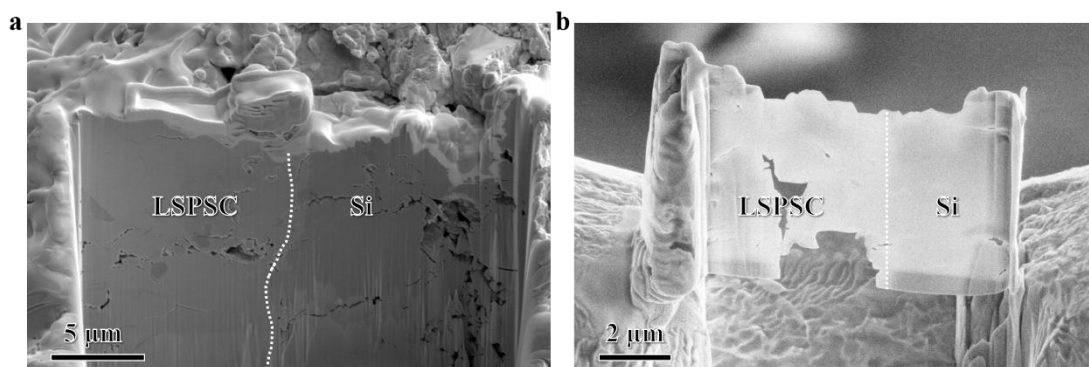


Figure S44. Characterization of the Si/LGPS lamellar at fully charged state after 100 cycles. (a) SE image of the Si/LSPSC interface after 100 cycles. (b) The lamellar of Si/LSPSC interface after FIB thinning.

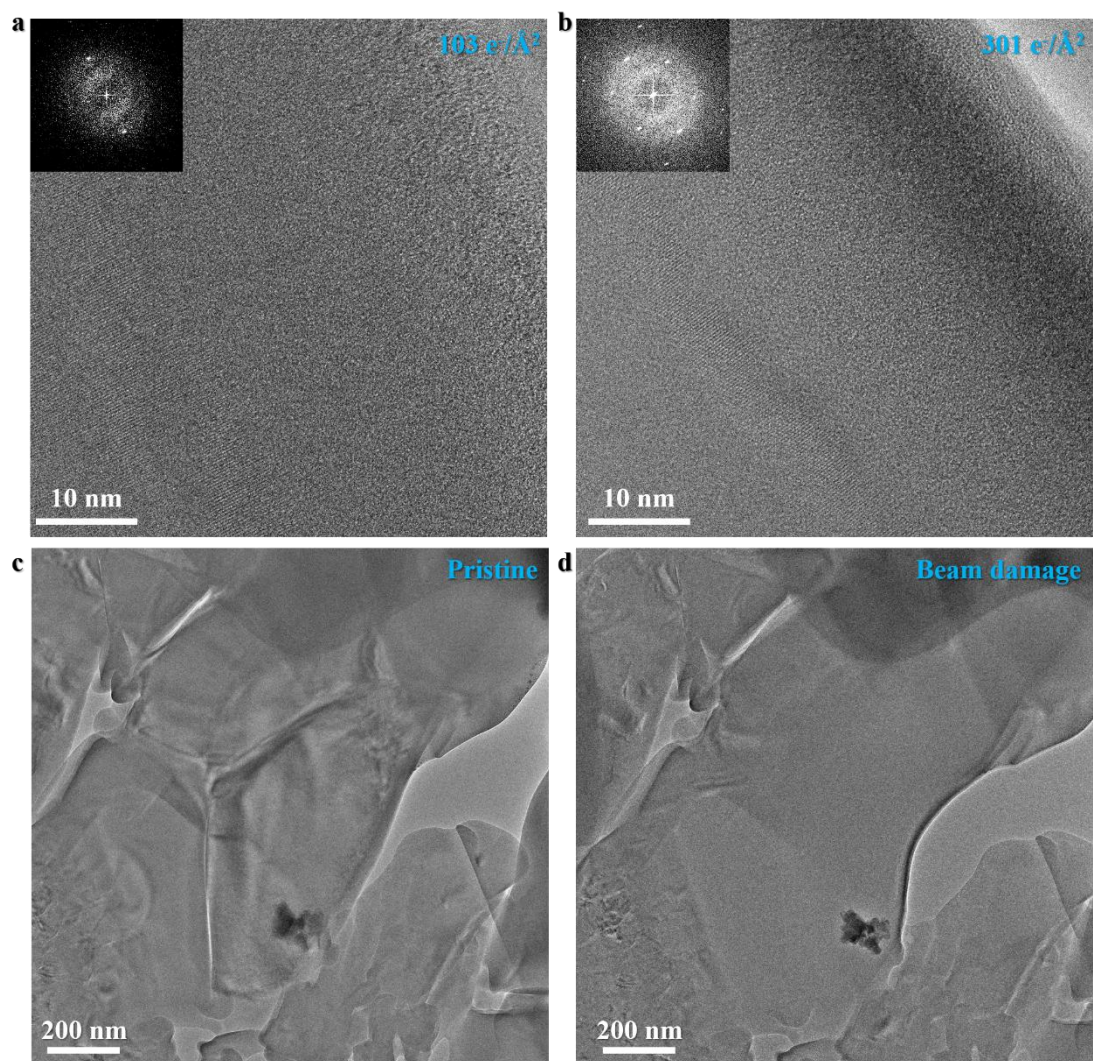


Figure S45. Dose damage test of the LSPSC electrolyte. (a) Cryo-HRTEM imaging and its corresponding FFT at 103 e⁻/Å². (b) Cryo-HRTEM imaging and its corresponding FFT at 301 e⁻/Å². (c) The pristine BF image of LSPSC. (d) The BF image of LSPSC after electron beam irradiation damage.

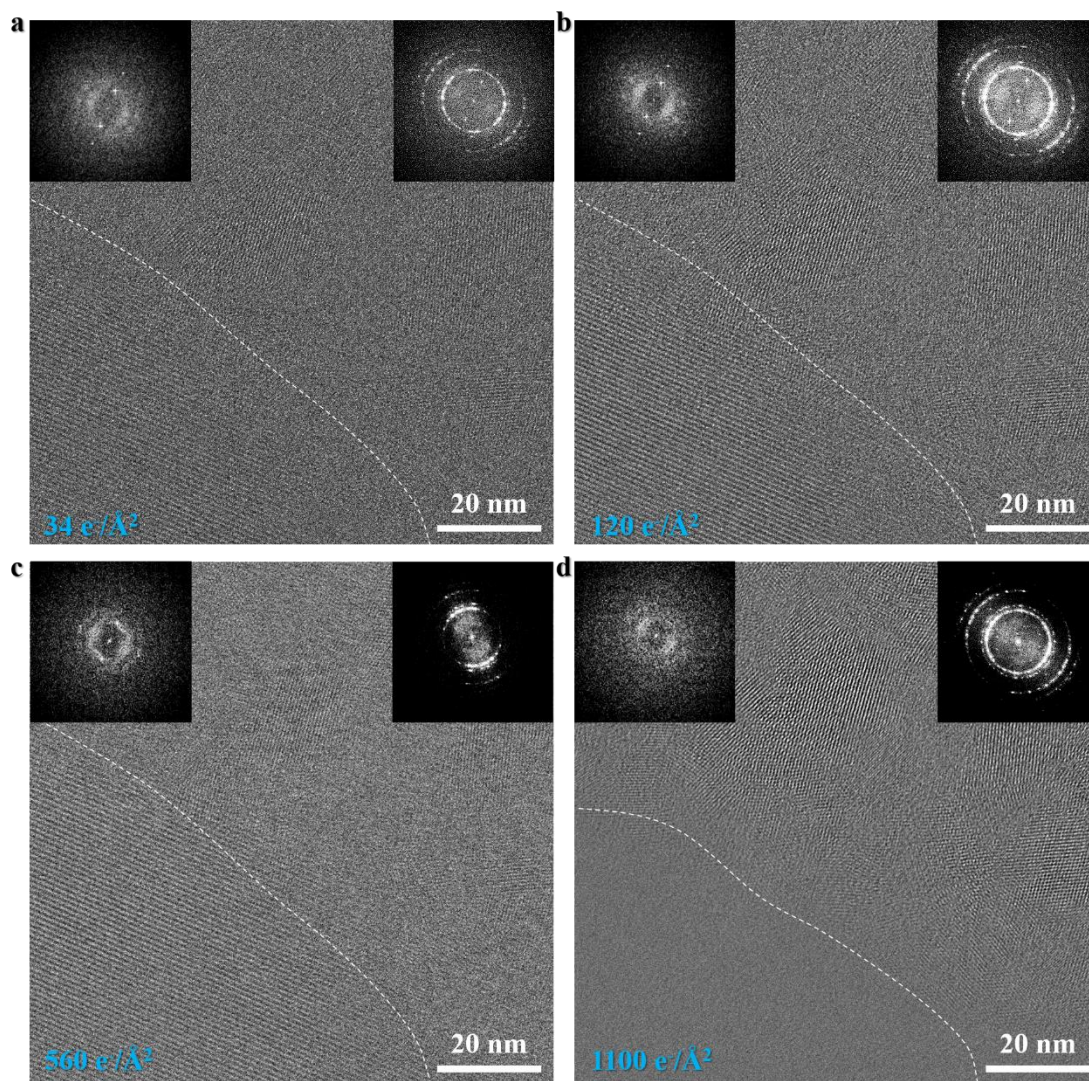


Figure S46. Dose damage test of the IRL of the Si/LSPSC interface. Cryo-HRTEM imaging and its corresponding FFT at: (a) 34 e⁻/Å²; (b) 120 e⁻/Å²; (c) 560 e⁻/Å²; (d) 1100 e⁻/Å².

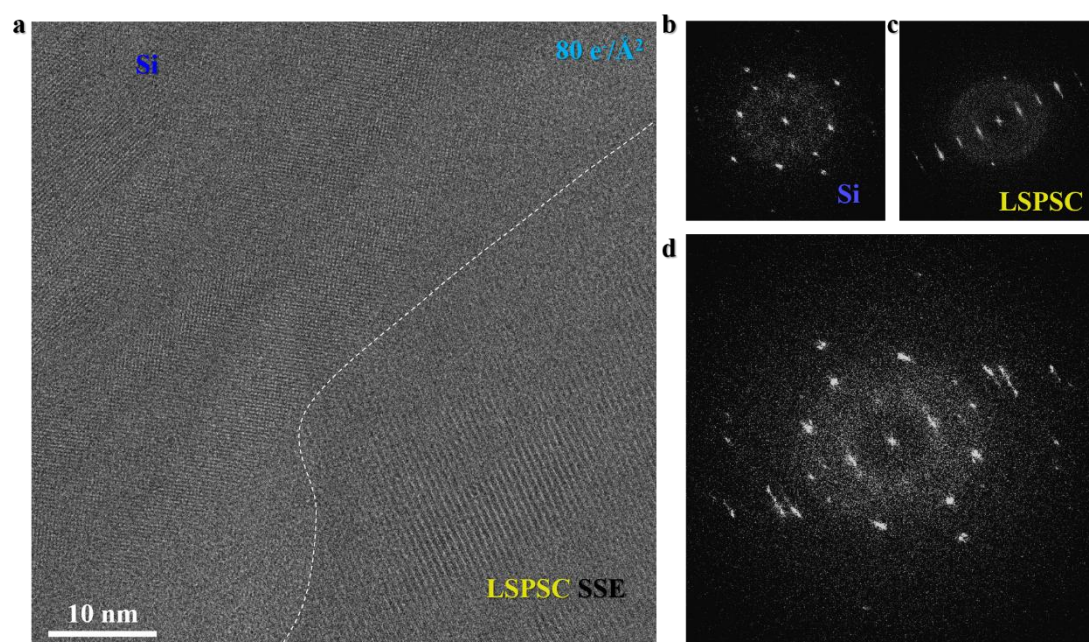


Figure S47. Cryo low-dose HRTEM imaging of the pristine Si/LSPSC interface before electrochemical cycling. (a) HRTEM image. (b) Fast Fourier Transform (FFT) of Si. (c) FFT of LSPSC. (d) FFT of the Si/LSPSC interface.

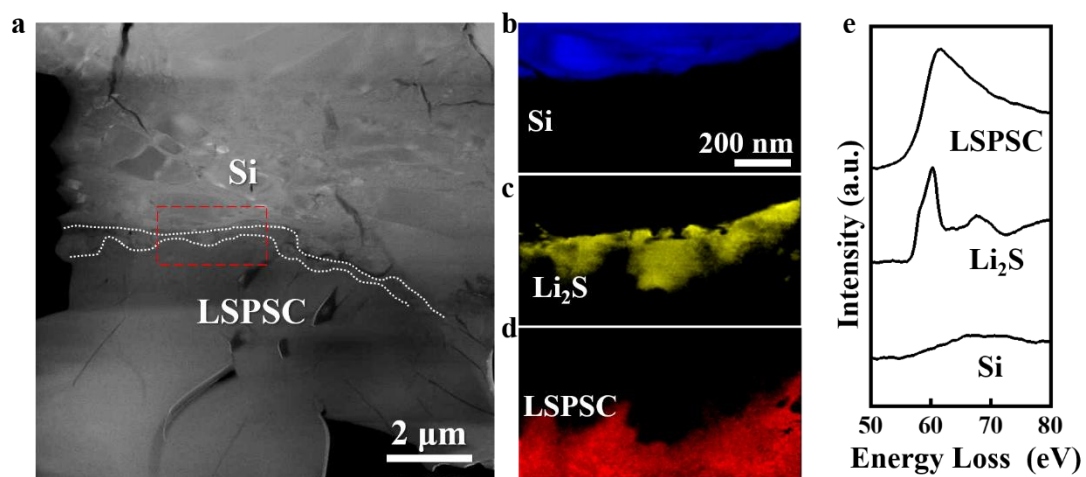


Figure S48. Cryo-STEM characterization of the Si/LSPSC interface at fully charged state after 100 cycles. (a) Cryo-STEM image of the Si/LSPSC interface. (b) EELS mapping of Si negative electrode. (c) EELS mapping of Li₂S production. (d) EELS mapping of LSPSC electrolyte. (e) Reference Li K-edge EELS spectra obtained from LSPSC, Li₂S, μ -Si.

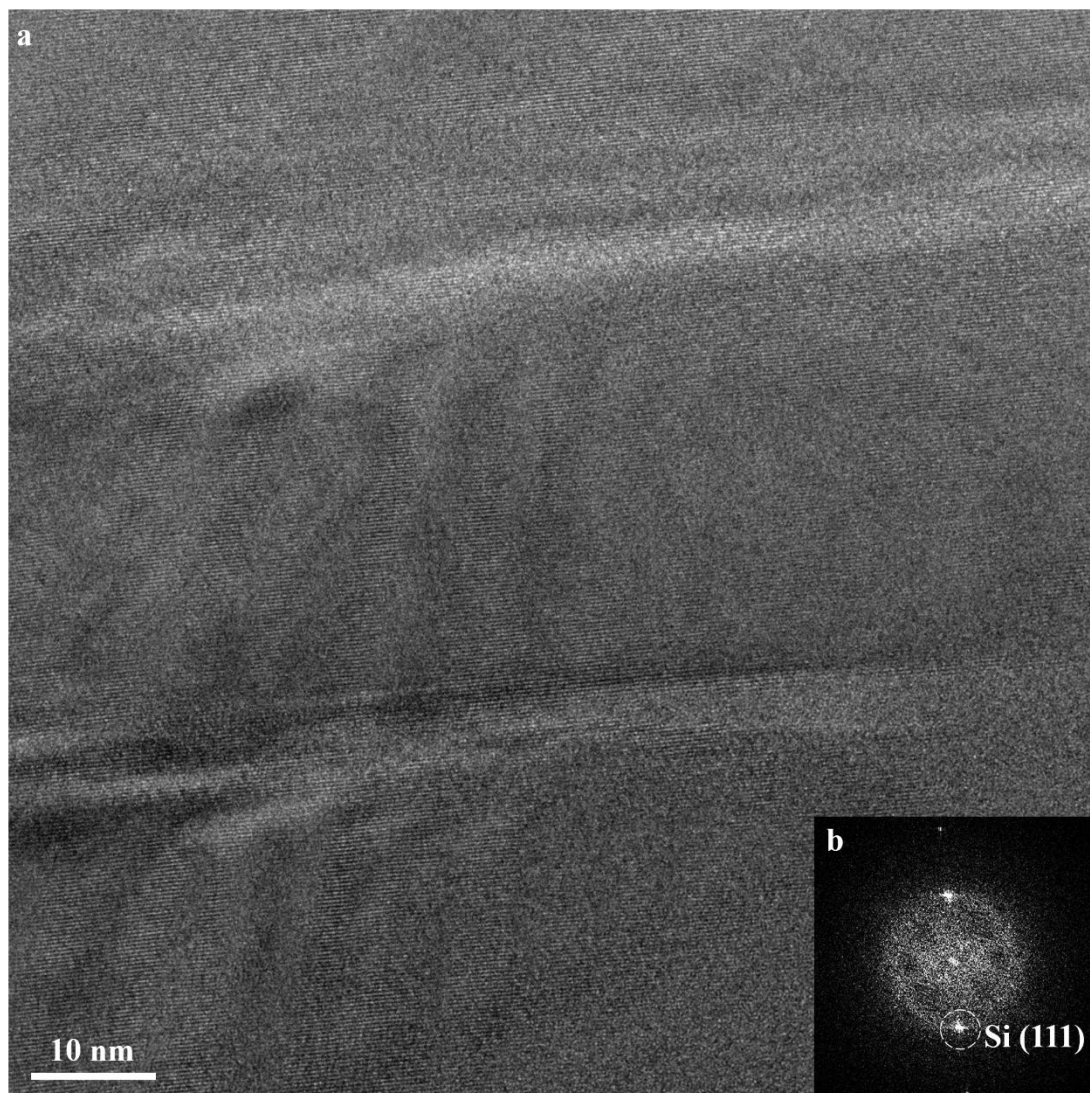


Figure S49. Characterization of the Si/LSPSC interface at fully charged state after 100 cycles. (a) Cryo-HRTEM image of Si negative electrode in the Si/LSPSC interface, and (b) the corresponding FFT.

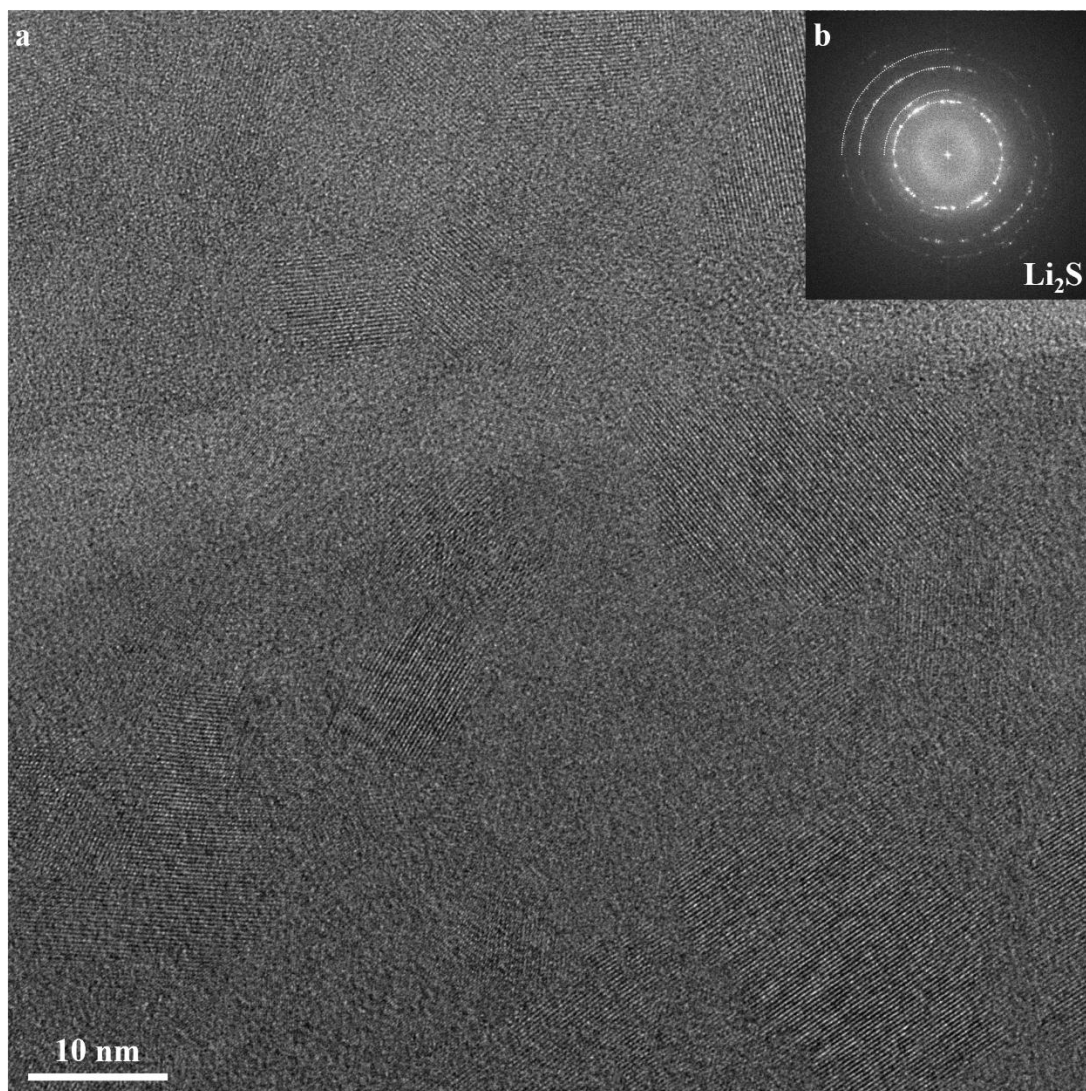


Figure S50. Characterization of the Si/LSPSC interface at fully charged state after 100 cycles. (a) Cryo-HRTEM image of the IRL from the Si/LSPSC interface, and (b) the corresponding FFT.

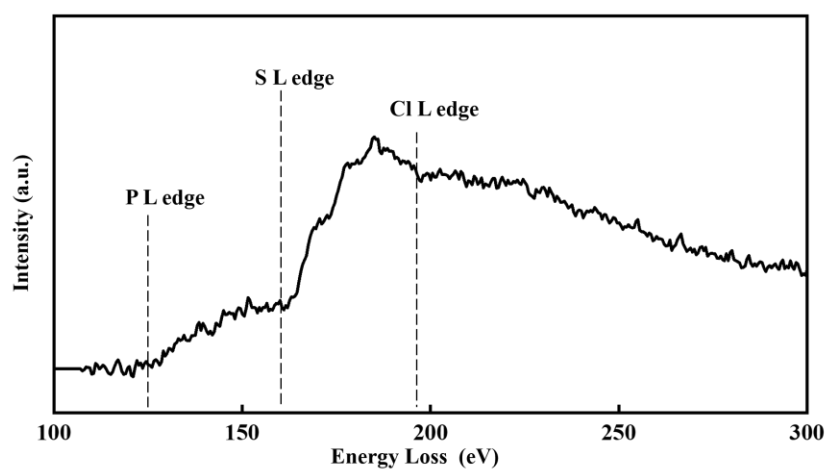


Figure S51. Cryo-STEM EELS of the IRL from the Si/LSPSC interface at fully charged state after 100 cycles of the Si|LSPSC|NMC811 ASSB.

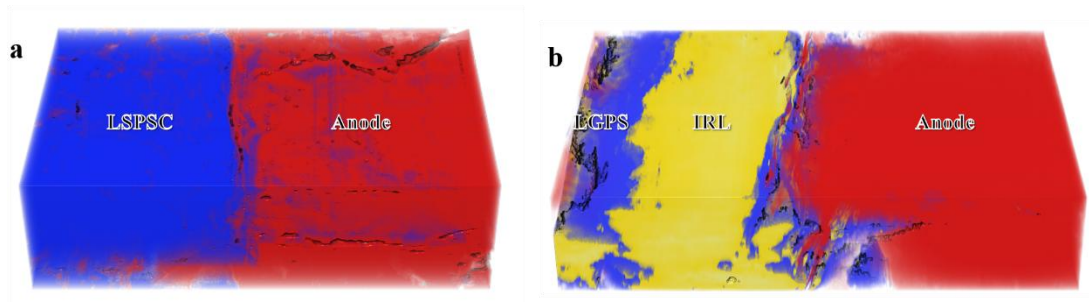


Figure S52. Three-dimensional FIB reconstruction of the μ -Si negative electrode and sulfide electrolyte interface: (a) Si/LSPSC, (b) Si/LGPS.

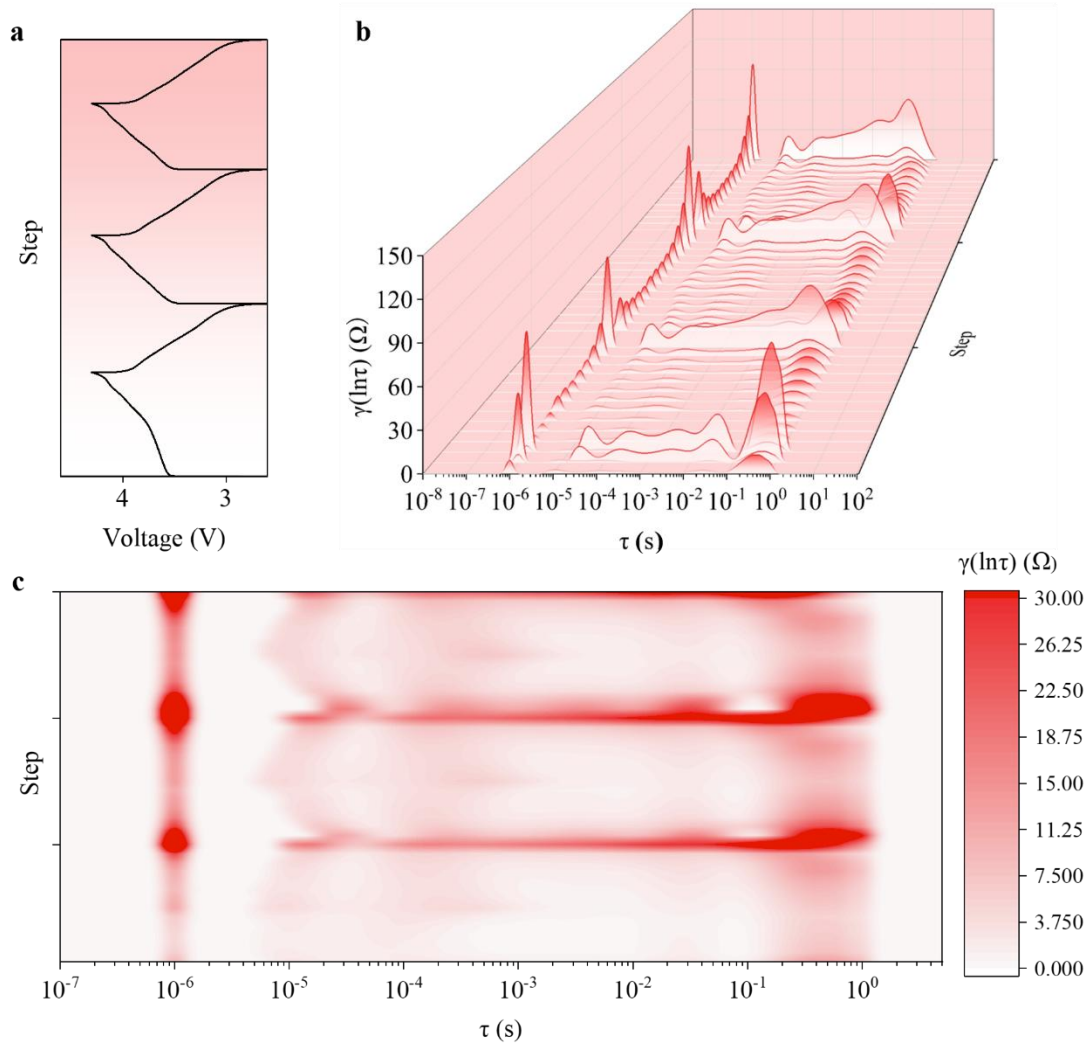


Figure S53. The EIS evolution of the first three cycles from the Si|LGPS|NMC811 ASSB at 70 mA/g at 25 °C. (a) Time–voltage profiles. (b, c) DRT transformation obtained from the EISs.

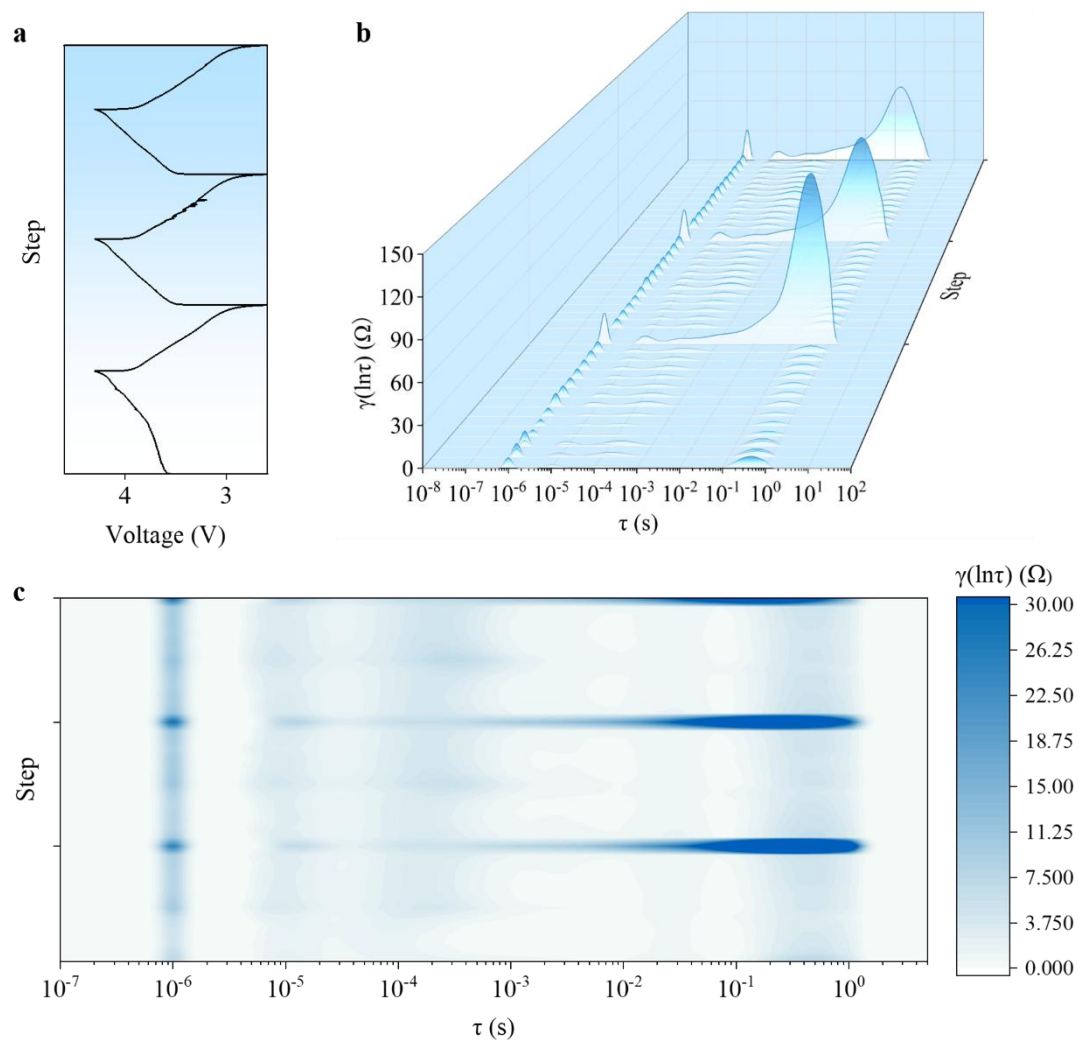


Figure S54. The EIS evolution of the first three cycles from the Si|LSPSC|NMC811 ASSB at 70 mA/g at 25 °C. (a) Time–voltage profiles. (b, c) DRT transformation obtained from the EISs.

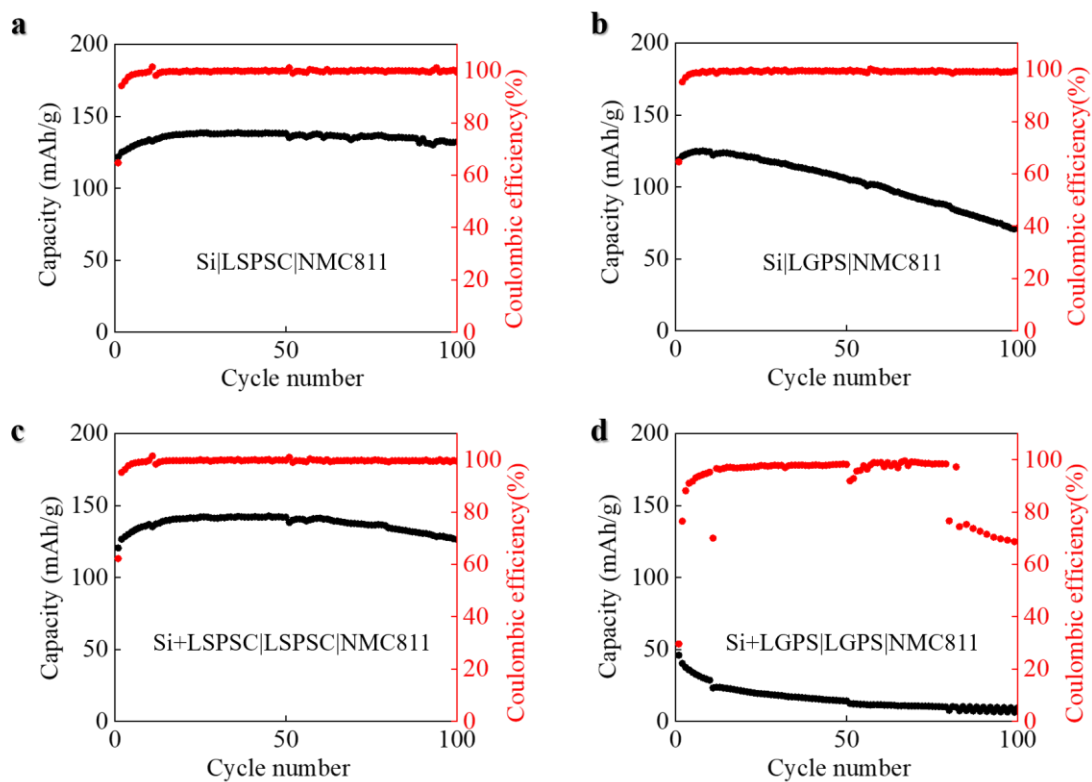


Figure S55. Electrochemical tests of ASSBs assembled with pure Si and Si + sulfide SSE negative electrodes. (a) Si|LSPSC|NMC811. (b) Si|LGPS|NMC811. (c) Si+LSPSC|LSPSC| NMC811. (d) Si+LGPS|LGPS|NMC811.

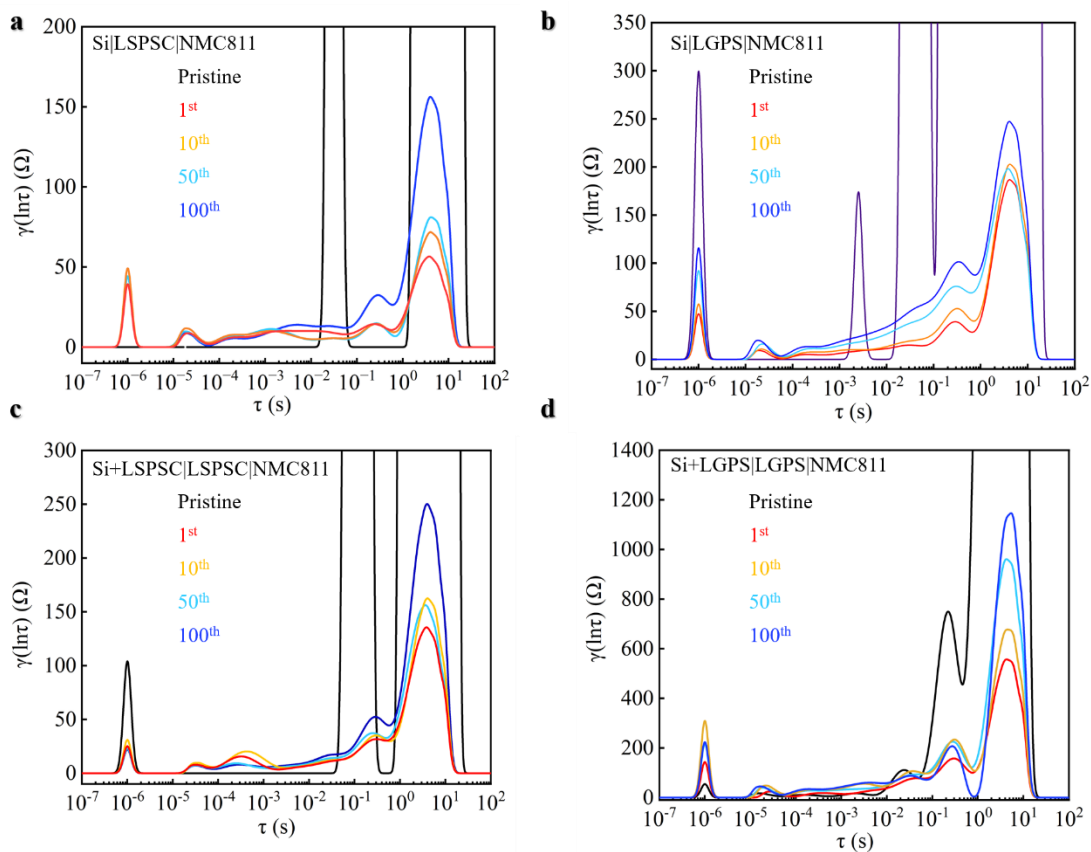


Figure S56. DRT analysis of ASSBs assembled with pure Si and Si mixed with sulfide SSE negative electrodes. (a) Si|LSPSC|NMC811. (b) Si|LGPS|NMC811. (c) Si+LSPSC|LSPSC|NMC811. (d) Si+LGPS|LGPS|NMC811.

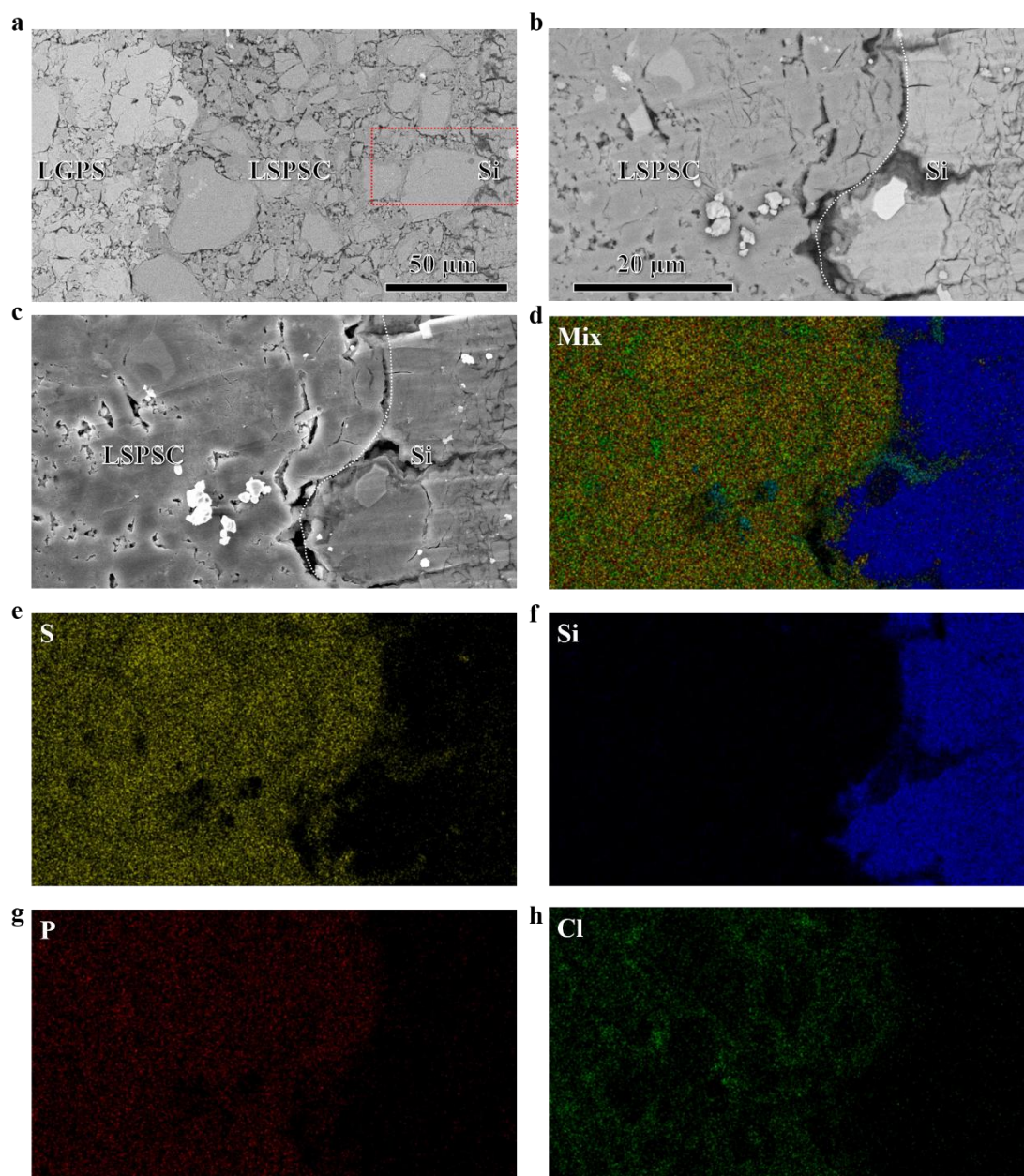


Figure S57. Cryo-SEM Characterization of the Si|LSPSC|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Introducing an LSPSC layer to stabilize the Si/LGPS interface. (b) CBS image and (c) SE mage of the Si/LSPSC interface, and (d-h) the corresponding EDS mapping.

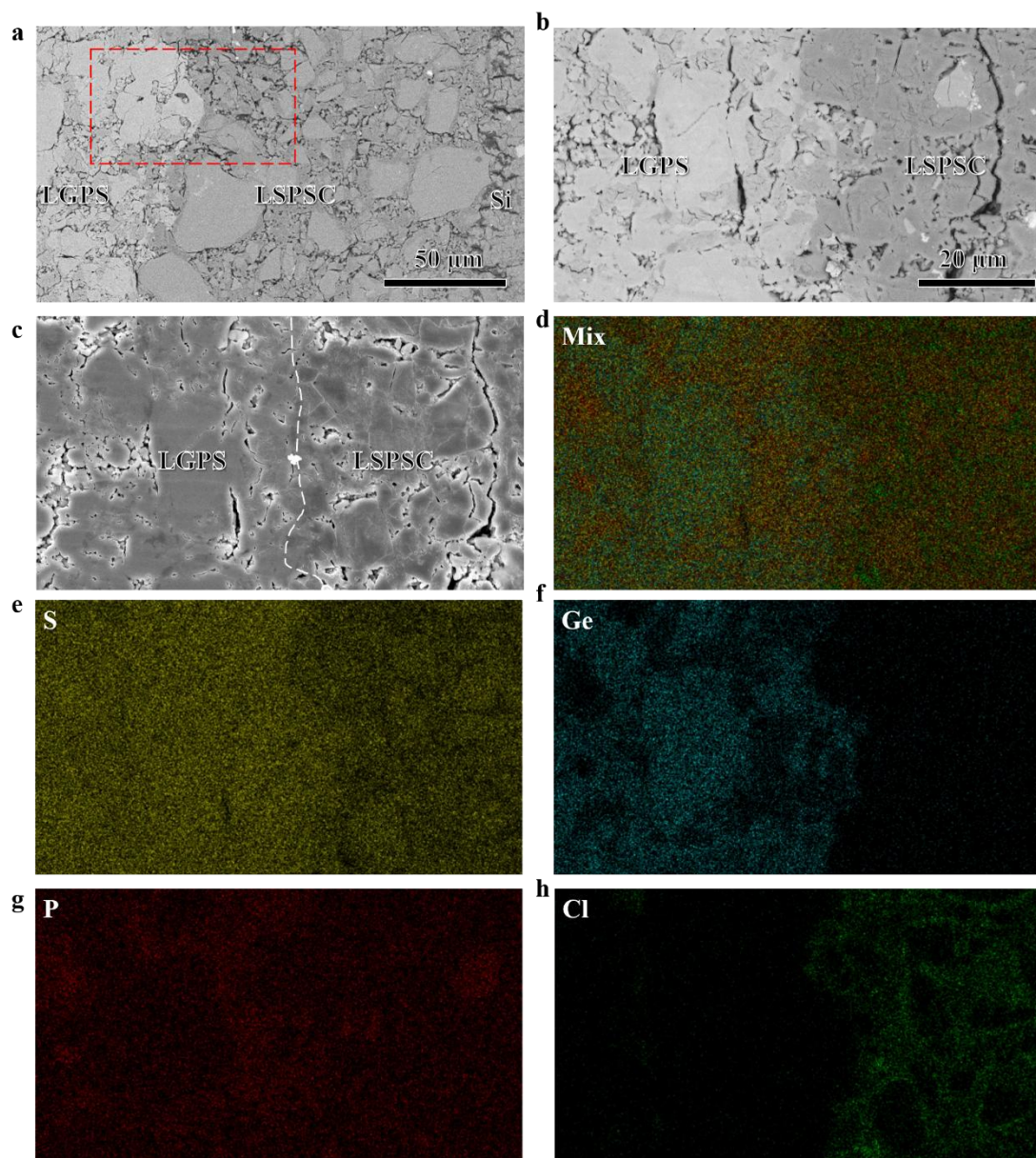


Figure S58. Cryo-SEM Characterization of the Si|LSPSC|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) Introducing an LSPSC layer to stabilize the Si/LGPS interface. (b) CBS image and (c) SE mage of LGPS/LSPSC interface, and (d-h) the corresponding EDS mapping.

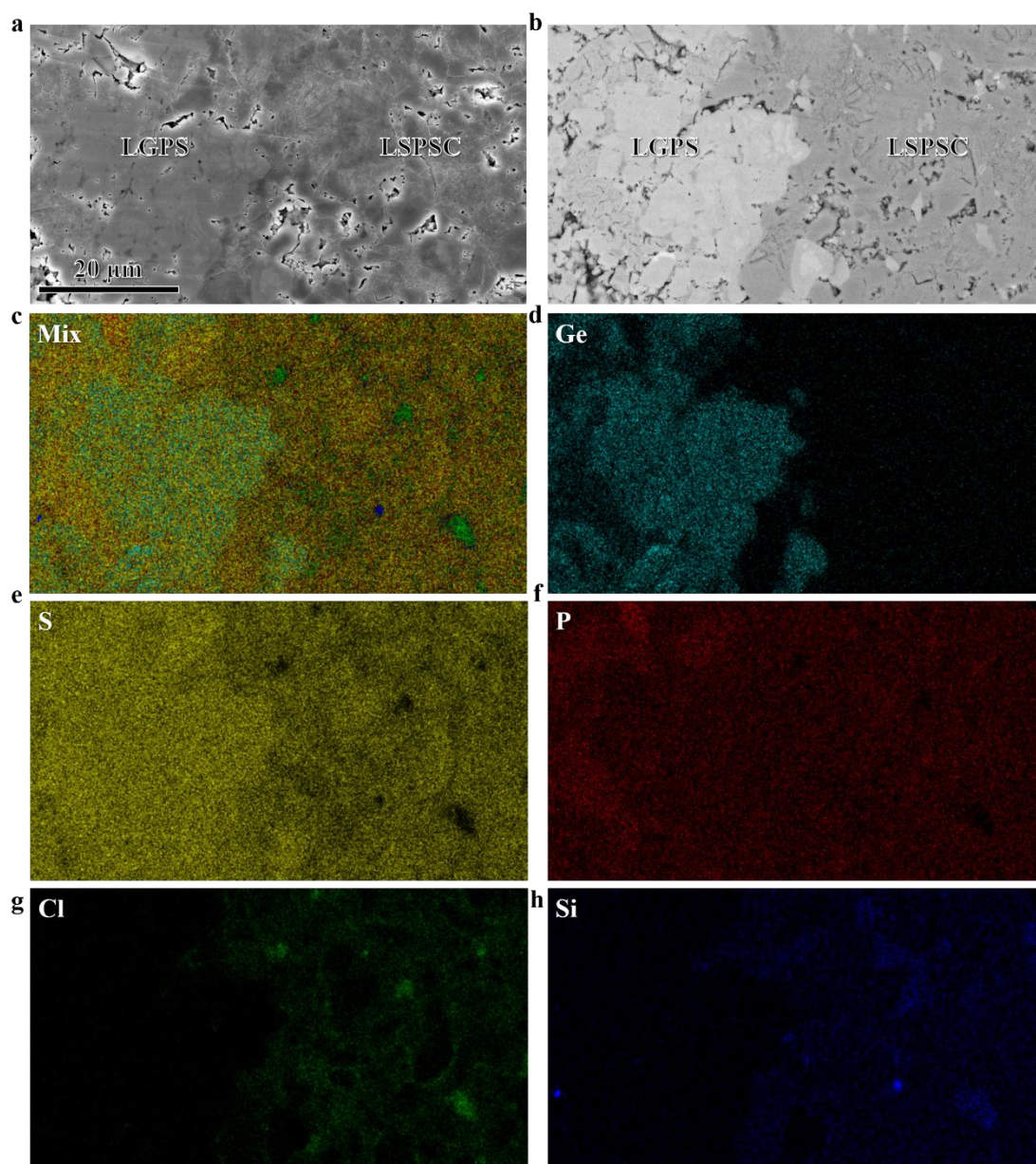


Figure S59. Cryo-SEM Characterization of the Si|LSPSC|LGPS|NMC811 ASSB at fully charged state after 100 cycles. (a) SE image, (b) CBS image of LGPS/LSPSC interface, and (c-h) the corresponding EDS mapping

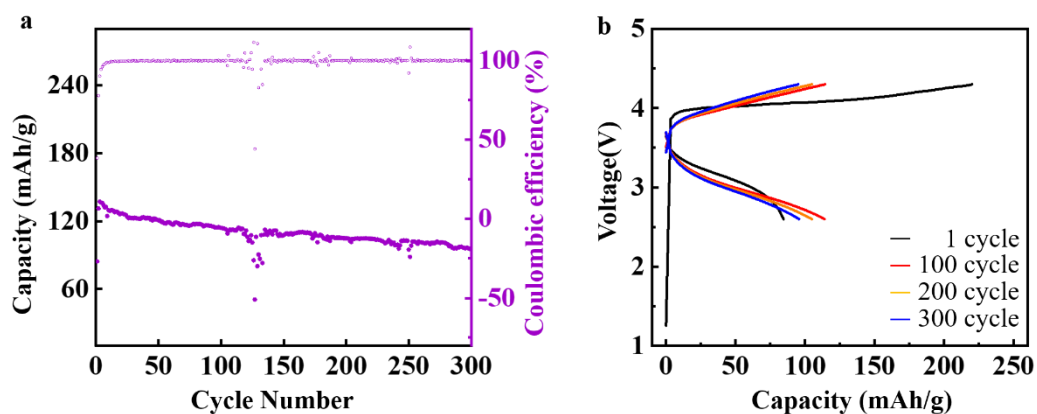


Figure S60. The electrochemical performance of the Si|LSPSC|LGPS|NMC811 ASSB. (a) Cycling performance and coulombic efficiency of the Si|LSPSC|LGPS|NMC811 ASSB cycled at 90 mA/g at 25°C. (b) The voltage profiles of the 1st, 100th, 200th and 300th cycles from the Si|LSPSC|LGPS|NMC811 ASSB.

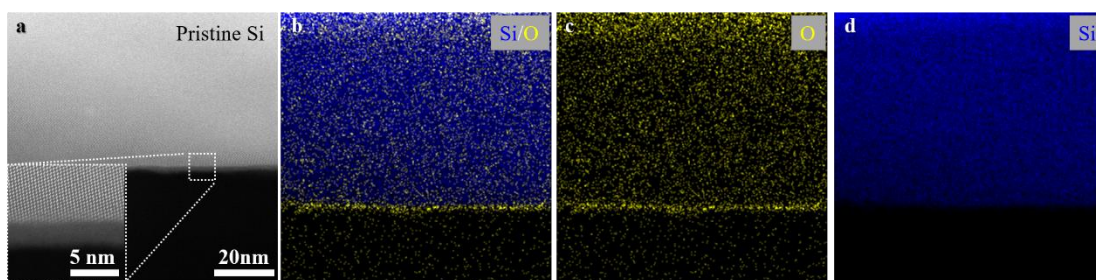


Figure S61. Atomic-level characterization of the oxide layer on the surface of the Si negative electrode. (a) STEM-HAADF imaging. (b) Si/O overlaid EDS mapping. (c) O EDS mapping. (d) Si EDS mapping.

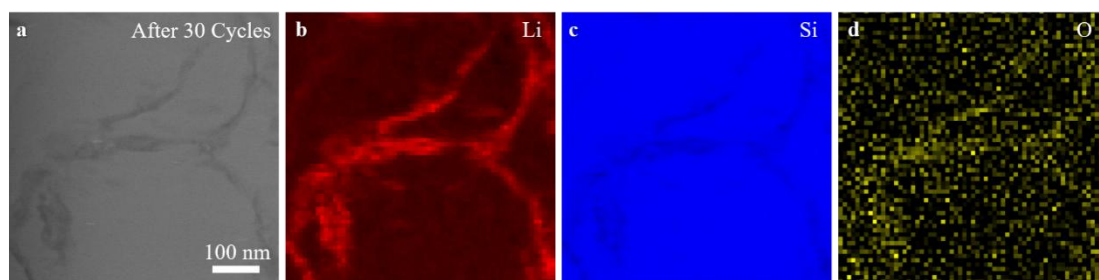


Figure S62. The μ -Si negative electrode of the Si|LGPS|NMC811 ASSB at fully charged state after 30 cycles. (a) Cryo-STEM ADF imaging. (b) Li EELS mapping. (c) Si EELS mapping. (d) O EELS mapping.

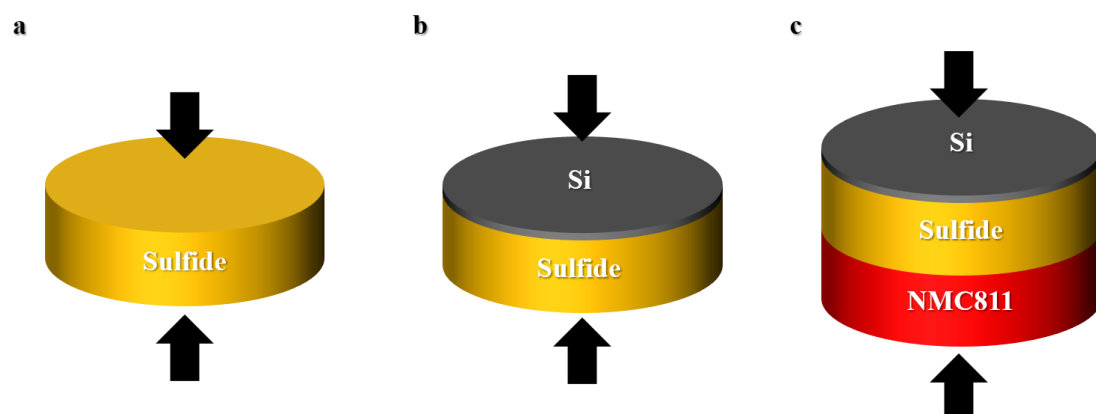


Figure S63. Assembly process of the ASSB. (a-c) Schematic showing the ASSB assembly steps.

Supporting Text

Calculating the interfacial layer thickness.

First, the molar amount of lithium ions in the entire battery can be calculated:

$$Li_{mol} = \frac{Q}{F \cdot n} \quad (1)$$

Where F is Faraday's constant (96485 g/mol·C), Q is the battery capacity, and n is the amount of elementary charge carriers. Next, calculate the amount of substance of lithium-ion loss through coulombic efficiency. Then, using the chemical formulas of the electrolytes $Li_{10}GeP_2S_{12}$ and $Li_{10}Si_{0.3}PS_{6.7}Cl_{1.8}$, assuming sulfur elements completely decompose into Li_2S , obtain the amount of substance of Li_2S generated from lithium-ion loss.

Calculate the mass of Li_2S based on the amount of substance of lithium-ion loss:

$$n_{Li_2S} = \frac{m}{M} \quad (2)$$

Where n_{Li_2S} is the amount of substance of Li_2S , m is the mass of Li_2S , and M is the molar mass of Li_2S .

Finally, determine the thickness h of Li_2S generated from lithium-ion loss:

$$m = V \cdot \rho \quad (3)$$

$$V = S \cdot h \quad (4)$$

where V is volume, ρ is density (1.67 g·cm⁻³), and S is the area of the solid-state battery electrode.