Supporting information
Direct Electrodeposition of Cu_2Sb for Lithium-ion Battery Anodes
James M. Mosby and Amy L. Prieto

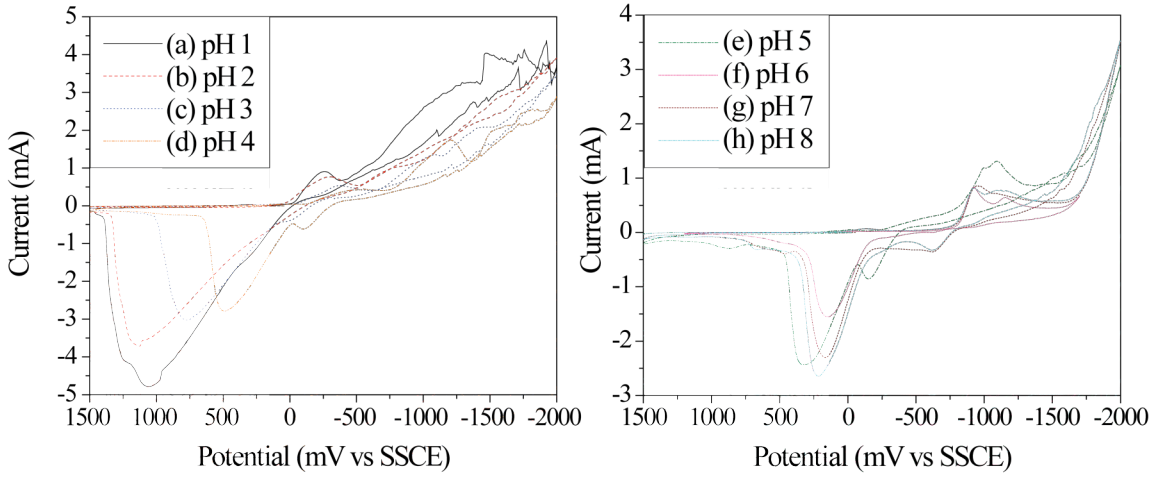

Figure S1: CVs of the deposition solution (0.1 M $Cu(NO_3)_2$ 0.025 M Sb_2O_3 and 0.4 M citric acid) at pH: (a) 1, (b) 2, (c) 3, (d) 4, (e) 5, (f) 6, (g) 7, (h) 8. The reference electrode was a SSCE, the working electrode was Pt, and the scan rate was 500 mV/s.

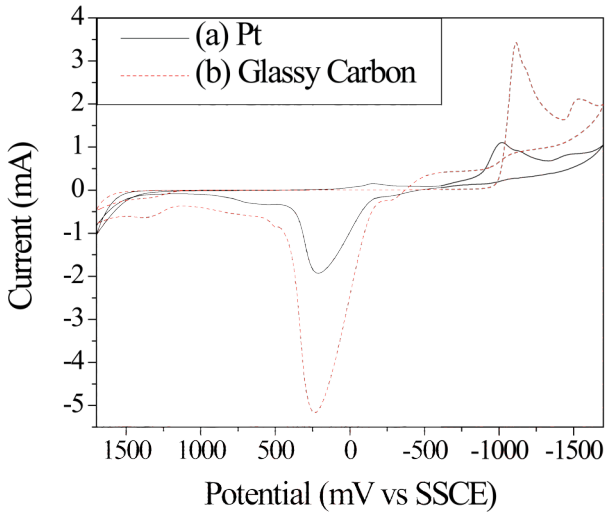

Figure S2: CVs of 0.1 M $Cu(NO_3)_2$ and 0.025 M Sb_2O_3 in 0.4 M citric acid at pH 6. The reference electrode was a SSCE and the scan rate was 500 mV/s.

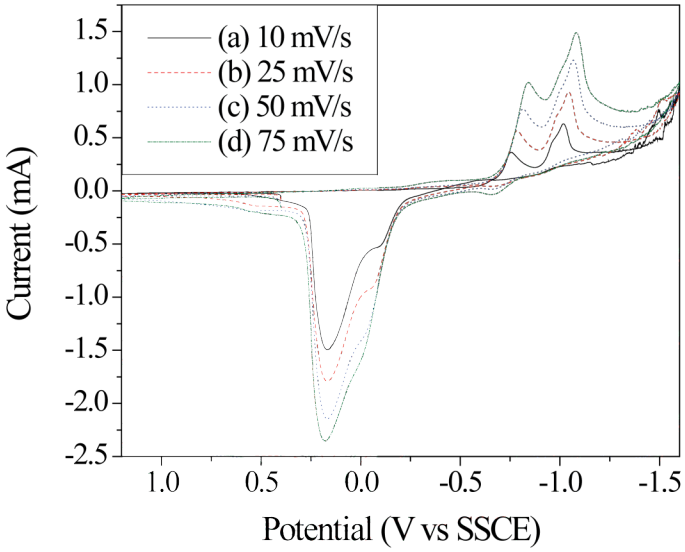

Figure S3: CVs of 0.1 M $Cu(NO_3)_2$ and 0.025 M Sb_2O_3 in 0.4 M citric acid at pH 6 at different scan rates. (a) 10 mV/s, (b) 25 mV/s, (c) 50mV/s and (d) 75 mV/s. The reference electrode was a SSCE and the working electrode was Pt.

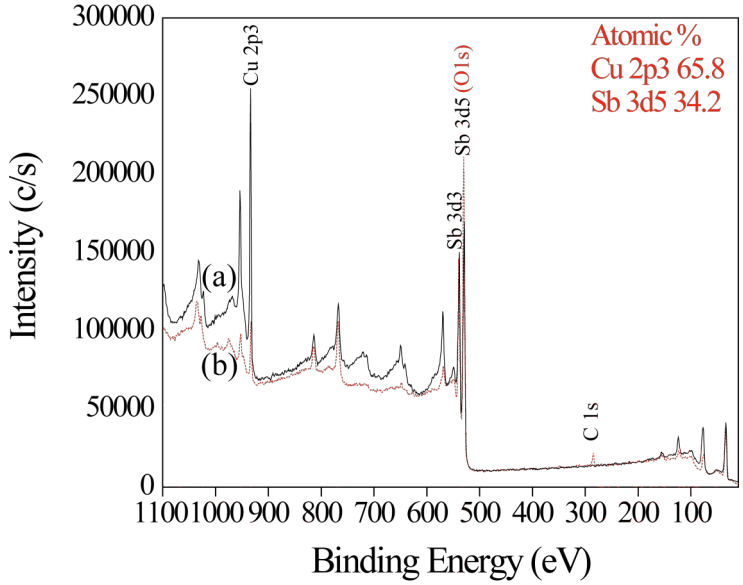

Figure S4: XPS spectrum of a film deposited at -1050 mV. The atomic percentages were taken from a high resolution scan with a P/N ratio of 300 for both the Cu 2p3 and Sb 3d5. (a) Survey scan after sputtering at 2 kV for 30.0 seconds, (b) Survey scan taken before sputtering.

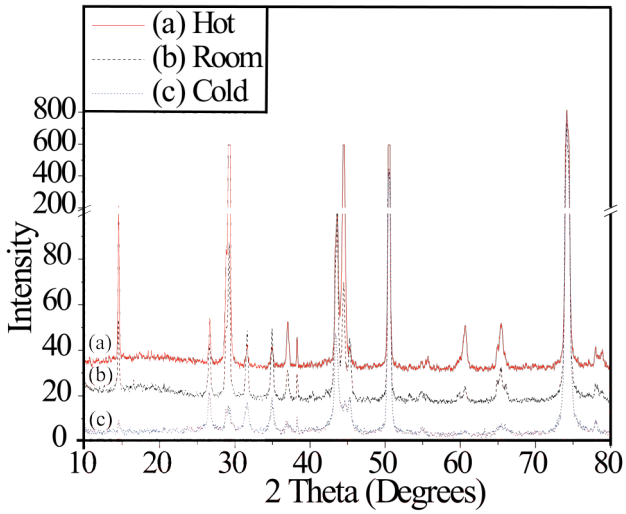

Figure S5: Powder XRD patterns of films deposited at different temperatures from solutions containing 0.1 M $Cu(NO_3)_2$ 0.025 M Sb_2O_3 and 0.4 M citric acid at a pH 6: (a) 60 ºC, (b) 22 ºC and (c) 5 ºC.

Effect of temperature on film crystallinity. We examined the effect of solution temperature on the deposition of Cu_2Sb films by depositing films at 5 ºC, 22 ºC, and 60 ºC. The films for this experiment were deposited at -1050 mV from solutions containing 0.1 M $Cu(NO_3)_2$, 0.025 M Sb_2O_3, and 0.4 M citric acid and at pH 6. The Cu_2Sb film deposited at 5 ºC is the least crystalline as determined by powder XRD, shown in Figure S5c. The SEM image (Figure S6a) indicates that both dendritic grains and spherical grains are present. The Cu_2Sb film deposited at room temperature is more crystalline as determined by XRD peak broadness, and peak positions match data for Cu_2Sb PDF 85-492. The grains present at the surface of this film are spherical as shown by SEM images (Figure S6b). The peak intensities in the XRD pattern of the film deposited at 60 ºC show that the films have strong preferred orientation as indicated by the anomalously large intensity for the (001) planes (Figure S5a). SEM images of this film (Figure S6c and d) show that the surface of the film is composed of stacked cubes. The results obtained from the temperature experiments demonstrated, not surprisingly, that increased solution temperature results in more crystalline films with preferred orientation.

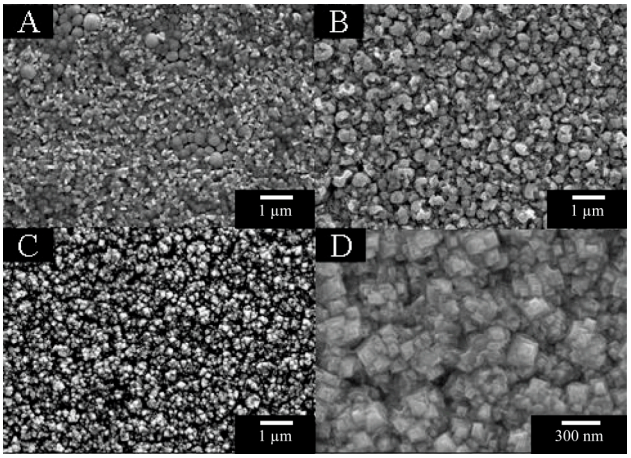

Figure S6: SEM images of the Cu_2Sb films deposited at different temperatures: (a) 5 ºC, (b) 22 ºC, (c and d) 60 ºC.

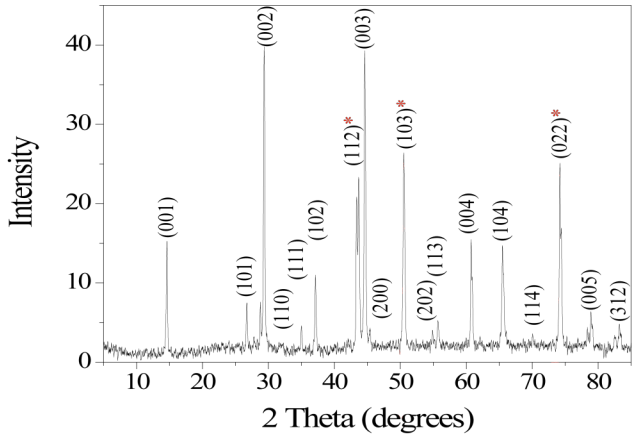

Figure S7: Powder XRD pattern of a Cu_2Sb film deposited using optimized deposition solution concentrations and conditions and annealed for 5 hours at 220 °C under Ar. The asterisks indicate the copper substrate diffraction peaks. The other peaks are indexed to Cu_2Sb PDF 85-492.

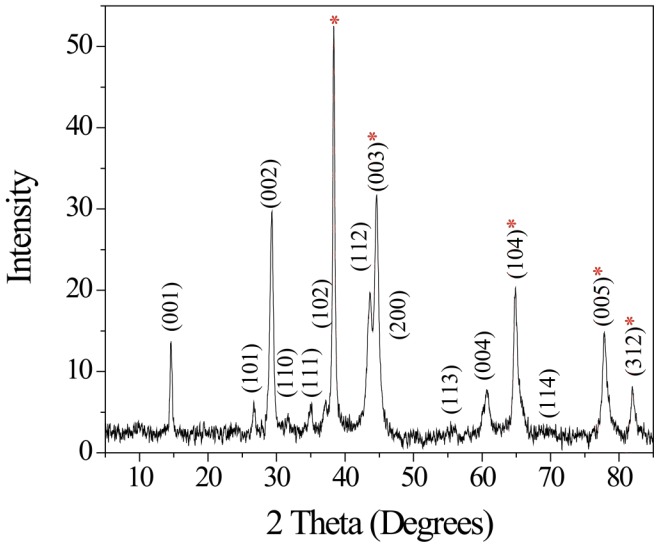

Figure S8: Powder XRD pattern of a Cu_2Sb film deposited on a gold substrate using optimized deposition solution concentrations and potential. The asterisks indicate the gold substrate diffraction peaks, the other peaks are indexed to Cu_2Sb using PDF 85-492.