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4D-STEM Characterization of Low *q* Scattering in Conductive Polymers Used for Li-ion Battery Anodes

Hadas Sternlicht[1], Tianyu Zhu[1], Benjamin Savitzky[1], Colin Ophus[1], Gao Liu[1]* and Andrew M. Minor[1,2]*

[1.] Lawrence Berkeley National Laboratory, Berkeley, California, United States
[2.] Department of Materials Science and Engineering, University of California, Berkeley, California, United States
* Corresponding authors: gliu@lbl.gov, aminor@lbl.gov

Characterization of soft materials, such as polymers, using conventional transmission electron microscopy (TEM) techniques typically applied to harder materials (i.e. metals and ceramics), normally results in beam damage while essentially providing no structural information. Four-dimensional scanning TEM (4D-STEM) captures the complete reciprocal space information while allowing for the practical evaluation of applied dose, addressing the challenges associated with the characterization of soft materials [1].

Conductive polymer binders provide both electronic conductivity and mechanical support for Si-based composite anodes in Li-ion batteries [2,3,4]. They moderate volume changes during electrochemical cycling and provide improved capacity retention. Promising polymer binders include poly(9,9-dioctylfluorene-*co*-fluorenone-*co*-methylbenzoic ester), abbreviated as PFM, and some of its derivatives.

In this work, spin-coated TEM samples of polyfluorene-based polymers were characterized using 4D-STEM in order to address the in-plane structure of the polymer chains at the nanometer length scale at different annealing conditions. The polymer exhibits stronger scattering at lower *q* values as compared to typical crystalline materials. Using 4D-STEM, we could discern between rotationally homogeneous and oriented lobes of low-q scattering, indicating crystallinity associated with π-π stacking between the polymer chains. Example diffraction patterns are shown in Figure 1(a-c).

The orientation of the polymer chain alignment (and associated π-π stacking) was then mapped to reveal the granular morphology (see Fig. 1 (d-e)), where in this context a grain is defined as a region of continuous single orientation of the π-π stacking, which does not change abruptly. The implication of the grain morphology will be discussed in terms of electrochemical behavior and compared to X-ray diffraction and other characterization techniques [5].

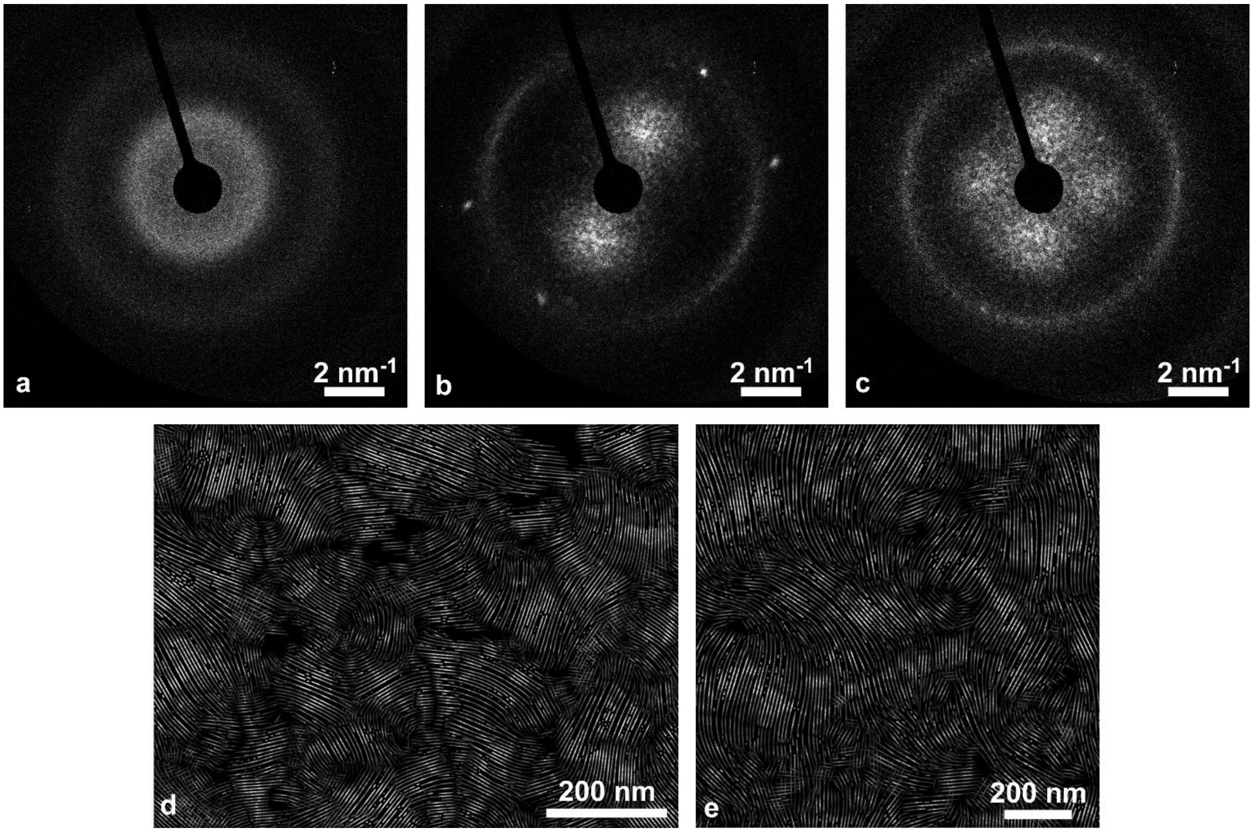

Figure 1. (a)-(c) examples of diffraction patterns from different annealing conditions (weighted average over 3x3 pixels). (d) and (e) flow line maps of the polymer chains corresponding to some of the samples with diffraction patterns shown in (a-c).

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