WHITE PAPER

Is the Impurity in a Battery Cathode Powder Conductive?

Four lithium iron phosphate lots, a few unexplained XRD peaks, and what SEM contrast adds

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Key finding

Backscattered-electron contrast suggests the secondary phase is not an iron phosphide. The phase is tentatively lithium phosphate, an electrical insulator, pending EDX analysis of the particles.

The question

A battery materials company sent four lots of lithium iron phosphate ($LiFePO_4$) cathode powder. XRD showed highly crystalline $LiFePO_4$ and 1 to 3 wt% graphite, which is added on purpose to improve conductivity. It also showed a few weak, unassigned peaks near 34° and 48° 2θ, estimated below 1 wt%. The open questions were what the extra phase was and whether it mattered for cell performance.

Two explanations tested

First, the weak peaks might be background noise. Iron fluoresces under the Cu Kα X-rays used by most diffractometers, which raises the background and can hide small peaks. Narrowing the detector's pulse-height window from 48–80% to 52–80% reduced the background by about 40%. The peaks remained.

Second, the impurity might be an iron phosphide, which conducts electricity and could affect cell performance. In backscattered-electron contrast, phases with a higher average atomic number appear brighter, so an iron phosphide (average atomic number 18.0 for FeP) should stand out against $LiFePO_4$ (11.6). SEM showed no bright particles. It showed a second population of larger, rounded particles (1 to 5 µm) that looked darker than the $LiFePO_4$ crystallites.

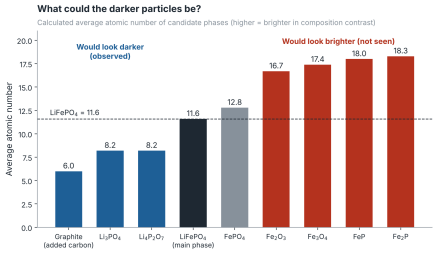


Figure 1. *Calculated average atomic number of candidate phases. Higher values look brighter in composition contrast.*

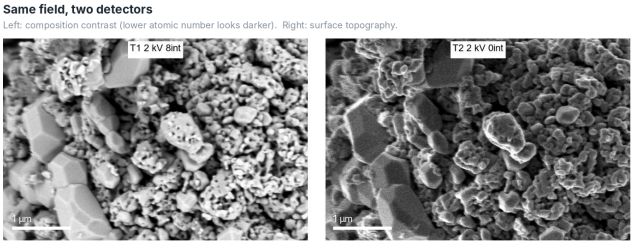


Figure 2. *One field imaged with the composition detector (left) and the topography detector (right), 2 kV.*

What remains

Two candidates with a low average atomic number remain: lithium phosphate (8.2), an electrical insulator, and carbon (6.0). Graphite does not produce a peak near 34°, while lithium phosphate does, so the XRD points to lithium phosphate. The assignment is tentative. I recommended EDX analysis of the rounded particles, which would separate lithium phosphate from iron phosphate. At the estimated level, an insulating phase should have little effect on a cell.

About this work

Cubic Labs LLC analyzes materials by powder XRD, XRF, and SEM-EDX at Purdue University and helium pycnometry in its own lab. Client details are withheld. If you need to know what a lot contains beyond the datasheet, send a sample and your question to info@cubiclabsllc.com or (701) 330-1911. Results stay confidential to you.