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Novel Laser-Based Manufacturing of nano-LiFePO₄ Based Materials
for High Power Li Ion Batteries

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Introduction:

Reducing particle size into the nanoscale is one approach to enhancing the rate capability of known LIB active materials for use in high-power applications like portable power tools and hybrid electric vehicles. In particular, nanoscale LiFePO₄-based materials have exhibited significantly higher rate capability than powders comprising micron-sized particles (1,2). One drawback commonly attributed to deploying nanoscale materials in LIB is the inability to achieve commercially-attractive electrode loadings (2+ mAh/cm²) and densities (porosity < 30 vol%).

NanoGram's unique laser-based process for synthesizing nanomaterials, NanoParticle Manufacturing (NPMTM), has been used to synthesize a wide range of nanoscale materials, (including LIB active materials) with controlled stoichiometry, surface morphology, and size (3,4). The unique battery active materials synthesized by the NPMTM process (branded as *n*PWRTM) are readily dispersible and compressible.

This paper describes the results to date of NanoGram's efforts to refine the structure and properties of *n*PWRTM C-coated LiFePO₄ as well as their suitability for electrode fabrication.

Experimental:

Nano carbon-coated LiFePO₄ materials were synthesized by atomizing a mixed solution of Li salt, Fe(NO₃)₃·9H₂O, and phosphoric acid into an NPMTM reactor. The powder collected from the NPMTM reactor was coated with a proprietary carbon precursor and heat treated at moderate temperatures to yield nanoscale C-coated LiFePO₄.

The structure and physical properties were characterized by a suite of analysis techniques including BET surface area, SEM, FTIR, XRD, and C-content analysis via combustion IR detection. Additionally, the powder tap density and compressibility were evaluated.

The electrochemical properties and performance were evaluated in 2032 coin cells by fabricating the powders into uncompressed electrodes comprising 80 wt% active material, 12 wt% carbon, and 8 wt% PVdF with active loadings from 1 to 2 mAh/cm². Rate capability performance was evaluated by successively discharging the cells at progressively higher C-rates from C/25 up to 5C using the same

charge rate and voltage limits (C/25, 4.0 to 2.0V, respectively) for all cycles.

Results:

Powders have been synthesized with high phase purity (via XRD and FTIR analysis) and BET surface areas of 12 m²/g and higher. The amount of carbon is typically less than 3 wt% while tap densities are between 1.0 and 1.4 g/cc. An evaluation of powder compressibility showed that the *n*PWRTM C-coated LiFePO₄ can be formed into pellets with > 60 % theoretical density which is a level similar to the active material in a highly-loaded, commercial electrode.

Fig. 1 compares the rate capability of two *n*PWRTM C-coated LiFePO₄ samples with a powder synthesized by conventional methods. The *n*PWRTM C-coated LiFePO₄ exhibits better rate capability despite 50% greater electrode active loading. Note that 125 mAh/g was delivered capacity at 3C.

This work shows the potential for NanoGram's NPMTM process to synthesize low-cost nanoscale C-coated LiFePO₄ that are suitable for fabricating electrodes suitable for high-rate LIB. .

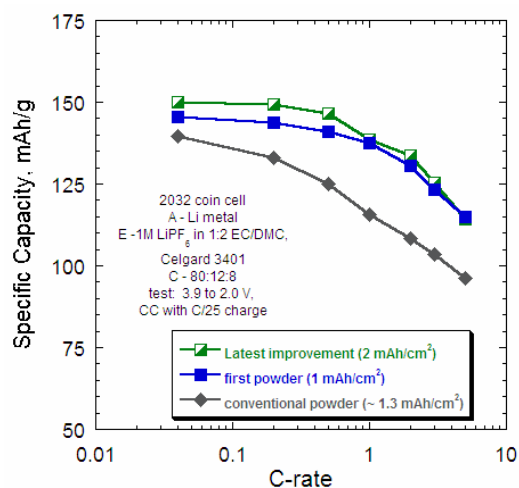


Figure 1. Modified Peukert plot comparing the rate capability of two *n*PWRTM C-coated LiFePO₄ samples with conventionally-synthesized material.

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