Application note | AN41506

X-ray diffraction

Determining the degree of graphitization for battery anodes using ARL X'TRA Companion XRD

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Introduction

Accurate characterization of the degree of graphitization in carbon-based battery anodes is essential for optimizing their electrochemical performance and energy storage capabilities. X-ray diffraction (XRD) analysis has emerged as a valuable technique for precisely assessing the graphitization degree. By determining the location of the (002) reflection, the graphitization degree can be quantitatively evaluated. This quantity must be >90%, but the optimal value depends on the cathode as well as the anode, and therefore the value of graphitization needs to be monitored for each specific type of battery as part of regular quality control by operators. Such analysis is ideally automated using Thermo Scientific™ SolstiX™ Pronto Software which streamlines the analysis process and provides rapid and reliable results. Additionally, adherence to the Chinese norm GB/T 24522-2019 ensures compliance with industry standards. The insights gained from XRD analysis, combined with automated analysis capabilities, enable researchers and manufacturers to optimize the analytical workflow and ensure expected battery anode performance.

Instrument and software

The Thermo Scientific™ ARL™ X'TRA Companion (Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the state-of-the-art solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System).

Figure 1: ARL X'TRA Companion diffraction system.

Experimental

Graphite anode material was mixed with Silicon as an internal standard (2:1 ratio) and measured in reflection mode using Cu Kα (1.541874 Å) radiation for 10 minutes. Using a certified internal standard yields more precise absolute peak positions in refinements. The sample was prepared in a zero-background sample holder to minimize penetration depth error, and acquisition was performed with spinning sample. According to GB/T 24522-2019 GB norm[1] and [2], the degree graphitization is calculated as:

$$g=(3.4400-d_{002})/(3.4400-3.3540)$$

Results

Analyzing the position of the (002) reflection (see Figure 2) of graphite in an automated Rietveld refinement yields a degree of graphitization of 96.396% and a d_{002}=3.3571Å. The goal is to optimize cathode/anode interaction by finding the most suitable g value, which is generally >90%.

Conclusion

The ARL X'TRA Companion is perfectly suited to determine the degree of graphitization for battery anode material. Results can be obtained in a one-click analysis in accordance with GB/T 24522-2019 norm.

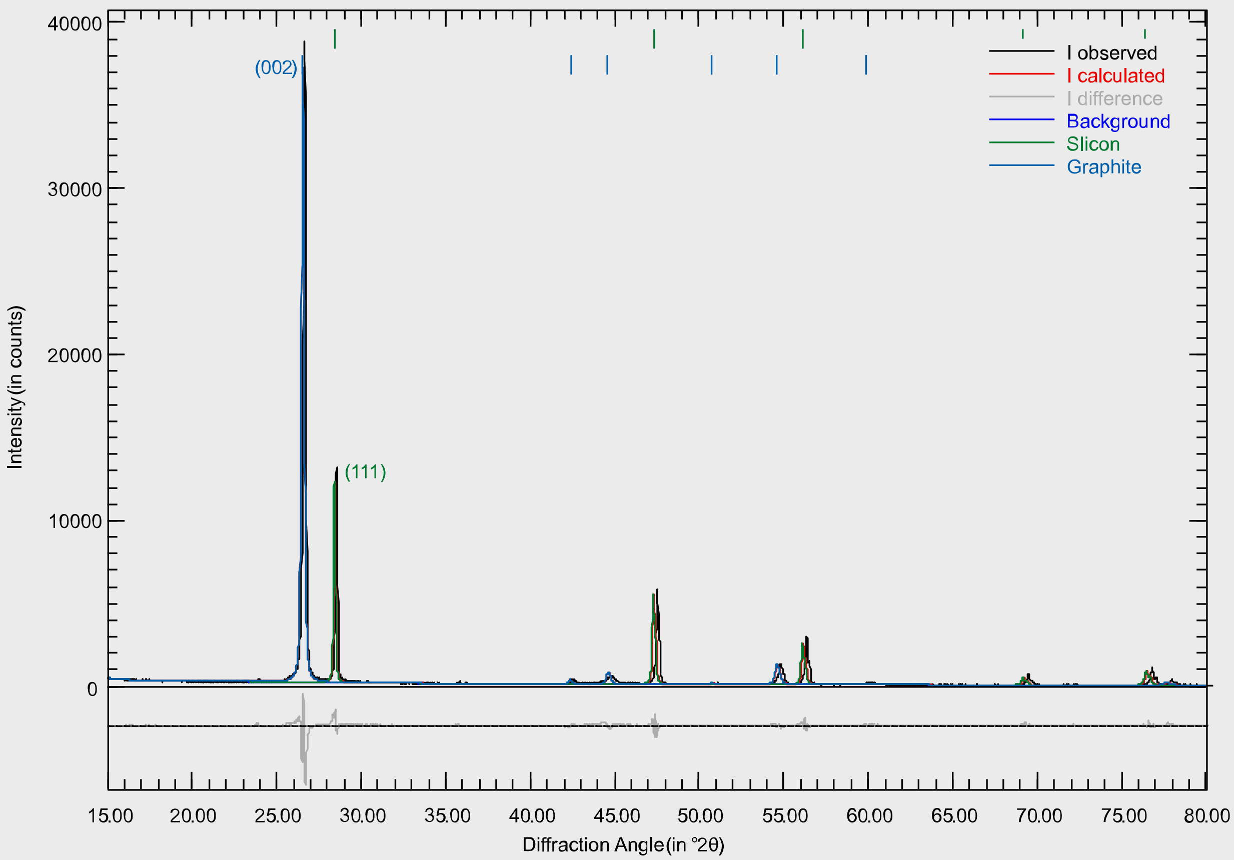

Figure 2: Measurement (10 minutes) of a graphite anode material sample (silicon added as internal standard).

References

1. GB/T 24533-2019 Graphite negative electrode materials for lithium-ion battery (English Version)
2. C.N. Barnakov, G.P. Khokhlova, A.N. Popova, S.A: Sozinov, Z.R. Ismagilov, ECTJ **2015**, 17, 87-93.

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