

In-Situ XRD Coin Cell: Setup, Alignment, and Data Quality

A practical guide to using the XRD in-situ coin cell: how the Kapton window works, single-window vs dual-window configurations, alignment on the diffractometer, and how to get clean diffraction data during electrochemical cycling.

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What Is In-Situ XRD and Why Does It Matter?

Conventional XRD gives you a snapshot of your material's crystal structure at one state of charge. You assemble a cell, charge or discharge it to a target voltage, disassemble it, extract the electrode, and measure. The problem is that this ex-situ approach introduces artifacts at every step: the electrode oxidizes during disassembly, the structure may relax when current stops flowing, and sample preparation can mechanically disturb the electrode.

In-situ (or operando) XRD solves this by measuring diffraction patterns while the cell is actually cycling. You see the crystal structure evolving in real time—phase transitions, lattice parameter changes, peak broadening from disorder—as a continuous function of state of charge. For materials that undergo reversible phase transitions (LiFePO₄ between FePO₄ and LiFePO₄, for example, or layered oxides like NMC during cycling), the in-situ data tells you things that a series of ex-situ snapshots cannot.

The in-situ XRD coin cell makes this measurement accessible using equipment most battery research labs already have: a standard coin cell crimper and a laboratory X-ray diffractometer.

How the Cell Works

The cell is a modified coin cell housing with a hole machined through one or both faces. The hole is sealed with a thin film of **polyimide (Kapton)**—a material chosen specifically for its low X-ray attenuation.

When the X-ray beam passes through the Kapton window, it enters the cell and diffracts from the electrode material inside. The diffracted signal exits through the same window (reflection mode) or the opposite window (transmission mode) and reaches the detector.

The cell is assembled exactly like a standard CR2032 coin cell: electrode stack, separator, electrolyte, and spring inside a 20 mm diameter housing. The only difference is the window. The electrochemical performance is essentially identical to a sealed coin cell—the Kapton film adds negligible internal volume and does not contact the electrodes or electrolyte directly.

Single-Window vs Dual-Window: Which Do You Need?

Cathode-Side Window (Single Window, XRD-POS-1)

The window is on the positive case (cathode side). This is the configuration for **reflection mode XRD**, where the incident beam and detector are on the same side of the sample—the standard geometry for most laboratory diffractometers (Bragg-Brentano geometry).

Use this if:

- Your diffractometer uses Bragg-Brentano geometry (most powder diffractometers do)
- You are studying the **cathode material** (the positive electrode faces the window)
- You want the simpler, lower-cost option

Dual-Side Window (XRD-DUAL-1)

Both the positive and negative cases have windows. This configuration supports:

- **Transmission mode XRD**: the beam passes straight through the cell, enabling measurements at lower angles and better signal from thin electrode films
- Simultaneous access to both electrodes (by alternating the beam direction)
- Synchrotron measurements where transmission geometry is standard

Use this if:

- Your measurement requires transmission geometry
- You need to study both electrodes without disassembling
- You are working at a synchrotron facility

For most university laboratory diffractometers in standard Bragg-Brentano configuration studying cathode materials, **the single-window version is the correct and more economical choice**.

Kapton Film: Properties Relevant to XRD

Polyimide (Kapton) is the standard window material for in-situ electrochemical XRD cells across the research literature. Its key properties:

X-ray transmission: For Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$, the most common laboratory source), a 25 μm Kapton film transmits approximately 90–92% of incident intensity. The absorption edge of carbon and nitrogen (the main components of polyimide) is below the Cu-K α energy, so attenuation is minimal in the 10–80° 2 θ range most relevant to battery materials.

Kapton fluorescence: Kapton does not produce fluorescence peaks with Cu-K α radiation. This is important—some window materials produce their own diffraction or fluorescence features that overlap with electrode diffraction peaks.

Chemical stability: Kapton is stable in contact with standard carbonate-based and ether-based electrolytes. Do not use strongly oxidizing electrolytes (e.g., concentrated H₂SO₄-based) without first verifying

compatibility.

Mechanical integrity: The window is sealed around its circumference during cell fabrication. The window will not hold pressure—do not attempt to purge or pressurize the cell interior.

Cell Assembly

Assemble the in-situ cell exactly as you would assemble a standard CR2032 cell. The key difference in procedure is ensuring the electrode is centered on the window.

Electrode Positioning

The diffraction measurement averages the structure over the beam footprint on the electrode. For quantitative data, the electrode must:

1. Cover the entire window area (typical window diameter: 4–6 mm, depending on the cell design)
2. Be centered on the window
3. Be flat and uniformly loaded—thick or lumpy electrodes create path length variation across the beam

For slurry-coated electrodes on flat current collectors, use a punch to cut circular electrodes slightly larger than the window diameter. For free-standing or powder-pressed electrodes, verify that the active material is uniformly distributed across the window area before assembly.

Electrolyte Volume

Use the same electrolyte volume as for a standard coin cell (60–80 μL). Excess electrolyte increases the effective path length through liquid for the beam and can increase background scatter.

Crimping

Crimp at the same setting as for a standard CR2032. The window does not affect the mechanical crimping process. After crimping, inspect the window visually: it should appear flat and undeformed. If the window is wrinkled or dented, discard the cell—the window geometry must be flat for consistent beam path.

Mounting on the Diffractometer

Flat Plate Sample Stage

The in-situ cell sits on the flat plate sample stage of the diffractometer, replacing the powder sample. The cell must be positioned so that the electrode surface (inside the cell, behind the Kapton window) is coplanar with the diffractometer rotation axis.

Most flat plate stages have an adjustable height. The correct height is the same as for a powder sample mounted on a zero-background holder—verify by checking that the primary beam illuminates the center of the window at the lowest 2θ angle you will measure.

Electrical Connections

The positive terminal (cap) and negative terminal (can) need electrical connections to your potentiostat or battery cycler. Use spring-contact leads or alligator clips to connect without applying mechanical pressure that could deform the cell.

Route the electrical cables so they do not block the beam path or detector at any point in the 2θ scan range. For automated scans, verify the cable routing at the maximum 2θ angle you plan to measure.

Beam Size and Window Coverage

Adjust the incident beam size to fit within the window. A beam footprint larger than the window area will illuminate the metal cell housing, producing strong steel diffraction peaks (primarily at 43.6° and 50.8° 2θ for austenitic stainless steel) that contaminate the pattern. For most laboratory diffractometers, a 1–2 mm $\times$ 5–10 mm beam footprint fits comfortably within a 4–6 mm window.

Measurement Strategy

Electrochemical Protocol

Cycle the cell from your electrochemical workstation using a separate cable set from the diffractometer connections. Use galvanostatic cycling at a slow C-rate (C/20 to C/50 is typical for in-situ experiments) so the structure has time to equilibrate between diffraction scans.

Synchronize the electrochemical data with the XRD timing: most modern diffractometers can output a timestamp or TTL trigger at the start of each scan. Log the cell voltage and current with sufficient time resolution (1 reading per second is adequate for C/20 cycling) to match each diffraction pattern with its state of charge.

Scan Parameters

For most cathode materials, the informative 2θ range is $10\text{--}70^\circ$ (Cu-K α). A single scan at 0.02° step size takes approximately 30–60 minutes depending on count time. For fast structural changes (phase transitions that occur over a narrow voltage window), use faster scans with lower angular resolution.

For materials where the key peaks are at high angles (lattice parameter changes in layered oxides, for example), extend the scan to 80° or higher.

Interpreting the Data

Peak Shifts

A continuous shift in peak position during cycling indicates a **solid-solution mechanism**: lithium is inserting into or extracting from the lattice continuously, changing the lattice parameter without a phase transition.

New Peak Appearance / Disappearance

Abrupt appearance or disappearance of peaks indicates a **two-phase reaction**: the material transforms between two distinct crystal structures. The ratio of the two phase intensities changes with state of charge, but the peak positions remain constant during the transformation.

Peak Broadening

Increasing peak width (FWHM) during cycling indicates growing structural disorder—crystallite size reduction, microstrain accumulation, or stacking faults. This is commonly observed in layered oxide cathodes at high state of charge or after many cycles.

Background and Artifacts

Common sources of artifact peaks in in-situ cell patterns:

- **Stainless steel peaks** at 43.6° and 50.8° (from the cell housing if beam overfills the window)
- **Kapton halo** at ~15–25° (broad amorphous peak from the polyimide film—constant across all patterns, easy to identify)
- **Electrolyte peaks** at low 2θ if the electrolyte partially crystallizes (rare at room temperature)

The steel and Kapton peaks appear in every pattern at constant position and intensity. Subtract them or simply exclude those 2θ ranges from your analysis.

Handling the Window

The Kapton window is the most fragile part of the cell. Handle with care:

- Do not touch the window surface
- Do not wipe the window—any abrasion creates surface damage that scatters the beam
- Do not store assembled cells under mechanical pressure
- If dust lands on the window, remove it with a gentle nitrogen blow, not contact cleaning

Each cell ships in a bubble-wrapped sealed bag. Inspect the window before assembly by holding the cell up to a light source—the window should be uniformly translucent without pinholes or wrinkles.

Related Products

- [CR2032 Coin Cell Kit](#) — standard coin cell for ex-situ XRD reference samples
- [Nickel Foam](#) — electrode substrate for electrodeposited or hydrothermally grown active materials
- [18650 Battery Case](#) — cylindrical cell format for full-cell performance validation

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