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Perspective

Standardizing Operando Diffraction Studies for Battery Systems

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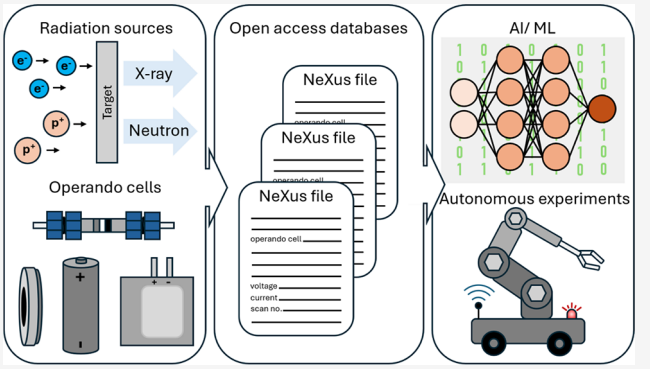
ABSTRACT: Operando diffraction experiments enable real-time, nondestructive correlation of the electrochemical behavior of battery cells with the physicochemical changes occurring in their internal components. These experiments can now be directly performed on standard cell formats, identical to those used for electrochemical testing, at unprecedented scattering, temporal, and spatial resolutions. This can generate significant volumes of new multimodal datasets, opening exciting possibilities for probing and parameterizing complex battery performance and degradation mechanisms. To fully leverage the wealth of data produced by such experiments via data-driven machine learning methods, the open sharing of data *via* standardized, machine-readable formats, in line with FAIR principles, is essential. Currently, this is hindered by differences in the cell design, experimental protocols, and data-processing workflows. In this perspective, we discuss the need for standardizing operando battery diffraction experiments, the associated challenges, and propose a NeXus-based application definition for the standardized exchange of experimental metadata and results as a pathway toward reproducible research practices and transparent analytical workflows.



The electrochemical performance degradation of battery systems is fundamentally linked to the changes in material and chemical properties that occur within their internal components during operation.[1,2] A comprehensive characterization of battery cell degradation is a multifaceted challenge: cycling-induced chemical transformations occur across multiple components (particle–electrode–cell) and spatiotemporal scales (10^{-10}–10^{-2} m, 1 cycle – 100 s of cycles). They are influenced by multiple external parameters including cell design, form factor, and operating conditions.[3,4] In many cases, the individual degradation mechanisms may interact with each other, leading to cascading effects. Consequently, a comprehensive mechanistic understanding of electrochemical degradation in battery systems requires a holistic, multi-faceted approach that integrates electroanalytical methods with complementary material characterization techniques.

X-ray and neutron powder diffraction are among the most widely used techniques for the diagnostic characterization of battery cells,[6,7] which may be performed ex situ, in situ or operando. An ex situ measurement involves the characterization of the component of interest after it is removed from the cell. In situ and operando experiments involve direct characterization of the cell – the former involves measurement of the cell under *quasi*-equilibrium steady-state conditions, whereas in the latter data are continuously collected as the cell is cycled, enabling real-time monitoring of the crystallographic and chemical evolution of a cell's internal components (Figure 1). Therefore, for operando experiments, it is vital that the system under investigation is as close as reasonably possible to its actual operating condition to obtain truly meaningful insights.

Over the past two decades, advances in scientific instrumentation have greatly expanded the possibilities for such studies, enabling the rapid generation of electrochemical–diffraction datasets of unprecedented resolution, volume, and complexity.[8,9] In parallel, developments in data science, driven in part by advances in machine learning (ML) and artificial intelligence (AI), offer powerful pathways for accelerating experimentation, data processing and extracting deeper insights from such high-dimensional datasets,[10] beyond conventional methods such as Rietveld analysis.[11,12] However, to fully capitalize on these advancements and translate the outcomes of such complex experiments into practical guidance for designing better battery systems, it is vital that these experiments are "reliable, representative and reproducible".[13] Equally important is the open and transparent sharing of data, together with experimental

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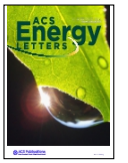

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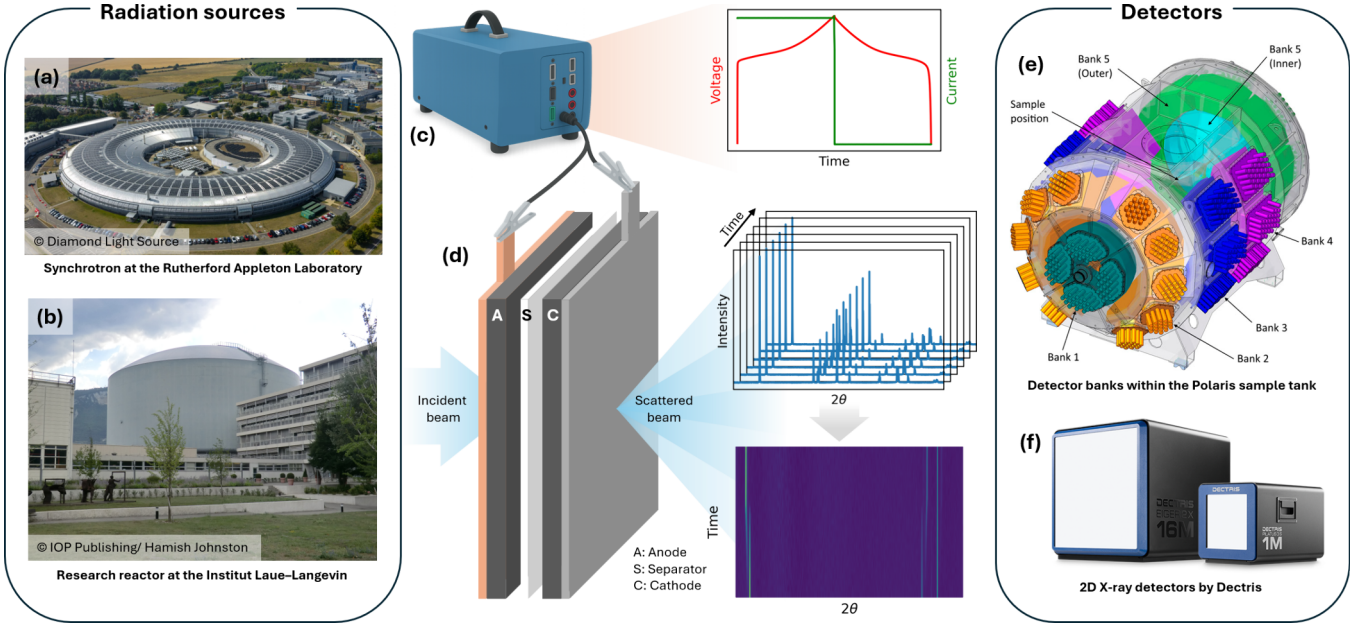

Figure 1. Schematic of an operando diffraction experiment, which typically consists of a radiation source (a, b), an electrochemical cell (d), a potentiostat (c), and a detector (e, f). (a) Synchrotron: Reused with permission from Diamond Light Source. Diamond Light Source. (b) Research reactor: Photo originally published on Physics World website September 2011. IOP Publishing/Hamish Johnston. Reused with permission. All rights reserved. (e) Neutron detectors: The multi-bank detector setup of the Polaris time-of-flight diffractometer.[5] Reused under the terms of the Creative Commons CC BY licence. (f) 2D X-ray detectors: Reproduced with permission from Dectris.

metadata, in machine-readable formats that comply with the FAIR (Findability, Accessibility, Interoperability, and Reusability) data principles.[14] Such sharing is critical for data-driven methods, as these often rely on curated and well-labelled training datasets. Moreover, the open, transparent sharing of data from diverse sources, e.g., the Battery Imaging Library,[15] can also help mitigate model bias and broaden the parameter space explored by the model. Achieving this requires the measurement protocols, data-acquisition methodologies, and analysis workflows to be standardized.[16]

Unlike electrochemical battery data, the need to standardize material characterization data, especially diffraction, has long been recognized. For example, the crystallographic information file (CIF) is a widely accepted community standard for sharing crystallographic data.[17] However, such formats generally do not include detailed experimental metadata by default, making it difficult to verify the provenance and quality of the data. This becomes increasingly important for measurements at synchrotron X-ray, neutron and muon research facilities, where the instrumental setups and data processing workflows are more complex than at laboratory instruments. This was addressed in part with the introduction of the NeXus file format,[18] which is now the *de facto* standard for characterization data exchange and archival at central research facilities. It is built on the HDF5 (hierarchical data format)[19] scientific data format and includes domain-specific conventions for organizing technique-related information and structured storage of raw and processed data. Although the NeXus format offers a possible pathway to standardize operando battery diffraction measurements, the variability of electrochemical cells, measurement protocols, and analysis methodologies complicates a straightforward implementation, necessitating the development of new, dedicated experimental application definitions.

In this perspective, we outline the need for standardizing operando battery diffraction research and discuss the associated challenges. Given that operando battery diffraction data are often interpreted alongside electrochemical data, it is also crucial that electrochemical testing is performed in a standardized manner. To this end, we first address issues related to electrochemical cell testing, including cell configurations and formats, data analysis and reporting practices. We then extend this discussion to operando diffraction studies of battery cells, highlighting specific challenges related to sample environments (cell formats), experimental methodologies, and data-processing workflows. Finally, we propose a customized NeXus application definition aimed at standardizing operando X-ray and neutron diffraction studies of battery systems. Looking ahead, the adoption of autonomous experimentation may necessitate further extension of such standardized file format, e.g., inclusion of metadata on how experimental decisions are made, particularly for model training purposes.

■ STANDARDIZING BATTERY CHARACTERIZATION RESEARCH

Battery cells are multi-component multi-phase systems, whose real-world utility depends on several performance criteria being met at once.[20] Some of these requirements, e.g., raw material cost, synthetic scalability and safety, are often, perhaps understandably, overlooked in academic research, which is why academic breakthroughs often fail to translate to the industrial scale.[20] Consequently, the translation of academic battery research into industrial gains has been slower and more incremental than the volume of published work might suggest. Progress is also hindered by the lack of standardization, wherein new developments are often evaluated using arbitrary metrics,

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making it difficult to distinguish genuine breakthroughs from artifacts of testing methodologies.[16] Encouragingly, initiatives have begun to introduce standardized FAIR-compliant data standards in the battery research field, including activities within the *Battery Data Genome* and *BIG-MAP* projects.[21,22] A noteworthy example is *BattINFO*,[23] aimed at creating a formalized ontological description of battery systems to support interoperability and analytical workflows. There are also efforts to standardize sharing of electrochemical data via standardized formats such as *Voltaiq Data Format* and the *Battery Information Format*.[24,25]

In battery research, electrochemical testing is an integral part of diagnostic characterization. However, there is no consensus on the experimental test protocol and data reporting, making it difficult, if not impossible, to compare data between different (material) systems or studies.[26] At the heart of the challenge to standardize the electrochemical characterization is the wide variety of electrochemical configurations (half-cells, full cells) and cell form factors (coin, pouch and cylindrical). Due to the inherent limitations associated with each variant, it is sometimes difficult to draw comparisons between studies. Furthermore, the suitability of each cell format varies depending on the characterization technique employed. For example, coin cells are ill-suited for operando diffraction measurements due to their steel casing. In lithium-ion battery research, half-cells are commonly used to study a working electrode relative to a lithium metal reference, which also acts as the counter electrode as it is thought to offer a stable voltage reference, and a near-infinite lithium inventory relative to the cell capacity. However, lithium metal can react with the electrolyte leading to its consumption[27] and the formation of a solid electrolyte interphase, which can contribute to significant polarization of the reference electrode.[28] The cross-over of electrolyte degradation products to the working electrode can also occur, forming an impeding cathode electrolyte interphase.[29] Taken together, these effects limit the use of lithium half-cells to short-term cycling tests. Full cells (vs graphite, $Li_4Ti_5O_{12}$ *etc.*) are often a better option, as they offer improved cycling stability and testing conditions that resemble practical operation. While this is generally true, it is important to be aware of the intricacies of full cells to avoid misinterpreting the results and appreciate the scope of the conclusions. For instance, because of the gradual consumption of lithium inventory in a full cell due to solid electrolyte interphase growth, the average electrode potential of graphite can shift to higher values (a manifestation of electrode slippage). As the cycling of full cells is typically controlled by the cell voltage, the gradual shift in the anode potential can cause the cathode to be exposed to higher electrode potentials that can trigger cathode degradation.[30] The negative-to-positive electrode capacity ratio (N/P ratio)[31] and the anode overhang[32] have also been shown to impact the cycling performance of full cells, which can further complicate comparison. Depending on the research objectives, other setups such as symmetrical cells have also been suggested.[33] To facilitate standardization of electrochemical configurations, a workflow to pick the appropriate setup for a particular study is detailed in the work by Nölle *et al.*[33]

The form factor can also impact the cell performance. Coin cells are commonly used in academic research as they do not require significant amounts of active material (<<1 g) and are easier to fabricate compared to pouch and cylindrical cells. However, they often exhibit significant deviation from the larger cell formats. For instance, they have unrealistically high electrolyte-to-capacity ratios, suffer from steel casing corrosion and higher impedances,[20] all of which limit their practical relevance. Due to the inherent differences, coin cells and pouch cells often yield different cycling performance, and, in some cases, even enable different degradation modes for the same battery chemistry.[34] For instance, coin cells have been shown to exhibit poorer rate performance, cyclability, and greater spread in results, than pouch cells.[32] Furthermore, they also fail to reproduce thermal ageing effect and may have non-uniform stack pressure, both of which are vital factors for real-world-representative testing.[35,36] Due to the difference in corrosion resistivity, the stainless-steel grade of the coin cell casing can also have an impact on the coulombic efficiency and cycle life, among other factors such as spacer thickness and separator type.[35] Nevertheless, coin cells will remain indispensable for early-stage studies due to their accessibility and ease of operation, but it is important to be aware of the aforementioned limitations while interpreting the results. There are ongoing efforts to bridge the gap between coin cell and pouch cell studies. For example, as the cycling stability of lithium metal battery coin cells is highly sensitive to the amount of electrolyte used, thickness of the lithium metal anode, and the cathode loading, Chen et al. have published a set of coin cell testing parameters based on a 300 Wh/kg stacked pouch cell in order for the coin cell testing results to better reflect those of practical cells.[27] Numerous best practices and tutorials on cell assemblies have also been published, detailing the impacts of assembly parameters on cell performance, as well as guidance to achieve reproducible results.[32,35,37] Relative trends derived from such cells are still informative, particularly for emerging battery chemistries where extensive benchmark datasets are lacking. Whereas for technologically mature battery chemistries such as lithium transition metal oxide cathodes, any report of breakthrough needs to be validated against the more stringent parameters relevant to practical application. The key is to report experimental details transparently so that the context and limitations of the conclusions drawn are clearly understood. It is also important to adopt a consistent approach to compute the performance metrics, such as specific capacities, energy densities and specific energies. To facilitate the consistent calculation of these figures of merit, several protocols have been reported.[38] In particular, Betz et al. demonstrated a systematic approach to computing specific energies and energy densities, starting at the active material level and then progressing towards the cell level, by taking into consideration the theoretical capacities of the anode and cathode active materials, excess electrolyte, inactive components, electrode expansion and cell housing at different steps.[38] Cross-study comparisons must be made on a consistent basis. For instance, electrode fabrication parameters and operating conditions can significantly influence the electrochemical behavior of a cathode active material, and therefore its performance evaluation. This is exemplified by comparing a cathode with 96% active material loading at 200 gsm (grams-per-square-metre) cycled at 1 C rate, against one with 80% loading at 15 gsm cycled at C/5; these configurations can yield markedly different performance metrics for the same material. Another example is the reported specific capacities of 3856 and 4210 mAh/g for lithium metal and silicon anodes, respectively, which might lead one to conclude that silicon anodes have higher specific capacity than lithium metal anodes. This is misleading as the former was normalized to the mass in the discharged state, whereas the latter was normalized to the mass

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in the charged state.[39] Recalculation of the capacities normalized to the mass of active material in the discharged state yielded 3856 and 2012 mAh/g for lithium metal and silicon anodes, respectively, highlighting how values can change significantly depending on how they are normalized. In view of the diversity of research objectives within the battery community, standardization should not be understood as enforcing a single, universal experimental setup. Rather, it involves acknowledging the community-wide concerns surrounding the limitations of different cell formats and electrochemical configurations, respecting these intrinsic differences, and drawing balanced and well-contextualized conclusions that accurately reflect the capabilities and constraints of each setup.

Inconsistency in data reporting, lack of standardized testing methodologies, coupled with the huge publication volume, pose a challenge to reliable comparison within a cell chemistry and between cell chemistries to identify the state of the art. A potential approach can be adapted from the photovoltaic community, which benchmarks technological progress through widely recognized reference charts such as the National Laboratory of the Rockies' "Best Research-Cell Efficiencies" and "Champion Module Efficiencies" charts.[40] Battery performance metrics go beyond energy and power densities, and commonly used plotting conventions fail to capture the cycle and calendar life, temperature sensitivity, and the energy and power densities at pack level. For example, the superior thermal stability of $LiFePO_4$ (LFP) has allowed LFP battery packs to achieve energy densities comparable to $LiNi_xCo_yAl_zO_2$ (NCA, $x + y + z = 1$) battery packs, despite the significantly higher theoretical specific capacity of NCA.[20] As such, bubble plots, termed the ENPOLITE (energy–power–lifetime–temperature) plots, have been suggested to capture the multidimensional, intertwined nature of battery performance metrics,[41] which can serve as the template for the reference chart to track technical progress in the battery field.

In battery research, electrochemical tests are often supplemented with other materials characterization techniques. Electrochemical data alone are insufficient for a complete mechanistic understanding; several failure modes are driven by changes in internal states that are either non-electrochemical in nature, do not produce identifiable electrochemical signatures, or remain latent until failure is imminent.[42] Consequently, early detection of these failure modes requires complementary inputs from other sources, e.g., physics-based modelling, which in turn relies on representative and well-curated experimental data. Similar to electrochemical testing described above, it is crucial that these characterization studies must be performed in a representative and standardized manner if the data generated are to be useful for further studies. However, efforts to standardize diagnostic battery characterization methods appear to have progressed slowly compared to their electrochemical counterparts, perhaps owing to the inherent diversity of techniques and their implementation. This has prevented the community from exploiting advances in AI and ML methods, which fundamentally depend on large, consistent, and curated machine-readable datasets. Without uniform data-sharing practices and reporting standards, the vast body of published work cannot be aggregated into any meaningful datasets for text mining or AI–ML-based analysis. This will become increasingly important as we move toward more chemically complex battery systems and sophisticated cell designs, which may require multi-modal data-analysis workflows for a comprehensive parameterization of battery degradation, potentially with the help of advanced AI and ML methods. However, this is a difficult task due to the inherently multi-faceted nature of battery research; battery performance and degradation depend not only on intrinsic material properties but also on the quality of material synthesis and cell fabrication and format, as well as the operating conditions. For example, when using non-standard electrochemical cells and testing methods for operando studies to evaluate performance, it is possible to introduce external factors that may lead to electrochemical performance variations.[43] However, these challenges are starting to be addressed, and steps are being taken to introduce standardized research practices into all facets of battery research.

THE CHALLENGES TO STANDARDIZING OPERANDO DIFFRACTION EXPERIMENTS FOR BATTERIES

As batteries are kinetically stabilized systems, operando methods are indispensable for capturing the non-equilibrium phases that form during electrochemical cycling. They are regularly used in fundamental investigations of structure–electrochemistry relationships in new material systems as well as the evaluation of cycling-induced performance degradation in mature cell chemistries under real-world conditions. Powder diffraction is particularly useful for both because[6,44,45]

1. it can be performed in research laboratories, as well as large-scale facilities, under ambient conditions with minimal human intervention and without any ancillary sample environment,
2. it allows for the direct time-resolved study of industrially relevant standardized cells with practical design specifications under representative operating conditions,
3. it provides space- and time-averaged crystallographic information about multiple cell components simultaneously,

it can be performed using both X-ray and neutron radiation, which, in principle, provide complementary insights due to differences in their scattering behaviour,[45]

5. it can be performed spatially (sometimes in three dimensions as X-ray diffraction computed tomography) with up to sub-micrometre resolution across the cell,
6. it benefits from well-established methodologies for high-throughput data processing and crystallographic analysis.

These factors enable the vast parameter space of battery research (e.g., manufacturing conditions, voltage windows, cycling rates, operating temperatures) to be covered in a reliable, reproducible and representative manner to produce datasets at the scale necessary to meet the three 'V's for AI–ML-driven analysis – *volume*, *velocity*, and *variety*,[46] and leverage the possibilities offered by these methods. Efforts to standardize operando diffraction experiments of battery cells and the transparent sharing of experimental data are currently hindered by inherent differences in the sample environment (the electrochemical cell), the experimental methodology (electrochemical cycling and diffraction data collection protocols), and data processing workflows. Each of these must be addressed to standardize the field and enable effective sharing of data.

For operando diffraction studies to yield meaningful results, the electrochemical cell design and operating conditions must not introduce additional degradation mechanisms or exacerbate existing ones. This is particularly important because electrochemical measurements reflect the behavior of the cell as a whole, whereas diffraction measurements are sensitive only to bulk crystal-structure changes; any influence of the cell design on electrochemical performance can therefore obscure correlations between crystallographic evolution and

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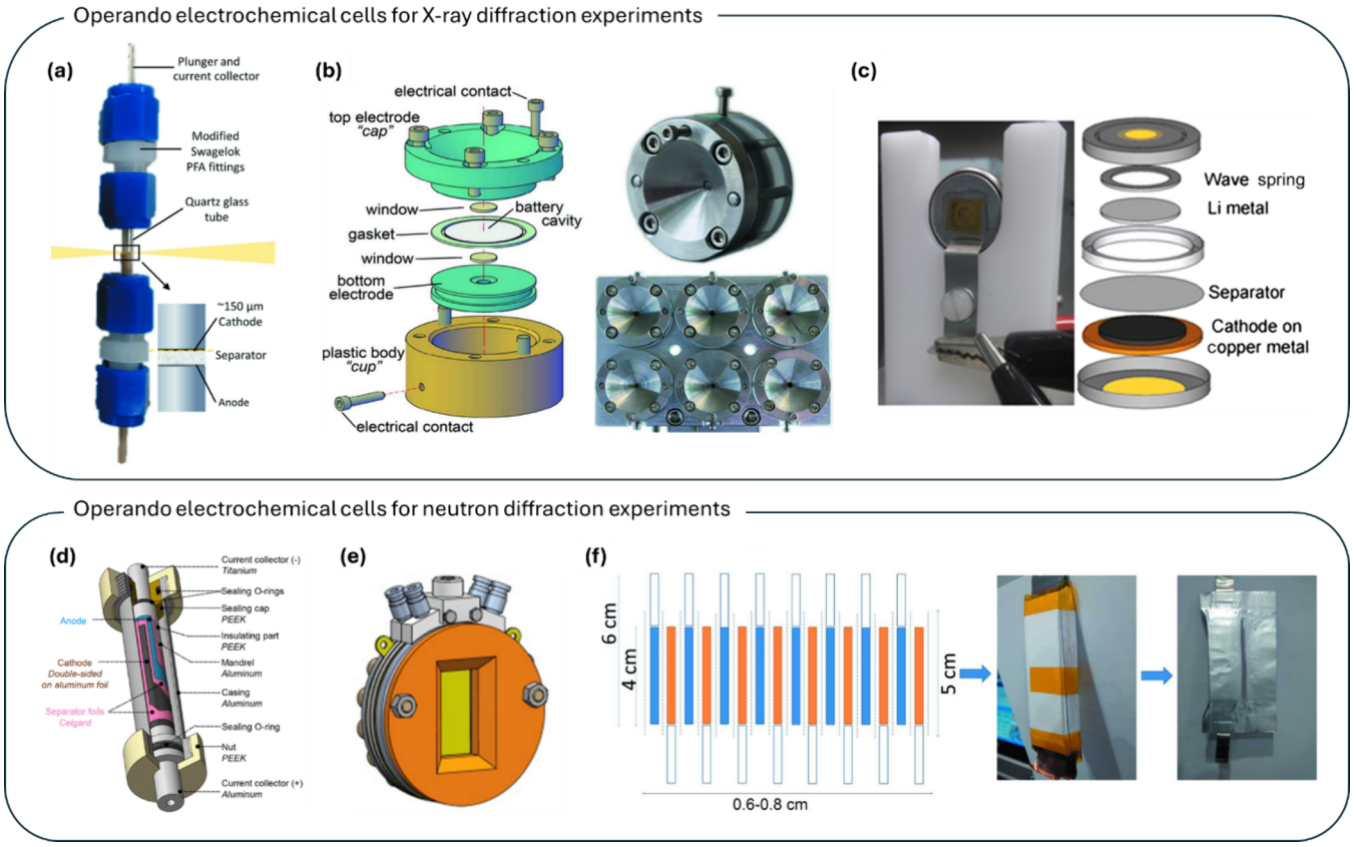

Figure 2. Selected operando (a–c) X-ray and (d–f) neutron diffraction cells. (a) DRIX cell,[47] reproduced under the terms of the Creative Commons CC BY 4.0 licence. (b) AMPIX cell,[48] reproduced with permission of the International Union of Crystallography. (c) A modified coin cell,[49] reprinted with permission. Copyright 2015 Elsevier B.V. All rights reserved. (d) A cylindrical cell,[50] reproduced under the terms of the Creative Commons CC BY 4.0 licence. Copyright 2018 Vitoux, Reichardt, Sallard, Novák, Sheptyakov and Villevieille. (e) A modified coin-cell-based cell,[51] reprinted with permission. Copyright 2013 The Authors. Published by Elsevier B.V. (f) A multi-layer pouch cell,[52] reproduced with permission of the International Union of Crystallography.

electrochemical response. As mentioned previously, the performance of a battery cell is significantly affected by its format and configuration, which can lead to misleading conclusions during electroanalytical testing. The same applies to operando diffraction experiments, perhaps to an even greater degree. These studies often rely on custom-designed cells (≤0.005 Ah) to accommodate the constraints of the diffractometer setup, optimize X-ray or neutron transmission for adequate signal-to-noise ratio, and in certain cases, isolate the signal from the component or material of interest[6,43,47,48,53] (see Figure 2 for a range of operando diffraction cells developed for X-ray and neutron diffraction experiments). For example, although modified coin cells are straightforward to fabricate, the additional build modifications for operando studies, such as the introduction of a X-ray-transparent polyimide window on the cell casing, can lead to the electrochemical behavior of the electrode encapsulated by the window differing from that of the surrounding regions due to differences in the applied pressure, creating property gradients within the electrode.[54] As a result, the measured response is not fully representative of the voltage profile, which captures the ensemble behavior. This may be overcome to an extent using a rigid window such as glass or glassy carbon, resulting in a more homogeneous pressure and reaction. Similarly, in cases where the electrodes are fabricated following unconventional methods, such as free-standing electrodes, uncalendared electrodes, and those with high carbon content and/or low active material loading, it is likely that the electrochemical performance is not representative of standard operation and testing. Therefore, it is necessary to benchmark the electrochemical performance of such custom-made cells to ensure reliable comparison between different studies and to provide all the necessary information about the electrochemical cycling and diagnostic testing history, along with the fabrication details of the cell. While some cells have demonstrated cycling performance comparable to standard coin cells,[47,48] it is also important to consider the specific conditions under which those results were obtained and to exercise caution when applying different cycling protocols. In addition, the variability introduced by the manual fabrication, such as the researcher's dexterity, experience and assembly procedures, may influence the electrochemical performance of custom operando cells.[35] In certain cases, especially for experiments at central user facilities, repeat measurements are often not feasible due to time constraints. While it may be impractical to develop a global benchmarking protocol for operando cells due to diversity in cell chemistries and operating conditions, the reliability and representativeness of such cells for operando measurements may be ensured by including a direct comparison against an unmodified (standard) cell of the same kind fabricated using the identical electrodes and cycled under the same cycling protocol.

Recent advances in X-ray and neutron technologies have now made it possible to directly study larger cell types such as (single-/multi-layer) pouch, cylindrical (0.1–5 Ah) and even prismatic (>10 Ah) cells using diffraction (Figure 3).[57] We have previously demonstrated that single-layer pouch cells are suitable for operando X-ray (laboratory and synchrotron) and (cold-)neutron diffraction,[58–61] absorption spectroscopy,[58,62] and small-angle scattering,[62] without any modification or compromise to electrochemical performance. Crucially, these cell types can be fabricated at scale with practically relevant design specifications

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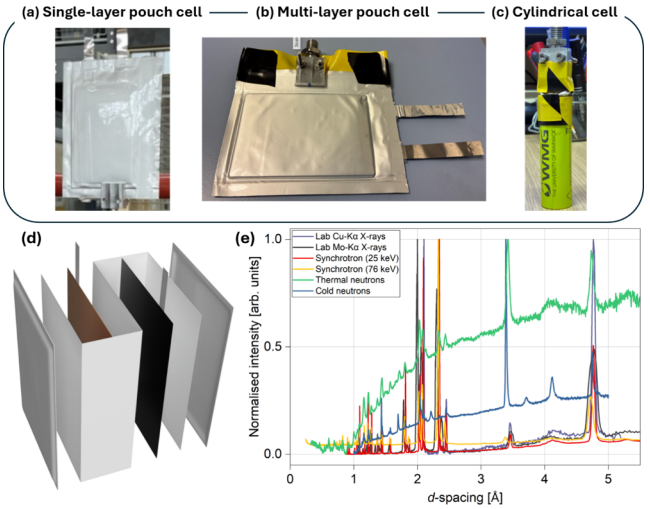

Figure 3. (a–c) Large format cells used for operando diffraction measurements. (d) An illustration of a single-layer pouch cell. (e) Diffraction patterns from a SLP cell obtained using different sources (Lab Cu-Kα: Xenocs Xeuss 2.0; Lab Mo-Kα: Panalytical Empyrean; Synchrotron (25 keV): I11 Long duration experiments facility,[55] Diamond Light Source; Synchrotron (76 keV): I15-1, Diamond Light Source; Thermal neutrons: Polaris,[5] ISIS Neutron and Muon Source; Cold neutrons: WISH,[56] ISIS Neutron and Muon Source.

(active material loading, electrolyte content *etc.*) and hence, offer better reproducibility and reliable long-term electrochemical performance representative of practical operation. However, it is worth noting that some formats, specifically the multi-layer pouch, cylindrical and prismatic ones, are sub-optimal for iterative academic studies, as they are resource- and time-intensive to fabricate. Furthermore, they introduce additional challenges for operando diffraction studies, as data from such unmodified cells are inherently complex due to the overlapping contributions from all the cell components including the casing, metallic foils, electrode active materials, conductive additives, binders, electrolyte and separator. This leads to greater beam attenuation and higher background scattering, complicating the crystallographic analyses of the electrode active materials, which are often the focus of such studies. In addition, the larger probe volume and the anisotropic form factor of certain cell formats mean that sample geometry effects may also contribute to data significantly. These factors make conventional Pawley and Rietveld refinements difficult due to the higher risk of over-parameterization. Notwithstanding these difficulties, single-layer pouch cells with electrodes fabricated to representative specifications appear to be the most suitable for academic studies investigations at an industrially relevant scale.

In an operando diffraction experiment, there is an implicit assumption that the diffraction measurement in itself does not alter the electrochemical reaction under investigation.[54] For high-energy synchrotron X-ray diffraction in particular, however, this assumption does not always hold. Here, the measurement itself can perturb the system, and depending on the cell chemistry, electrode thickness, beam energy, dose, and dose rate, the effects can range from partial or total reaction inhibition to artificially induced phase transitions at the probed area. These are typically referred to as beam damage or beam inhibition, depending on whether the beam-induced changes are irreversible or reversible, respectively.[54,63,64] Multiple explanations have been put forward to explain the reaction latency at the probed area, including the formation of gas bubbles and decomposition of electrolyte and carbon-binder matrix. In most cases, the probed area is significantly smaller than the total electrode area, contributing only a fraction of the overall battery capacity. Consequently, when beam-induced effects occur, their influence is often masked in the global electrochemical response because the unexposed portions of the electrode continue to cycle normally, rendering the correlation between the X-ray diffraction and the electrochemical data unreliable. While the relatively small X-ray beam sizes may be advantageous for localized studies, electrochemical reactions in battery cells are inherently spatially heterogeneous, especially at high (dis)charging rates.[3,4,65] Experimental observations can depend strongly on the chosen region of interest and may not reflect the overall electrochemical behavior of the cell, necessitating careful experimental design to disentangle cell-derived, electrochemistry-derived, and beam-derived effects in order to draw valid and representative conclusions. These concerns can be mitigated to some extent by ensuring that the total X-ray dose delivered during the synchrotron experiment remains below system-specific dose thresholds by reducing the flux using an attenuator, and by collecting data intermittently from unexposed regions of the sample. Spatial heterogeneity in electrochemical reactions can be further addressed by performing XRD mapping of the electrode, rastering the beam across the region of interest.[60] In neutron diffraction studies, the larger footprint of the beam (on the order of centimetres) means that diffraction data are averaged across a greater area of the cell, giving a signal more representative of the cell as a whole. Furthermore, due to the nature of neutron–matter interactions, neutrons are considered to not cause any significant beam damage.[45]

The analysis of operando diffraction data is more straightforward than the experimental execution. It benefits from well-established methodologies such as sequential Pawley or Rietveld refinements, which are now standard options in analysis software suites.[66–68] More recently, machine and deep learning methods have also been applied to the analysis of operando diffraction data, although their application requires rigorous validation to ensure reliability.[69] Nevertheless, as discussed previously, diffraction data from battery cells, especially unmodified ones, can contain contributions from up to eight or nine components exhibiting varying degrees of crystallographic order. Analysis of such multi-component, multi-phase data are not straightforward using conventional methods and may necessitate the incorporation of data-driven approaches such as non-negative matrix factorization (NMF) into analysis workflows. Equally important to the diffraction data analysis is the accurate temporal correlation of electrochemical and diffraction datasets, which is an essential step in the measurement workflow. This is typically achieved using the timestamps of each dataset as references, either manually or through bespoke programming scripts. However, this is often non-trivial due to differences in the recording and sampling frequencies of the two datasets; diffraction data are usually collected at a significantly lower frequency than electrochemical data. The challenge is further compounded by experimental uncertainties such as cell-to-cell performance variations and by interruptions due to beam loss. Together, these issues complicate robust temporal alignment and limit the ability to share raw data in a user-friendly and meaningful manner. This is particularly acute when data collection occurs rapidly (under one second per frame), involves non-standard cycling protocols, or extends over long durations. In the absence of proper correlation and reporting standards, such inconsistencies may go unnoticed.

In summary, the challenge of standardizing operando battery diffraction experiments can be distilled into two core issues, while acknowledging that a universal solution is unfeasible given the diversity of research objectives and experimental methodologies. The first concerns the electrochemical cell: it must be ensured that the cell design and its operation remain as representative of standard electrochemical testing as possible, and that any compromises made to optimize operando diffraction data quality do not introduce spurious degradation. Benchmarking the electrochemical performance of the cells, using protocols similar to reference performance tests is a simple way that this can be ensured.

The second concerns data sharing: achieving full transparency of operando diffraction experiments requires the sharing of three key datasets: (1) cell fabrication and electrochemical testing/benchmarking

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data; (2) the operando measurement data, including all metadata and the correlated electrochemical and characterization data; (3) and the resulting data-processing and analysis outputs. The second of these is a data-intensive endeavor that may be addressed by the development of modified NeXus application definitions tailored for operando diffraction data. However, standardizing the analysis outputs of the third, e.g., Rietveld refinement results, remains an open problem. These are both timely and necessary, particularly in light of the growing opportunities afforded by AI- and ML-driven data analysis. Addressing these challenges and establishing such standards will be central to the future directions discussed in the following section.

■ FUTURE DIRECTIONS

When conducted transparently with standardized, machine-readable outputs, operando diffraction studies of battery cells can generate multi-modal electrochemical and diffraction datasets that could catalyze data-driven discoveries through machine learning. A compelling precedent is *AlphaFold*, the protein structure prediction algorithm,[70] whose success was built upon decades of experimentally solved crystallographic structures hosted in open resources such as the Protein Data Bank. While highly promising, the application of data-driven approaches in materials science domains faces distinct challenges arising from fundamental differences in data generation and structure. In particular, protein diversity is shaped by evolutionary processes acting on a limited set of 20 amino acids, whereas functional materials are deliberately designed by humans for a wide range of applications, spanning a combinatorial space that covers much of the periodic table.[71] Consequently, data are often fragmented, necessitating more targeted computational approaches for different use cases that explicitly incorporate domain knowledge. Analogous challenges exist in battery research, where the lack of standardized approaches for characterization and data reporting has limited the use of data-driven methods across different studies, wherein varying methodologies and characterization techniques are employed. As highlighted in Attia et al., the complexity of dynamic multi-component battery systems requires a multi-modal approach for a comprehensive study, which in turn depends on consistent, machine-readable data.[42]

A practical route to standardizing operando diffraction datasets is the incorporation of electrochemical data into pre-existing NeXus application definitions for diffraction techniques. NeXus application definitions specify the minimum set of terms that must be present in a compliant data file, together with optional terms whose spelling and description are formally reserved, thereby ensuring that certain information will be available in a consistent, machine-readable form. Rather than creating new definitions, the approach proposed here extends two existing ones that already encode the essential diffraction-specific metadata for their respective techniques: NXmonopd, which covers monochromatic (neutron and X-ray) powder diffraction, and NXtofnpd, for time-of-flight neutron powder diffraction. The extensions follow the same structure in both cases: the electrochemical additions are technique-agnostic and sit in a separate layer on top of the diffraction-specific information. This approach organizes the full experimental record (electrochemical data, diffraction patterns, and the corresponding metadata) within a single NeXus-format HDF5 file, streamlining both data sharing and archiving. Figure 4 describes the structures of the proposed operando definitions modelled after NXmonopd and NXtofnpd definitions. At the top level, a single NXentry group holds the global metadata that applies to the whole experiment: the instrument description, the sample information, and the electrochemical cycling protocol. Each acquisition links to the instrument group rather than copying it, since the measurement setup remains invariant during the experiment. To ensure data comparability and reusability, a consistent description of cell chemistry, format, and fabrication will be treated as mandatory within the sample information. Each individual diffraction scan is then stored as a sequentially indexed NXsubentry, with sub-entries named *scan_000001* to *scan_N*. This structure follows the NeXus International Advisory Committee (NIAC) recommendation for structuring multi-modal NeXus data, and mirrors approaches already demonstrated at BESSY II.[72] Within each sub-entry, three groups capture the acquisition-level information. The *ENVIRONMENT* group is the electrochemical extension that records the instantaneous state of the battery cell at the moment the diffraction pattern was collected: voltage, current, and a timestamp. As discussed previously, proper temporal correlation between the electrochemical and diffraction data streams is essential, since electrochemical data are collected at a far higher frequency than diffraction patterns. In practice, the timestamp of each diffraction acquisition may be used to identify the closest matching electrochemical data point in the raw potentiostat file. The *MONITOR* group records the incident beam monitor data for that acquisition, for use in normalization and related corrections. The *DATA* group stores the diffraction pattern itself: intensity as a function of scattering angle for NXmonopd, or intensity as a function of time-of-flight for NXtofnpd, with the abscissa linked directly to the detector description in the top-level instrument group rather than duplicated. Where X-ray diffraction data are recorded using a 2D area detector, the raw detector images are typically too large to embed directly within the NeXus file. In such cases, a link to the image stored in an external repository may be provided instead, preserving accessibility without compromising file portability.

To support the practical adoption of these standards, we have developed OPERA*XN*,[73] a software tool aimed at making the correlation, interrogation, and dissemination of large operando diffraction datasets more accessible. Currently in beta, OPERA*XN* enables the interactive visualization of correlated electrochemical and diffraction data and automates the generation of NeXus files conforming to the definitions described above, consolidating electrochemical cycling data, experimental metadata, and diffraction patterns into a single, self-describing file. However, for tools such as OPERA*XN* to become broadly useful, their functionality must continue to expand to accommodate additional instruments, data formats, experimental workflows, and sample histories in a standardized form.

A standard is only as useful as its adoption, which depends on both formal governance and practical incentives. NeXus application definitions follow an established governance pathway maintained by the NIAC. The operando extensions proposed here will have to be reviewed and ratified by the NIAC for formal adoption. As NeXus is already the native or supported data format at most major synchrotron and neutron facilities, the incremental cost of implementing an operando extension at diffraction beamlines is minimal. It is equally important to align incentives across the different stakeholders to ensure widespread adoption. For researchers, the burden of compliance can be minimized through conversion tools such as OPERA*XN* that generate NeXus files directly from potentiostat

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NXoperando_monopd/NXoperando_tofnpd

Application definition, extends NXmonopd or NXtofnpd

Symbols: nP = echem. points

ENTRY: (required) NXentry experiment metadata
- title: (required) NX_CHAR
- start_time: (required) NX_DATE_TIME
- end_time: (optional) NX_DATE_TIME
- experiment_identifier: (optional) NX_CHAR
- definition: (required) NX_CHAR (*NXoperando_monopd*)
- program_name: (required) NX_CHAR (*software*)
- **PROCESS:** (required) NXprocess (*correlation provenance*)
  - correlation_method: (required) NX_CHAR (*absolute/relative*)
  - data_source: (optional) NX_CHAR (*instrument*)
- **INSTRUMENT:** (required) NXinstrument Inherited from NXmonopd/NXtofnpd
- **SAMPLE:** (required) NXsample (*global sample metadata*)
  - name: (required) NX_CHAR
  - description: (required) NX_CHAR (*cell chemistry, fabrication details, cycling history*)
  - preparation_date: (optional) NX_DATE_TIME
- **CYCLING_PROTOCOL:** (required) NXnote
  - technique: (required) NX_CHAR (*aging protocol before operando experiment*)
  - voltage_window_lower, _upper: (optional) NX_FLOAT {NX_VOLTAGE}
  - C_rate: (optional) NX_CHAR
  - instrument: (optional) NX_CHAR (*potentiostat model*)
  - software: (optional) NX_CHAR
  - raw_data_file: (optional) NX_CHAR (*link or DOI*)
- **operando_electrochemistry:** (required) NXdata
  - attributes: @signal = voltage, @axes = time
  - time: (required) NX_FLOAT[nP] {NX_TIME}
  - voltage: (required) NX_FLOAT [nP] {NX_VOLTAGE}
  - current: (recommended) NX_FLOAT [nP] {NX_CURRENT}
- **SUBENTRY[scan_000001 ... scan_N]:** (required, repeatable) NXsubentry
  - start_time: (required) NX_DATE_TIME
  - end_time: (recommended) NX_DATE_TIME for long diffraction scans
  - instrument: link (target: /NXentry/NXinstrument)
  - **ENVIRONMENT:** (required) NXenvironment (*state during scan*)
    - voltage: (required) NX_FLOAT {NX_VOLTAGE} (*at midpoint of scan*)
    - current: (required) NX_FLOAT {NX_CURRENT}
    - capacity: (optional) NX_FLOAT {NX_CHARGE}
    - scan_timestamp: (required) NX_DATE_TIME
    - midpoint_adjusted_timestamp: (optional) NX_DATE_TIME
    - exposure_time: (recommended) NX_FLOAT {NX_TIME}
    - voltage_min, voltage_max: (required) NX_FLOAT (*voltage window*)
    - current_min, current_max: (required) NX_FLOAT (*current window*)
    - voltage_log, current_log: (optional) NXlog
    - echem_index_start, _end: (optional) NX_INT
  - **MONITOR:** (required) NXmonitor Inherited from NXmonopd/NXtofnpd
  - **DATA:** (required) NXdata Inherited from NXmonopd/NXtofnpd

Figure 4. Application definitions for NXoperando_monopd and NXoperando_tofnpd extended from NXmonopd and NXtofnpd, respectively. The highlighted sections (in green) are the extensions beyond the original. Note that NXtofnpd also has 'pre_sample_flightpath' as a required field in the ENTRY section.

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and diffractometer outputs, removing the need for manual reformatting. For facilities, native export of operando data in the extended NeXus format embeds the standard into the measurement workflow itself, requiring no additional effort from users. Finally, journals and perhaps, funders, hold the strongest lever for making standardized data sharing the default rather than the exception. Policies should encourage, and mandate (where appropriate), the deposition of operando datasets in compliant formats, analogous to the crystallographic community's long-standing CIF deposition requirements.

The application definitions proposed above are also a prerequisite if operando diffraction experiments are to be driven, at least in part, autonomously. Considering the vast parameter space for battery manufacture and operation, autonomous experimentation will be key to growth in this field. Discussions of autonomous experimentation tend to focus on machine learning models that analyze diffraction patterns in real time. But a harder problem sits one layer below: before any model can usefully drive an experiment, the data it receives must be consistent across instruments, complete, and properly synchronized with the electrochemical state of the cell. Without these foundations, even a sophisticated algorithm will make decisions based on unreliable inputs, and the errors it introduces may not become apparent until long after the beamtime has ended.[13,74] Real-time data processing is especially challenging for event-mode acquisition at time-of-flight sources, where a single continuous data stream must be reduced on the fly. Standardized data formats directly address this problem. When diffraction patterns and electrochemical measurements are stored together in a shared, machine-readable structure, they can be compared across beamlines, fed into analysis pipelines without manual reformatting, and used to trigger automated decisions with confidence. The reliability, representativeness, and reproducibility concerns already widely discussed in operando battery research[13] are therefore not separate from autonomy: they are its precondition.

Autonomous operation also introduces a metadata requirement that current operando schemas do not yet address: a record of how decisions were made. An autonomous campaign needs to log not only what data were collected, but what the algorithm observed, what action it recommended, whether a human approved that recommendation, and what the beamline actually executed. Without this provenance record, even a richly described dataset cannot answer the most important question when something goes wrong. Several concrete requirements follow from this. Electrochemical and diffraction data must be synchronizable as structured data, with explicit handling of timing gaps caused by beam loss or detector readout.[74] Beam dose should be stored alongside the data as a quantifiable variable, not mentioned only in the discussion section as an unexplained artefact.[74] Calibration provenance, integration masks, background models, and phase-tracking choices should link raw detector counts to the processed patterns that any machine-learning stage will later consume, since automation built on undocumented analysis steps cannot be audited or trusted.

Deployment of autonomy at research facilities will be gradual. It is likely to begin with shadow-mode monitoring, in which an algorithm watches the experiment without intervening. From there it would progress to human-in-the-loop operation, in which the algorithm recommends actions that a beamline scientist approves, before eventually being granted bounded authority to trigger acquisitions within a pre-agreed experimental envelope. Responsibility for any individual measurement must remain visibly with the scientist throughout. Metadata frameworks, including the application definitions proposed here, therefore need to be designed from the outset to record those shifting levels of human and algorithmic authority alongside the data they govern. Looking forward, three developments would be particularly valuable. First, a shared benchmark culture, in which datasets are published alongside matched experimental protocols, would allow claims about the benefits of autonomous experimentation to be tested objectively.[75,76] Second, the standards architecture should be built around a small mandatory core, extended by community-curated fields aligned with ontologies such as *BattINFO*,[23] and designed to accommodate the full multimodal picture, since many battery failure modes cannot be identified from diffraction alone. Third, attention must extend beyond the measurement to the analysis outputs themselves, such as the results of Rietveld refinements, which remain unstandardized. The application definitions proposed are a first step toward both goals: by encoding the minimum information needed to make an operando diffraction dataset machine-readable and interoperable, they lay the groundwork for autonomous experimentation, open benchmark datasets, and a shared data infrastructure, as a practical next stage in the development of the field.

In conclusion, operando diffraction has become indispensable for resolving the dynamic processes that govern how batteries perform and fail. Yet its value is increasingly limited not by what the technique can measure, but by how the resulting data are produced and shared. Towards this, it is vital that we standardize the workflow to ensure maximum utility of the data produced. Currently, the obstacles to standardization are, (1) keeping the electrochemical cell representative of standard testing despite the demands of operando measurements, and (2) capturing the full experimental record (cell fabrication and history, correlated electrochemical and diffraction data, and the analysis linking them) in consistent, machine-readable formats. The latter can be addressed directly, through extensions to the NeXus-based NXmonopd and NXtofnpd application definitions that embed electrochemical state alongside diffraction data in a single self-describing file, compatible with laboratory, synchrotron, and neutron instruments alike. Such a scheme is a starting point, not a finished standard; its value will be realized only through community adoption and the sustained effort needed to accommodate the full diversity of cells, instruments, and workflows. This effort is worth undertaking, because standardized, FAIR-compliant data are the foundation for everything the field now hopes to build on them, e.g., reproducible cross-study comparison, machine-learning analysis, and autonomous experimentation. The framework set out here is offered as a pathway towards that.

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