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Publisher’s version / Version de l’éditeur:
https://doi.org/10.1107/S1600576721008839
Journal of Applied Crystallography, 54, 5, pp. 1416-1423, 2021-10

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Conflat cell for \textit{operando} electrochemical X-ray studies of Lithium-ion battery materials in commercially-relevant conditions

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\textit{In-situ} and \textit{operando} techniques play an important role in modern battery materials research and development. As materials characterization and application requirements advance, so too must the \textit{in-situ} / \textit{operando} test methods and hardware. The effects of temperature, internal mechanical pressure, and parasitic reactions due to e.g. cell sealing are critical for commercial scale up, but often overlooked in \textit{in-situ} / \textit{operando} cell designs. An improved electrochemical \textit{operando} cell for X-ray diffraction and spectroscopy using Conflat-style flanges in combination with a Beryllium window is presented. The cell is reusable, simple to fabricate and assemble, provides superior sealing, relevant and adjustable cell stack pressure and reproducible charge / discharge cycling for short and long term experiments. Cell construction, electrochemical performance, and representative \textit{operando} X-ray powder diffraction measurements with Carbon and Aluminum electrodes at temperatures between 30 - 120°C are provided. \textit{Operando} electrochemical cell testing at high temperatures allows access to and analysis of temperature-sensitive phase transitions and opens the way for analysis and development of new Lithium-based cathode, anode, and electrolyte battery materials.

1. Introduction

Lithium-ion batteries have emerged as the dominant energy storage technology not only for electronic devices but also for electric vehicles and grid-scale applications (Zubi \textit{et al.}, 2018; Li \textit{et al.}, 2018; Zeng \textit{et al.}, 2019; Hesse \textit{et al.}, 2017; Denholm \textit{et al.}, 2020). However, new types of batteries will be needed for the future (Turcheniuk \textit{et al.}, 2018) and improvements of the current technology and development of new materials depend heavily on reproducible characterization. The overall challenge is to replicate real-world battery operating conditions in laboratory measurements, which makes designing a robust \textit{in-situ} / \textit{operando} cell a challenging task. \textit{In-situ} cell materials should be as close to those used in production cells as possible (e.g. steel, aluminum, copper, nickel) (Zwick \textit{et al.}, 2020) and Lithium should only react with the test electrode, but not with the cell components or the test environment. Additional constraints include the need to apply significant mechanical pressure (0.1 - 1 MPa) to the electrochemically active material (Kermani & Sahraei, 2017), and in some cases, operate over a wide temperature range. In practice this rules out most plastics as some (e.g. polytetrafluoroethylene, PTFE) react with Lithium at low potentials (Periyapperuma \textit{et al.}, 2014) and others (e.g. polypropylene) do not retain their mechanical strength or ability to act as a gasket at moderate to high temperatures. While existing \textit{in-situ} / \textit{operando} electrochemical cell designs meet many of these criteria, no design meets all of them.

Lithium-ion test cell formats include coin, pouch, Swagelok and Conflat-style cells. Coin cells are very common for testing Lithium-ion electrode or electrolyte materials and are similar to a watch battery with a thin steel casing and a polymeric gasbag. These cells can be transformed into \textit{in-situ} cells (Richard \textit{et al.}, 1997; Yang \textit{et al.}, 2015) by machining a hole in one of the casings and incorporating an X-ray transparent window. Some window materials like Kapton are X-ray transparent but are electrically insulating and cannot support the required mechanical stack pressures. Carbon and Beryllium are good alternatives that can usually support mechanical pressures and withstand high temperatures. However, coin cells are designed to only operate at moderate temperatures as their plastic gasket, which acts as both a cell sealant and electrical isolation between electrodes, cannot sustain prolonged exposure to high temperatures. Coin cells generally suffer from inconsistent sealing and variable or uncontrolled stack pressure that represents a compromise on electrochemical test integrity.

Pouch cells (Walus \textit{et al.}, 2013) use plastic or aluminized plastic bags for the cell casing, and therefore are also designed for moderate temperature measurements. Aluminum is only moderately X-ray transparent and thus complicates investigations with standard laboratory X-ray sources and diffractometers. Pouch cells, when incorporated in a battery pack, are held tightly together with external supports as they can not provide sufficient mechanical pressure on their own. Pouch cells that are used for \textit{operando} X-ray experiments must either then include external support or sacrifice electrochemical performance in
order to allow straightforward X-ray measurements in transmission or reflection geometry at synchrotron sources.

Swagelok cells (Morcrette et al., 2002; Leriche et al., 2010; Sottmann et al., 2016) combine commercially available Swagelok fluid connectors typically made of thick stainless steel, with plastic or rubber gaskets for electrochemical and electrical isolation. Some designs have been used at high temperatures with alumina components which can crack when directly exposed to electrolyte (Muñoz-Rojas et al., 2007). Swagelok cells could offer commercially relevant stack pressure, however, cell sealing is based on torque not the absolute position of the sealing system, so the spring compression and stack pressure can vary substantially. Swagekok cells also have limited flexibility in size.

Other cell types include capillary cells (Johnsen & Norby, 2013) that have micro electrodes inserted in a glass capillary that is a few mm in diameter. Capillary cells are good for performing synchrotron diffraction, spectroscopy, imaging and tomography measurements (Wang et al., 2015), but cannot maintain commercially relevant stack pressure and use completely different geometry than commercial cells.

Specialized in-situ cells include, for example, Argonne’s multipurpose in-situ X-ray (AMPiX) cell (Borkiewicz et al., 2012) and some other examples (Bleith et al., 2015; Wujcik et al., 2016). These cells are typically sealed using rubber gaskets or o-rings that cannot sustain prolonged testing at high temperatures and may compromise on mechanical pressure. Specialized manufactured components of these cells also make them cost prohibitive for many laboratories, and difficult to reproduce or use in a highly parallel fashion.

Conflat cells, based on Conflat vacuum flanges and electrically insulating feedthroughs, employ common and readily available components and can deliver an ultra-high vacuum compatible seal, controllable and commercially relevant stack pressure and high temperature compatibility (Periyapperuma et al., 2014; Fleischauer et al., 2019b). The seal is maintained using knife edges impinging on a Copper gasket. Conflat cells have the potential to meet all of the criteria for a robust and reproducible in-situ / operando cell. In this paper we describe a new Conflat in-situ / operando cell with a Beryllium window, test its electrochemical performance and provide X-ray powder diffraction results from operando experiments on the lithiation of Graphite and Aluminum electrodes. The challenge lies in properly incorporating an X-ray transparent window for operando X-ray measurements. While Beryllium windows can be attached to the Conflat flange using metal bonding techniques, such bonding can be cost prohibitive for many laboratories (e.g. thousands of dollars per flange). In our design, we have used JB Weld steel reinforced epoxy to attach the Beryllium window to the Conflat flange, making it an inexpensive and high quality alternative even for long-term operando measurements at a wide range of temperatures.

2. Cell construction

The cell consists of two vacuum flanges sealed with a Copper gasket, as shown in Figure 1.

![Figure 1](image)

(a) Exploded view and cross-section of the cell assembly. Components are labeled and described in the text. (b) A nearly assembled cell without the top flange. (c) The underside of a top flange showing the epoxy (black ring) and Beryllium foil.

The bottom flange is a commercially available 304 stainless steel Conflat flange with an isolated 3-pin electrical feedthrough (Ceramtec, Laurens, South Carolina, USA, item 17020-01-CF). The top flange is a standard 304 stainless steel Conflat flange (ANCORP, Williston, Florida, USA, item CF275T-000) with two modifications - a 15 mm diameter through hole for the X-ray beam, and a large diameter blind hole for the Beryllium window, epoxy sealant, and internal components on the knife-edge side of the flange. Beryllium discs were purchased from Materion Electrofusion (Mayfield Heights, Ohio, USA) and attached to the underside of the top flange using J-B Weld steel-reinforced epoxy, as shown in Fig. 1. Uncoated Beryllium windows can be used with positive electrode materials that have operating voltages up to 3.5 V vs. Li/Li⁺, as higher operating voltages will cause Beryllium oxidation (Leriche et al., 2010).

Blind hole dimensions are critical to balance scattering angle, stack pressure, and physically isolating the epoxy from possible reactions with the cell electrolyte. Blind hole diameter was typically 36 mm with 30-32 mm diameter Beryllium windows to enable placement of the electrically insulating epoxy around the circumference of the window. After the blind hole was machined, approximately 0.7 mm of the flange remained to provide stack pressure.
The cell is assembled as follows: the stainless steel platform shown in Figure 1(a) (machined from a 304 stainless steel rod of the same diameter) is mounted onto the 3-pin feedthrough and provides the concentric alignment of the cell components. A spring (Belleville washer, to provide the stack pressure) is placed on the platform followed by a stainless steel spacer (whose thickness depends on the thickness of the test electrode and desired the spring compression, and can be adjusted with shim stock), an 18 mm diameter Lithium disc, 19 mm diameter fibreglass separator soaked with 200 µL of electrolyte, and finally the material to be tested. Freestanding metallic foils and electrodes coated on Copper foil were used directly, however, other types of electrodes could be deposited directly on the Beryllium or other types of X-ray transparent windows. Lithium was brushed only on the side facing the separator. Cell assembly was performed in an Argon atmosphere glovebox maintained at less than 5 ppm H₂O and O₂. Cell bolts (visible in Fig. 1(b)) were tightened to 17 N m of torque using a torque wrench, or until the flange faces were in contact. The minimum 2θ scattering angle is approximately 16° with a 0.3 mm thick Beryllium window and a 0.7 mm thick flange.

The cost of each cell is estimated to be close to 500 USD (not including the cost of machining the top flange and platform) with the Beryllium disc being the most expensive part of the cell and varying greatly depending on the specifications. Feedthroughs and standard flanges cost on the order of 200 and 20 USD, respectively. The rest of the cell components are standard coin cell components that are readily available from various manufacturers.

3. Mechanical performance

In order to provide a relevant stack pressure the components of the cell must be pressed against the Beryllium window. The Beryllium window and top flange will act as a spring and need to have a higher or equal spring constant compared to the spring (Belleville washer) mounted inside the cell. Force-displacement data on a flange with a 15 mm opening was collected using an Instron 5966 Universal Test System to ensure that the Beryllium window and flange assembly had a high enough spring constant to be able to withstand the pressure from the cell components. Force-displacement data collected using an 18 mm diameter piston is presented in Fig. 2.

Pressures are also provided in Fig. 2. Commercial cells typically have a stack pressure ranging from 0.1 to 1 MPa normal to the electrode surface. The spring (Belleville washer) mounted inside of our cell can provide up to 0.63 MPa of pressure (160 N) at 0.5 mm full compression, or 0.4 MPa (100 N) at 0.25 mm compression using 18 mm diameter electrodes. Smaller diameter pistons / electrodes would enable higher pressures to be reached at a given applied force. As seen in Fig. 2, the flange itself can sustain much higher forces at much lower deformation; an applied force of 160 N causes about 0.12 mm deflection and is well within the elastic deformation of the top flange assembly. Thicker flanges would further increase the stiffness of the assembly at the cost of a smaller angular range.

4. Electrochemical performance

Operando electrochemical cells are becoming a standard tool at many laboratories and large scale facilities, however operando experiments are still being treated as a niche technique and rigorous electrochemical testing of operando cells is rarely performed. As one of the most important parameters of Lithium-ion cells is their longevity, the same goes for operando cells designed to probe new materials. Overall lifetime is the product of the cell components and the active materials being studied. Electrochemical performance and the longevity of our cell was judged based on its capacity retention and coulombic efficiency with standard materials tested in standard and harsh conditions.

Capacity retention addresses the core need of reliable energy storage devices and is the most commonly reported metric of candidate energy storage materials. Laboratory electrochemical cells are usually tested over the course of many tens to hundreds of cycles over the course of days or even months, however, with some exceptions (e.g. (Braun et al., 2003)), operando electrochemical cells are usually tested over the course of hours to a few days (e.g. (Li et al., 2020)), in line with schedules at many synchrotrons. Lab-based in-situ / operando testing requires much more extensive cycling. It is common for the capacity of Lithium-ion cells to increase over a few first cycles and then gradually decrease until the cell is no longer functioning. The cell is considered to have reached its end-of-life when the capacity drops below 80% of its original capacity.

Capacity retention is a useful but sometimes coarse metric of material performance. Coulombic efficiency describes charge transfer efficiency, i.e. it is the ratio of the total charge / Lithium that is extracted from the test material to the total charge / Lithium that is introduced into an electrode over a discharge / charge cycle (Dahn et al., 2016; Schulze & Neale, 2021). Parasitic reactions, e.g. a leaky cell, reduce coulombic efficiency by consuming Lithium in irreversible reactions and preventing it from being electrochemically moved between electrodes.
during discharge / charge cycling. The coulombic efficiency of laboratory grade Lithium-ion cells at moderate charge rates and temperatures should exceed 99% for commercially-relevant materials, and for commercial cells with a long lifetime, should exceed 99.99% over hundreds of cycles (Dahn et al., 2016). The coulombic efficiency of operando and in-situ cells is not frequently reported, but critical to ensure ex- and in-situ experiments are investigating the same reactions.

Conflat-style cells with standard solid flanges can demonstrate high coulombic efficiencies and good capacity retention with standard electrode materials like graphite (Periyapperuma et al., 2014; Fleischauer et al., 2019b). For the purpose of testing at ambient temperatures, our operando Conflat cell was assembled with Lithium and Graphite electrodes. 17.5 mm diameter, 50 µm thick CMS Graphite coated on 9 µm thick Copper foil was punched from a commercial electrode sheet (MTI Corporation, Richmond, California, USA), a 19 mm diameter fiberglass separator (Whatman GF/A, Sigma-Aldrich, Oakville, Ontario, Canada) and 1 M LiPF$_6$ in 1:1 vol:vol. ethylene carbonate : diethyl carbonate electrolyte (battery grade, Sigma-Aldrich) was used. Galvanostatic cycling was performed between 1.2 V and 10 mV vs. Li/Li$^+$ using 0.5 mA charge and discharge currents (approximately C/10) on a lab-built multichannel system with integrated temperature control at 30$^\circ$C over the course of 22 days. Cell capacity and coulombic efficiency are provided in Fig. 3(a) and (b), respectively.

![Figure 3](image)

**Figure 3**

(a) Capacity and (b) coulombic efficiency as a function of cycle number and (c) potential as a function of capacity of the Lithium/Graphite operando Conflat cell. Measurements were performed at 30$^\circ$C.

After a few cycles, cell capacity (Fig. 3(a)) has reached nearly 295 mA h g$^{-1}$, typical of graphite electrodes and close to the nominal capacity of 330 mA h g$^{-1}$. Cell capacity subsequently fades after cycle 15. Coulombic efficiency values, shown in Fig. 3(b), match capacity retention values (99.9% per
cycle) over cycles 5 - 29 and are within experimental uncertainty of the limits of our multichannel charge / discharge system (Fleischauer et al., 2019a). Potential vs. capacity curves are provided in Fig. 3(c). The first insertion looks slightly different, as expected; subsequent curves overlap and are highly reproducible. It is important to note that our operando Conflat cell with Copper gaskets performs at least as well as coin cells and other reported Conflat cells near room temperature.

The use of Conflat flanges combined with a Copper gasket also allow operation of the cell at high temperatures (Periyapperuma et al., 2014; Fleischauer et al., 2019b). High temperature operation is important for applications like Measurement While Drilling (>100°C) (Guidotti et al., 2004), sensing in harsh environments (Hu et al., 2011), automotive (-40 - 60°C) (Zeng et al., 2019), and can also unlock new Li-rich phases that are inaccessable at ambient temperatures (Ghavidel et al., 2019). For the high temperature testing, the operando Conflat cell was assembled with Lithium and Aluminum electrodes. 18 mm diameter Al discs were punched from 13 µm thick, 99% pure Al foil (McMaster-Carr, Aurora, Ohio, USA) and brushed on both sides, a fibreglass separator and 1 M LiN(SO$_2$CF$_3$)$_2$ dissolved in 1:1 vol.:vol. ethylene carbonate : propylene carbonate electrolyte (99.9%, max. 20 ppm H$_2$O, Solvionic, Toulouse, France) were used. Galvanostatic lithium insertion was performed between 1.2 V and 1 mV vs. Li/Li$^+$ using 0.04 mA constant current (approximately C/600) at 110°C over the course of 37 days. Cell potential vs. capacity data during discharge is provided in Fig. 4 together with data from a regular Conflat-style cell in similar conditions.

![Figure 4](image)

**Figure 4**
Potential vs. capacity curve of the lithiation of Aluminum foil electrodes using a standard Conflat cell at 120°C (dashed blue) and the operando cell described here at 110°C (solid black). Both cells were subjected to a constant current of 0.04 mA.

The plateaus in the potential correspond to the regions of phase coexistence (Ghavidel et al., 2019). The cell was discharged only once, as aluminum cells cannot usually sustain prolonged cycling due to large and uneven volume changes with co-existing phases. Cell stack pressure was sufficient to realize a cell capacity of 2500 mA h g$^{-1}$ during Lithium insertion, in good agreement with standard cells (Ghavidel et al., 2019).

After both ambient and high temperature electrochemical cycling, the cell was used several more times at different temperatures with no apparent issues with only slight cracking of the steel reinforced JB Weld epoxy. After about ten operando experiments the cracked epoxy was removed and a new layer was added, prolonging the life of the cell. The Beryllium window had no cracks and stayed intact. It is important to note the limiting factor of repeated replacement of the epoxy is safe handling of the Beryllium window. We estimate that our cell can last at least ten experiments resulting in a cost of approximately 30 USD per experiment. With a more flexible high temperature epoxy, the cell might last even longer.

5. **In-situ x-ray powder diffraction**

Operando measurements were performed on a Bruker D8 diffractometer with a Copper source and a Vantec 500 two-dimensional detector. Separate cells were assembled based on Graphite and Aluminum electrodes to correspond with the experiments described earlier. The assembled cells were placed on top of the cooling platform on the diffractometer stage and wrapped with a flexible resistive heating element and a layer of fiberglass insulation on the sides and the bottom, as shown in Fig. 5.

![Figure 5](image)

**Figure 5**
Assembled operando cell mounted on the goniometer of a Bruker D8 diffractometer. Connections to the potentiostat (positive (red) and negative (blue) terminals), temperature controller (brown thermocouple, black heater power), and cooling water (clear) are shown.

A thin plate of glass-infused PTFE between the cell and cooling plate acted as an alignment plate and a thermal and electrical insulator. Chilled water was circulated through the cooling plate to prevent the overheating of the diffractometer stage. A thermocouple and the positive terminal were attached to the longer bolts on top of the cell. The negative terminal was attached to the feedthrough terminal at the bottom of the cell. The temperature at the position of the test electrode was calibrated using
a Pt1000 resistance temperature detector (Digikey, Thief River Falls, Minnesota, USA) embedded in a ceramic washer. Cell temperature was maintained within 0.5°C of the temperature setpoint using a lab-built temperature controller. The cell was connected using a four wire scheme to a Biologic SP-200 potentiostat operated outside the diffractometer enclosure. We have continuously measured diffraction patterns while discharging and subsequently charging the cell. Each diffraction pattern took approximately 1 hour to acquire.

![Diagram](Image)

**Figure 6**

Obtained *operando* X-ray diffraction patterns plotted as the colormap for (a) graphite electrode at 30°C and (b) Aluminum electrode at 117°C. Electrochemical cycling conditions are described in the text. The dashed red lines mark the formation of phases in the Al-Li system or the stages of intercalation in the C-Li system and are described in the text.

The obtained patterns are combined and plotted as a logarithmic-scale colormap together with the measured cell potential in Fig. 6(a) for Graphite and Fig. 6(b) for Aluminum electrodes. A 50 µm thick CMS Graphite coated on a 9 µm Copper foil was used as the electrode, therefore many of the peaks were overlapping either with the Beryllium or Copper peaks. The advantage of a Beryllium window is its low X-ray absorption, as a result, we were able to measure test electrodes that were overlapping either with the Beryllium or Copper peaks.

For the C-Li system in Fig. 6(a), only the C-Li peaks of interest are shown in the 2θ ranges of 20° – 30°, where C (002) and subsequently LiC₆ (001) peaks are located (Drüe et al., 2017), and 75° – 85°, where Li and LiC₆ (110) peaks are located. The C-Li system has been extensively studied (Zanini et al., 1978; Kambe et al., 1979; Guerard & Herold, 1975; Drüe et al., 2017; Fong et al., 1990; Billaud & Henry, 2002; Yao et al., 2004; Etacheri et al., 2011) and boasts a plethora of phases that are hard to define due to the Lithium intercalation process. Lithium intercalates into the graphite structure between the graphene sheets and the highest amount of Lithium that can be intercalated is one Lithium atom per six carbon atoms, forming a most well defined phase of LiC₆ (Migge et al. 2004). The less defined phases forming during intercalation have been reported as LiC₁₂, LiC₁₀, LiC₄, LiC₂, LiC₂, LiC₃, LiC₆ and LiC₆₆ (Fong et al., 1990; Billaud & Henry, 2002; Yao et al., 2004; Etacheri et al., 2011). From the voltage profile in Fig. 6(a), four intercalation stages can be discerned and are marked with dashed red lines. Each subsequent stage contains more and more Li atoms between the graphene sheets, ending in Stage 1 or LiC₆ phase with alternating Li and C layers (Kaskhedikar & Maier, 2009; Drüe et al., 2017).

Al-Li phases are not strictly formed via the intercalation mechanism, therefore can be well defined both from the voltage profile and the diffraction patterns. A free-standing Aluminum foil was used, placed directly beneath the Beryllium window. In Fig. 6(b), the solid vertical lines that do not change their intensity with time, at approximately 42°, 52° and 71°, are the peaks from the Beryllium window and the rest of the peaks that change their intensity and position with time can be attributed to the various phases of the Al-Li system that have been reported in our previous *ex-situ* studies (Ghavidel et al., 2019) and are: β-AlLi, Al₂Li₃, AlLi₂ (sometimes also called AlLi₂₋₋) and Al₄Li₉. The cell was discharged down to 0 V and held at 0 V for 3 hours before charging the cell back to 1.2 V vs. Li/Li⁺. The phase formation is reversible and a pure Al metal could be recovered when the cell was completely charged. In both *operando* experiments, the cell performed very well and high quality data was obtained. Each experiment lasted for about three days and the cell was rebuilt several times for subsequent measurements.

We would like to point out that, although not tested, the cell can be used in a similar reflection geometry for X-ray spectroscopy experiments, for example X-ray fluorescent spectroscopy (van Bokhoven et al., 2006) or X-ray fluorescent microscopy (Falcone et al., 2011).

6. Conclusions

A versatile and easy to manufacture and assemble, re-usable, low-cost electrochemical *operando* cell for X-ray diffraction was developed and tested for mechanical and electrochemical performance using two types of electrodes. Standard graphite-on-copper electrodes demonstrated excellent capacity retention and coulombic efficiency at ambient temperature. Free-standing Aluminum electrodes lithiated at high temperatures exhibited the same phase transformations, cell potentials and capacities as baseline electrochemical cells. Cell component manufacturing / acquisition, assembly, and operations are well suited for low cost acquisition and reliable operation.
Acknowledgements

Funding for this work was provided by the National Research Council Canada / University of Alberta Nanotechnology Initiative. We are grateful to Steve Launspach for his support with X-ray diffraction measurements, and Martin Cloutier and Jason Pitters for helpful discussions.

References


ECS Interface


