



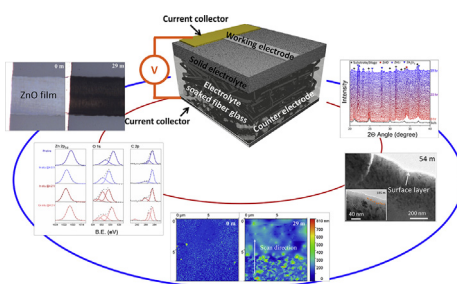
Short communication

A pseudo-solid-state cell for multiplatform *in situ* and *operando* characterization of Li-ion electrodesLin Feng^{a,*}, Zhijie Chen^a, Ruqi Chen^b, Shen J. Dillon^a^a University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA^b Center of Experimental Teaching for Common Basic Courses, South Agricultural University, Guangzhou, 510642, China

HIGHLIGHTS

- A novel and simple pseudo-solid-state Li ion battery platform has been designed.
- *In situ* TEM, AFM, SEM, XPS, and optical imaging are obtained from a ZnO anode.
- New insights gained into the structural and chemical changes in ZnO during cycling.
- Changes in electrical properties are related to the microstructure evolution.

GRAPHICAL ABSTRACT



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ABSTRACT

In the last decade, reacting Li ion electrodes *in situ* using vacuum based methods such as transmission electron microscopy (TEM) has become prevalent for assessing their reaction pathways. However, the vast majority of these studies do not perform electrochemical reactions at potentials relevant to batteries and/or characterize electrolytic reactions. Here we demonstrate a simple and flexible approach combining the benefits of solid and liquid electrolytes to enable diverse *in situ* characterization methods, including optical imaging, electrical measurements, X-ray photoelectron spectroscopy, X-ray diffraction, atomic force microscopy, and transmission electron microscopy. This work demonstrates these methods applied to the novel cell during electrolytic lithiation of the conversion anode ZnO at electrochemically relevant potentials.

Due to their sensitivity to environmental exposure and the importance of sample history in affecting reaction pathways, structural and analytical characterization of Li ion electrodes should ideally be performed *in situ* or *operando*. This fact has long been understood with *operando* X-ray diffraction being employed to study Li primary batteries as early as the 1970's [1]. However, rapid growth in the development and application of advanced *in situ* Li ion battery characterization techniques has primarily occurred in the last decade or so [2,3]. A variety of X-ray, optical, electron, and neutron based scattering, absorption, and spectroscopy approaches, as well as scan probe and

magnetic techniques, have been applied to *in situ* and *operando* Li ion characterization [4–7]. The topic has been the subject of several recent review articles that thoroughly discuss the state-of-the-art [3,8–10]. Techniques whose stimuli and resultant signal have long mean free paths can be applied relatively simply to electrochemical cells that are only slightly modified from standard laboratory or commercial formats. This explains why *in situ* X-ray diffraction was already being employed prior to the advent of commercial Li ion cells [1]. However, many techniques based on stimuli or signals with short mean free paths, such as low energy electrons, require the development of specialized

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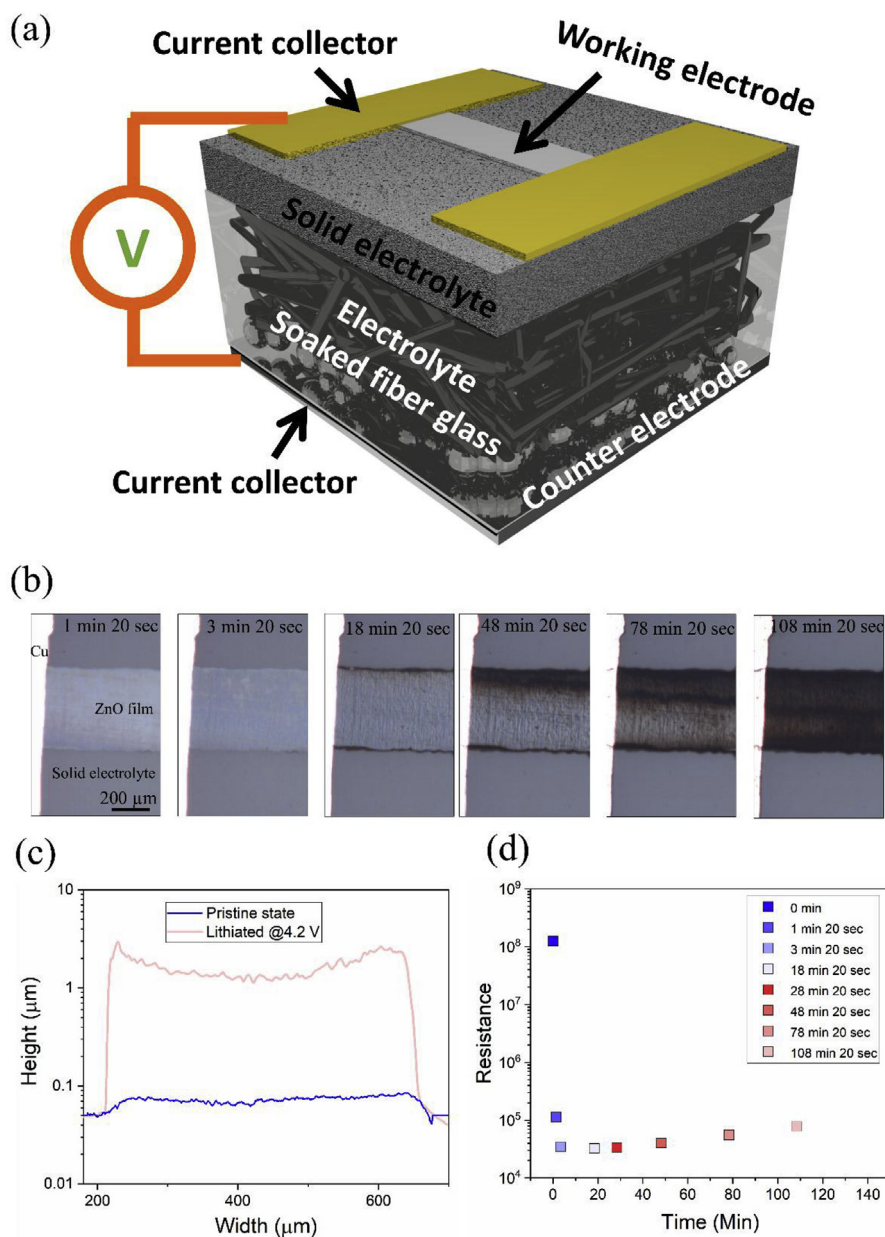


Fig. 1. (A) Schematic of pseudo-solid-state cell configuration; (b) *Operando* optical micrographs of ZnO lithiation at 4.2 V, along with (c) profilometry measured before and after the reaction, and (d) electrical conductivity measured intermittently.

electrochemical cells. For these reasons, *in situ* electron source and signal based approaches have primarily been developed in the past decade [2,11–16]. Unfortunately, many so-called “open cells” (i.e. reacting in vacuum) popular for electron-based characterization rely on electrochemically problematic cell designs. For example, > 100 *in situ* transmission electron microscopy (TEM) studies of Li induced reactions in vacuum have been reported in recent years [3], but most utilize galvanic reactions, often just direct chemical reactions with Li metal, and only a few demonstrate reactions at electrochemical potentials consistent with standard reactions or electrolytic reactions. Such studies often utilize overpotentials on the order of 3 V–5 V (e.g. –3 V to –5 V versus Li metal or versus a cathode such as LiCoO₂) to drive the reaction; see the following review for several examples [2]. These potentials are not electrochemically meaningful to Li ion batteries. This fact is important because the overpotential can affect the reaction pathway of the system. When measurements are made at poorly defined overpotentials it is unclear how the results relate to electrochemical batteries.

Solving scientific and engineering problems typically requires data from multiple sources and techniques. However, specialized electrochemical cells developed for one apparatus are often not amenable to use in different instrumentation. Experiments should be performed under similar electrochemical conditions for direct comparison, since reaction pathways can be sensitive to overpotential. Solid-state batteries are an ideal solution, since they can be cycled in vacuum, atmosphere, or submerged in liquids, they function well electrochemically, and can be characterized in almost any structural or analytical characterization equipment [17]. Certain solid electrolytes are less sensitive to X-ray and electron irradiation than liquid electrolyte. Reactions between liquid electrolyte and electrons or X-rays severely limited our prior *in situ* X-ray photoelectron spectroscopy (XPS) and TEM experiments [11,18,19]. Unfortunately, solid-state batteries are notoriously difficult and time consuming to fabricate, particularly outside of dedicated laboratories [20]. Here we seek to develop and demonstrate a flexible *in situ* Li ion cell amenable to multipatform characterization that can be constructed simply using commercially

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