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The technical and educational requirements in establishing a Li-ion coin-cell assembly and testing research facility laboratory in a university environment

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Abstract:

Li-ion batteries are now the main rechargeable electrochemical energy storage source in systems ranging from small portable electric-powered devices to autonomous robots and hybrid and all-electric plug-in vehicles. As increased production demands accelerate there is a corresponding challenge, particularly in North America, for universities to provide knowledgeable engineering technical talent to support the related Li-ion cell research. In the work presented here the authors review the major components of their Li-ion cell assembly and testing research laboratory, and the contributions that student research assistants provided in the effort to establish our Li-ion cell assembly and testing lab from its inception, equipment acquisition and set-up, to operation, commissioning and use for research activities at our university. The utilization of related research tools already available at the university are also discussed. Both undergraduate and graduate students share their experiences here in the process of learning and conducting Li-ion cell production and cell cycle testing. Finally, a list of suggested supporting classes and training is provided for other institutions interested in establishing such a laboratory.

1) Introduction:

Lawrence Technological University (LTU) established a Li-ion cell assembly and testing research laboratory in collaboration with Intecells, Inc., a start-up Li-ion battery equipment processing company. This collaboration started in November of 2019, and continued into 2023. This laboratory is an active on-campus research laboratory, and is currently not used for academic activities. No classes are taught at this time in this lab. Formal education classes may be possible with this laboratory in the future as additional funding is secured. However, limited funding does not currently permit this. Intecells, Inc. needed technical talent to support Li-ion cell processing, assembly and testing, and also needed engineering level skilled workers familiar with their electrode processes to join their operations. During this collaboration Intecells, Inc. provided equipment and production hardware to both assemble, and to conduct electrical charge and discharge cycle performance testing of Li-ion coin-cells (CR2032). Collaboration with LTU also included assessing electrode powders and deposited electrodes on substrate using the LTU scanning electron microscope, coupled with Energy Dispersive Spectroscopy (EDS) analysis to characterize electrode chemistries, blends and particle sizes.

By early 2022 a cell assembly lab on the LTU campus was prepared with upgrades to the room security and climate control. A glovebox using argon as the internal inert environmental gas, with ppm O_2 and H_2O monitoring capabilities was installed. Coin-cell electrode punching dies, a precision balance, cell assembly tools, micro-pipetting, and cell crimping capabilities were set up for use in the lab. A multi-channel cell cycling station was installed in the early spring of 2022 and full assembly and cycle-testing operations began in the late spring of 2022.

Four LTU engineering students (two undergraduate students, and two graduate students) were employed as research technicians for this project. Two LTU faculty members also supported this work. Li-ion materials handling, cell assembly, lab techniques, and cell cycling test methods presented an ongoing need to extensively train and educate the students in the electrochemistry theory, related laboratory techniques and safety requirements.

Since the initiation of this project the authors have had numerous informal conversations with faculty at other academic institutions, as well as Li-ion cell and battery manufacturers, automotive battery suppliers, and electric vehicle manufacturers. From these conversations it became evident there is a serious shortage of knowledgeable, and trained engineering talent in the entire Li-ion battery process ranging from pure research, through cell manufacturing, to battery pack and module assembly, and ultimately to end user applications. Frequently these other faculty expressed and interest to expand their educational efforts in this area, but do not have a clear understanding of how to do so. This paper attempts to document some of those key elements in the efforts to create, organize and carry out the work efforts required for the successful implementation of this Li-ion cell assembly, testing and characterization effort.

2) Background:

Between 1976 and 1986, John B. Goodenough developed the theoretical basis and basic operational characteristics of a Li-ion battery. Later in the 1980s, he comprehensively developed the Li-ion battery [2]. Li-ion battery technology was quickly embraced and in 1991 Sony Co. employed the first Li-ion battery system in a commercial product [3]. Since the early 2000's there has been a dramatic increase in the global demand of Li-ion batteries because they possess significantly higher electric energy storage capacity over other electrochemical cells. They are a superior secondary battery (rechargeable) electric energy storage system, and are now the main rechargeable energy storage source in devices ranging from small portable electric-powered devices to virtually all electric vehicles.

In the early 2000's notable manufacturing capacity resided in the United States, but by 2010 the demand for Li-ion cell production was almost exclusively met by off-shore, sole-source countries due to low-cost manufacturing. By 2009 concerns were being voiced regarding the fact that most Li-ion manufacturing was indeed located predominantly in Asia [4]. At that time the top three Li-ion battery manufacturers were China: 25% of the global capacity, South Korea: 27% of the global capacity, and Japan: 46% of the global manufacturing capacity. Also at that time the US Department of Energy started asking critical questions regarding the viability of this situation for US competitiveness [4], and how the DOE (U.S. Department of Energy) could play a role in energy innovation. By 2021 the list of the ten largest Li-ion battery manufacturing countries had changed, but the owners of those manufacturing facilities remained in China, Korea and Japan [5]. Today Chinese firms now dominate the electric vehicle (EV) battery market. See Table 1, below.

The COVID-19 pandemic significantly interrupted supply chain distributions from simple commercial products up to critical items. COVID-19 lockdowns created what has been termed a “perfect storm” of supply chain difficulties, which including shifts in demand, inconsistent labor shortages and profound structural factors. Adding the Russia-Ukraine conflict amid COVID-19 lockdowns in Asia exacerbated supply disruptions in consumer goods, metals, food, chemicals and commodities [6]. Now, in the post-pandemic era there is a documented need to reduce supply-chain disruptions and to increase local and domestic production competitiveness and manufacturing of Li-ion batteries in home countries in North America, Europe and the Pacific regions in order to meet user demands [7]. In the United States this is now formalized in approaches such as "National Blueprint for Lithium Batteries (2021-2030)", and in the United States Federal Government via legislation, the 2022 United States "Defense Production Act", and the "Mining Innovations for Negative Emissions Resource Recovery Program". So urgent is the perceived need that in early 2022, the US departments of Energy, Defense and State drafted a "Memorandum of Agreement" to begin stockpiling minerals for batteries and wind turbines, to meet national security needs [8]. There is a growing awareness of Li-ion battery users of their need to diversify and localize their battery cell supplier base.

Table 1: Top Ten Li-ion Manufacturing Countries

Rank	Country	Share of global lithium-ion battery manufacturing capacity
10	Australia	0.1%
9	United Kingdom	0.3%
8	Sweden	0.6%
7	Germany	1.6%
6	Japan	2.4%
5	South Korea	2.5%
4	Poland	3.1%
3	Hungary	4.0%
2	United States	6.2%
1	China	79%

With the growth and increased production driven by Li-ion battery user demands, there is a corresponding challenge, particularly in North America, for universities to provide knowledgeable engineering technical talent related Li-ion cell assembly/production, and testing needs. The engineering skills go beyond those provided by a typical engineering education. This critical role must, be met by North American universities. The work presented here reviews our approach in one way to possibly help meet those technical skill needs.

3) Producing Lithium Ion Cells and Batteries – More Than Just Electrochemistry

Establishing a Li-ion cell assembly and testing laboratory requires an array of tools, equipment, and workers possessing the required skill sets. Some of the basic equipment needs, such as precision balances, ultrasonic cleaners, small drying ovens, or vacuum drying ovens, glassware, lint-free wipes, etc., are commonly found in most academic chemistry or analytic labs. Conducting typical analytical assessment of Li-ion cell testing, however, requires analytical equipment not common in most undergraduate engineering labs. We needed a high-temperature box furnace, a multi-channel cell cycler, a controlled environment glove box with an argon gas delivery system to the glovebox, a high throughput vacuum pump for antechamber pump-down, and also the regular use of a scanning electron microscope (SEM).

We employed both undergraduate and graduate engineering student research assistants. These students required a collection of technical skills, some of which were only part of their traditional engineering education. They needed to learn about inert-gas controlled-environment systems, particularly low parts-per-million O_2 and H_2O environments. They also had to learn about clean handling and contamination prevention techniques that significantly exceeded common "clean" procedures. There were also numerous laboratory techniques and skills needed that are not typically emphasized in any undergraduate science or engineering laboratory.

A method of identifying, developing and documenting all critical standard operating procedures (SOPs) had to be created. All coin-cell assembly processes had to be documented, and formulated into an accepted written SOP. The SOP methods assured cell assembly consistency, cell repeatability and quality assurance for cell batch-to-batch assembly between student workers over multiple weeks of processing. Procedures were also documented for argon gas bottle connection to the glovebox to assure safe handling and hook-up activities. A library of Data Safety Sheets, or SDSs was also established for all materials used within the lab and in the cell assembly processes. We reviewed all SDSs with student researchers.

4) Critical Analytical Tools Used

A Li-ion cell assembly laboratory, requires common, and often standard laboratory supplies. But because cleanliness and contamination-free lab practices are also needed, latex powder-free gloves in various sizes are also required, and for cell handling, positioning and orienting within the cell crimping press non-conductive plastic tweezers are required. Non-conductive surfaces for cell assembly, cell transport and cell storage are required. One-cell lot sizes required tracking individual cells (sometimes after making dozens of cells at a time) for handling, storage and test cycling.

Li-metal-oxide electrode powders prior to electrode deposition were batch calcined at controlled ramping rates to temperatures between 500^{o}C and 1000^{o}C for a few hours order to drive off volatile matter using a Lindberg/Blue M Box Furnace temperature-controlled oven. We also used an Arbin Instruments LBT-21084 (48-channel) cell cycle for cell testing. The glovebox used for this project for cell assembly was a Vacuum Atmospheres double-sided, model HE 243-2 (donated to the university from AMPS Corp (Ann Arbor, MI). Our glovebox has accesses for two simultaneous workers, directly facing each other. We found this to be very helpful where two people working together could help move and position heavy objects (such as the coin-cell press) within, or out of the glovebox. We found that general visual inspection using an optical microscope with 10x to 80x magnification capabilities was sufficient. However, when more extensive surface and electrode morphology analysis was required, we used a scanning electron microscope available on our campus.

For much of our work on this project at LTU it was essential to use our FEI Quanta 200 Environmental Scanning Electron Microscope (ESEM) managed and supported by the College of Engineering's Biomedical Engineering department. See Figure 1 below. We used the High Vacuum mode for samples that are conductive or coated for conduction. The LTU ESEM was originally funded from an NSF Major Research Instrumentation (MRI) award (Award Number:1040607). The LTU ESEM has an image magnification range from 25 times (25x) up to

300,000 times (300,000x) magnification. Typical ranges for our work was in the 500x to 30,000x magnification. Our ESEM is coupled with an EDX detector that can be used for both qualitative and quantitative analysis, enabling users to identify both the type of elements that are present as well as the percentage of each element's concentration within the sample.

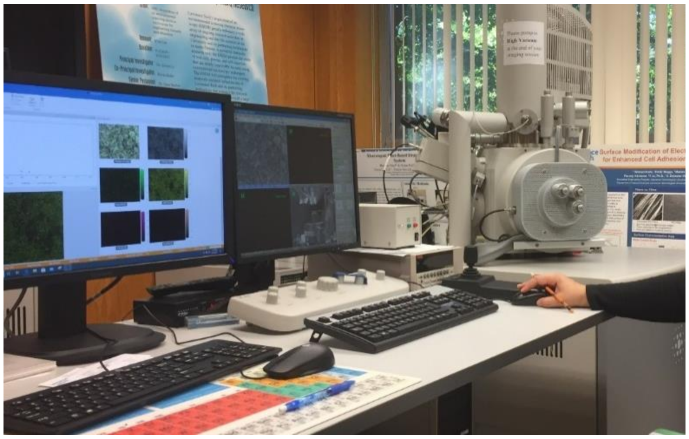


Figure 1: A project team member operating the LTU ESEM and doing EDS analysis.

5) Coin Cell Assembly

The focus of our lab was the preparation, assembly and testing of CR2032 coin cells. These cells were chosen because they are small, easy to assemble, and their parts are readily available and relatively inexpensive. Inside the CR2032 coin cell electrodes have conveniently small electrode areas, resulting in low watt-hour capacities. This yields shorter charge and discharge times and quicker turnarounds for our research. Having an overall small coin cell size also limits chemical safety risks with the electrolyte, and any other chemicals used.

The equipment used after obtaining the electrodes begins with an electrode disk punched to size (14 mm) and a separator punched to size (19mm). Figure 2 below illustrates the various disk punches used for separator and electrode disc production. A precision analytical balance was used to obtain accurate and precise weights for each electrode (see Figure 3 below). Coin cell components include a cathode cap, electrode, separator material, gasket, spacer disk, wave spring, anode cap, and electrolyte. Coin-cell assembly took place within a glovebox filled with argon as the inert interior glovebox gas.

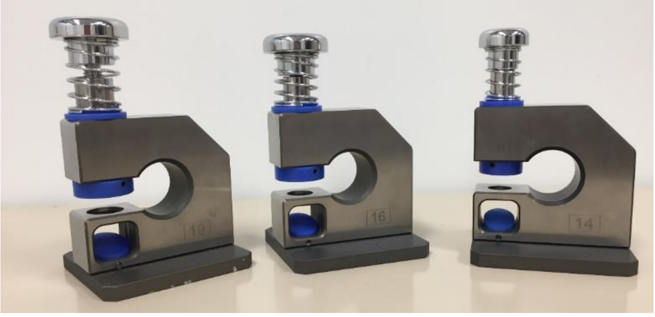
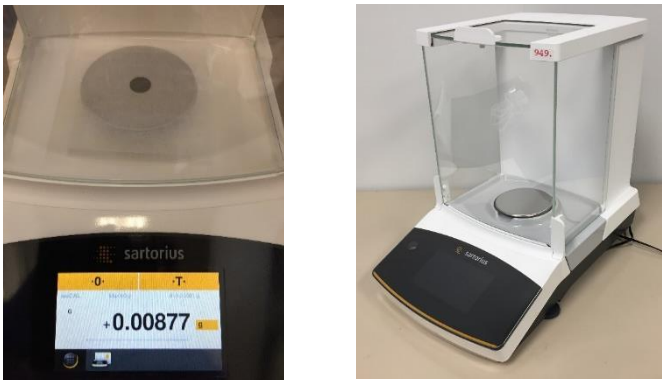


Figure 2: The manual 14 mm, 16 mm and 19 mm dies for punching electrode disks.



(a) (b)

Figure 3: An electrode disk weighed (a), and the balance used (b).

Preparation for the coin cell components begins with punching out the electrodes using the disk die-punch. Once punched, the electrode disc is then weighed, its weight is recorded, and then set into its designated numbered cup in a nonstick cupcake tray. This allows tracking each individual electrode. Electrode weight is used to calculate its specific theoretical capacity and “C-rating" of each disk. The weight of each electrode may vary, so each disc must be treated as an individual "lot-size of one". Coin cell components; cathode cap, gasket, spacer disk, wave spring, and anode cap were all ultrasonically cleaned in reagent-grade isopropyl alcohol and then vacuum oven-dried. Cell assembly takes place within the glovebox. See Figure 4 illustrating the glovebox. The antechamber is pumped down to a full vacuum of 29.9 inches Hg, and remains at vacuum for five minutes. Then the chamber is let up to atmospheric pressure with argon gas and pumped back down again for seven minutes. This is repeated to assure the O_2 and moisture levels within the antechamber are essentially at zero ppm. O_2 and moisture levels were monitored continuously in the glovebox using a digital gas monitor with sensors located within the glovebox. The antechamber is purged and filled with argon gas allowing the door isolating the glovebox and the antechamber to be safely opened.

Figure 4: The LTU glovebox with vacuum pump in the lower right of the photo.

For in-glovebox assembly, additional latex gloves are placed over the glovebox's gloves to ensure cleanliness. Each solid part is placed into a section of a clean plastic ice cube tray for organization and easy access. Plastic non-conducting tweezers were used for cell assembly to prevent accidental short circuiting the cell during assembly. Components are assembled in order and a manual micro-pipettor is used for dispensing appropriate controlled volume drops of electrolyte, as required per cell. See Figure 5-a below. Once metal cup, electrode, electrolyte, separator, gasket, anode, spacer and anode cap are placed and aligned, the assembly is lightly pressed together. Figure 6 below illustrates various sizes and composition of cells. The un-crimped coin cell assembly is then carefully placed inside the press and manually crimped to form the sealed cell assembly encapsulating all the components. See Figure 5-b below. Dust/lint-free lab wipes are used to remove any excess electrolyte while still within the argon-filled VAC Atmospheres glovebox. The completed coin cell is taken from the press and wiped with a lint-free paper wipe, visually inspected, and then removed from the glovebox for cycle testing.

(a) (b)

Figure 5: Assembling cells (a), and crimping to sealed cell (b).

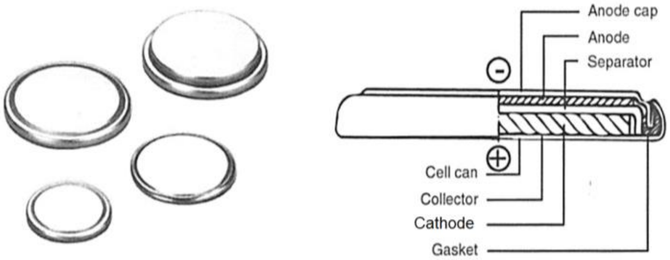


Figure 6: Assembly of a typical coin cell [9].

6) Cell Cycle Testing

An Arbin Instruments LBT-21084 (48-channel) cell cycling station with custom software for prescribed cycle testing was used for all cell performance testing. See Figure 7 below. This cycler had 48 independent cell testing stations and also has Electrochemical Impedance Spectroscopy (EIS) capabilities. The cycler has limits of 5 volts and 5 amps per station, but is never an issue as we were testing cells with currents as low as 10^{-3} amperes for a 1.0 C-rate and max and minimum voltages ranging from 2 volts to 4.5 volts. Testing currents are determined from active material weight of the cathode to yield the C-rate used for cycling.


(a) (b)

Figure 7: The front of the cell-cycler (a) with computer control and data review screen, and (b) the individual channel clips for holding individual coin cells.

6.1) Cycler programming and testing script development

The testing script of the cycler was developed over time as the needs of the tests changed and the program system was better understood. As more advanced testing was required the code script changed to accommodate testing demands and became more streamline in terms of number of cycles. A sample of the script is as follows, but many variations were also developed depending upon testing needs:

- a constant-current charge at a specific C-rate to maximum voltage
- a constant voltage charge until a minimum of half the specific C-rate used
- a rest-period
- discharge at constant current
- rest period
- charging cell

The rest periods were based on a time limit. Additionally, these tests were usually cycled at C/10 for 12 cycles, C/5 for 5 cycles and C/3 for 3 cycles depending on testing demands. The cell cycler is also capable of conducting EIS using additional equipment provided. This testing option was added after each charge/discharge cycle at relevant ranges of frequency and voltage. The value of this EIS testing is it provides an understand of the internal resistance of the cell.

6.2) Challenge of each cell as a "lot-size-of-one"

Due to need to track each individual coin cell produced it is necessary to treat each cell produced and tested as a "lot-size-of-one". This is a challenge because of the uniqueness of each electrode, as each individual cell tested has to have its own test program with its unique current manually entered into each cell test program. Sufficient organization and handling are needed to trace all testing and results to each specific cell for additional testing or disassembly. To accomplish this a spreadsheet is made with the sample name and each individually punched out cathode labeled. Each punched cathode disk is weighed and using the provided active ingredient and the theoretical specific capacity the nominal capacity and needed C-rates are calculated and recorded for future use in the testing script for each cell. Once the cells have been assembled, cleaned, and labeled they are then loaded onto the cycler, the channel number is recorded and the name of each individual script reflects the sample number. The cell cycling tests are then started. These test durations vary based on the performance of the cell and the type of test run which results in some running for less than 24 hours while others take almost 2 weeks. Once testing is completed the data is exported for analysis.

7) Summary of Process and Engineering Knowledge Gained

Typically engineering students learn broad aspects of general engineering areas in academic laboratories. Student learning about Li-ion cells or production methods tend to be specific and rather limited. Rarely do engineering students see the entire process from start to finish for coin cells [10] - [13]. The students working on this project, however, are research assistants and this is not an academic lab environment for academic credits, so they gain knowledge across areas not typically covered in undergraduate or graduate engineering classes.

These students require supplemental lab safety training regarding chemicals, potential gaseous vapors, and electricity. Students learn how to handle atmospheric/moisture-sensitive chemicals like the electrolyte and lithium. Students learn how to replace tanks of argon gas suppling the glovebox. The electrical skills needed are previewed and reaffirmed during assembly and during testing.

Lastly, students learn how to create, write and utilize Standardized Operating Procedures (SOPs). They are able to understand contend needs, and how to create multiple SOPs for all equipment used. This is done by assigning a student a specific piece of equipment, to think thoroughly about every possible safety measure, the chemicals used, supplemental supporting equipment used, all consumables used, and to then document the procedure steps. Once completed on their own, the students collaborate to double-check that nothing was missing. Then each SOP was cross-checked by a different student to assure the process steps were clear.

8) The Student Perspective: Student Documented Feedback

As a matter of routine, LTU students document their experiences and what they personally gained from research projects. Written below are student responses to a series of questions. The questions are listed below with student responses written in italics.

- What were your concerns about joining such a project?

"My concern was to not be overwhelmed by the amount of new information that I was receiving. There was a lot of new information but it was interesting and engineering knowledge from Battery and energy storage as well circuits and electronics helped tie the new information together."

"My concerns were not being far enough along with my engineering-based classes. I was worried I would not be able to understand what to do or how to do tasks."

- What areas did you feel your engineering education was deficient once you started working in the Li-ion cell development environment?

"I felt deficient in the requirements for testing cells and understanding the results of that information. The industry partner had to explain what the script should have for testing and whether the results were good or bad."

"Making cycler programs and analyzing the data was the hardest without any knowledge of programming or circuits and electronics."

- What were the things most challenging to learn?

"Assembly and programming of the Arbin cycler. There was little guidance in the assembly and programming of the unit beside the manual."

"One thing that was challenging to learn was what electrochemical impedance spectroscopy is and how to be able to integrate it into our normal charge and discharge cycling program."

"Another thing that was hard to learn was dexterity skills (in the glovebox). Using three pairs of gloves, with the middle set being very thick while using fine-tipped tweezers to pick up very tiny parts was challenging to get used to. I personally have very small hands which made it more difficult since the glovebox gloves were a set size."

- What were your most interesting things to learn?

"The different tests that were done on the cells and how that proved the cells were working as expected. Additionally, the electrochemical impedance spectroscopy was very interesting as it is still an area of confusion or multiple interpretations of results."

"One thing that was interesting to learn about was gas flow. It was cool to be able to turn on and off valves. Another thing was to see how coin cells are made and then to be able o make them. It is not often one can say I made a coin cell."

- What would you recommend that other universities developing such Li-ion battery development and testing facilities address with their engineering student technicians

"They should address the importance of lab techniques such as the importance of reducing cross-contamination, organization, and handling of materials."

"Yes, it was an awesome experience to be able to have hands-on learning about a very important topic in today's age of energy. Students will be able to gain laboratory and research skills that can be applied to any type of lab setting."

- Should there be separate courses for students working in this field? If so, what courses?

"Yes, classes specifically for batteries or cells as the general engineering classes don't provide enough information. This course should teach the manufacturing, testing, and components of cells. If these classes can also have physical labs for making cathodes and anodes and assembling cells that would be beneficial as well."

"I think so. I believe there should at least be a course or lab about analyzing electrode material, assembling cells, cycling cells, and analyzing cycled data. And even includes the deconstruction of a cell for possible recycling or analysis for side reactions and performance. This would have students see the construction of a cell from start to finish."

Due to the scope of funding available, this laboratory has been initially launched as a an active on-campus research laboratory, and currently is not used for academic class activities. No formal classes are taught in this lab. But we are, however, using this lab to identify areas where students do need additional education and background knowledge. As additional funding is received the lab will be expanded to also become an academic instructional lab.

9) Suggestions for Other Schools Developing Li-ion Cell and Battery Labs

Industries in the Li-ion battery field need engineering students with knowledge beyond basic chemistry. They also need a working knowledge of electrochemistry. Unfortunately, many engineering students do not like, nor do they enjoy their chemistry classes. The distaste for chemistry amongst students is been well-known by STEM educators and has been well documented by the chemistry instructional community [14]. Various ways to improve chemistry instruction have been proposed, but reversing the negative perception of chemistry engineering students continues to face roadblocks [15]. The work discussed here is not the place for comprehensively addressing this problem. But, this negative bias against chemistry must be addressed by the engineering education community. Future engineering workers in the Li-ion battery industry must understand chemistry. We believe those who teach chemistry at the university level must make it engaging, interesting and a subject enthusiastically embraced by engineering students. To also meet the growing need for electrochemistry knowledge-based talent there now needs to be a corresponding expansion of the electrochemistry elements of university inorganic chemistry classes, or perhaps even undergraduate class focusing only on electrochemistry.

Based on our student feedback, we found that a broader understanding of electronics is also needed by non-electrical engineering students. LTU currently requires only one undergraduate, three-credit circuits and electronics class (EEE2123) for most non-electrical engineering majors. This may need to be expanded for those students pursing engineering skills in the Li-ion battery field. This is especially true for majors such as mechanical engineering, chemical engineering and material science engineers who are often part of battery R&D departments in industry. The students participating in the research activities within our lab repeatedly voiced the need for a broader understanding of electronics. It may now be time to include some form of an across-all-majors general enrollment energy-storage technical elective class for engineering programs. Such a course need not be purely a Li-ion cell/battery class, but until a viable substitute for Li-ion cells comes along, Li-ion cell chemistries need to be a major part of such a course. Items in a Li-ion cell/battery class should include the following:

- Characteristics of electrode properties, and electrode material analysis
- Cell assembly methods
- Controlled environment and inert environment manufacturing procedures
- Cell charging and discharge cells characteristics, and power cycling of cells
- Analyzing cycled data

- Electrolyte safety and cell/battery safety, in general
- Component recycling methods

How other academic institutions implement our recommendations is up to those institutions, depending on their current academic offerings, the academic resources they have, and also the funding resources they can access. There are also other curriculum elements that should be added to current traditional academic classes typically taken be engineering students. Some changes to traditional writing and communication classes could include instructing students how to created standardized operating procedures (SOPs). Electronics courses could include battery/cell charge and discharge characteristics and how to analyze those results. Traditional lab courses could spend more time on lab safety and clean laboratory techniques.

10) Summary and Conclusions

There is a current and growing need to increase the knowledge-base of engineering students of electrochemical energy storage devices, and Li-ion cells and batteries, specifically [16]-[18]. LTU has been working towards addressing that need. Since the fall of 2019 LTU faculty and students – working as research assistants, have been in collaboration with Intecells, Inc. in the establishment of a Li-ion coin-cell assembly and testing laboratory at LTU. CR2032 cells, due to their small size and limited safety risks, were produced and tested in this lab. A review of those efforts has been presented here.

Typical laboratory equipment required for such endeavors have been listed and discussed. Several hundred Li-ion coin cells were assembled and cycle tested. Various cell charging and discharge testing script programs were written and evaluated using a cell cycler to assess optimal cell performance. Various C-rating levels and maximum and minimum voltages were used. Electrochemical Impedance Spectroscopy tests were also incorporated in to cell charge and discharge cycling.

The students involved in this project were able to learn important aspects of Li-ion cell assembly methods, cell performance, and vital laboratory skills. They gained valuable insights into cell testing and Li-ion electrochemistry. These are all necessary skills that engineering students need to bring to the Li-ion development and production industry. Such talent is absolutely needed to meet the growing demands and competitive international Li-ion marketplace [16]-[18].

The proof of these efforts is that two of the four participating students now have both internship and fulltime employment opportunities awaiting them upon the conclusion of this current academic semester. The feedback we have received is that industry is very interested in their skills.

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