Fifty years of lithium-ion batteries and what is next?

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The first rechargeable lithium batteries were built 50 years ago, at the same time as the Materials Research Society was formed. Great strides have been made since then taking a dream to domination of portable energy storage. During the past two decades, the demand for the storage of electrical energy has mushroomed both for portable applications such as the iPhone and electric vehicles and for more than 1-GWh grid applications. As storage and power demands have increased, the batteries have evolved with their chemistries being pushed to the limits. This has resulted in the energy densities almost doubling and the cost dropping by more than an order of magnitude. However, the present electrochemical systems are still too costly to penetrate major new markets, still higher performance is required, and environmentally acceptable and sustainable materials are required.

Background

The first rechargeable lithium batteries were made in 1972/1973 at Exxon's Corporate Laboratories,[1,2] at the same time that the Materials Research Society (MRS) was formed. These cells used the concept of intercalation reactions in which the guest lithium ions were intercalated into a host lattice, specifically, the layered dichalcogenides of the Group IV, V, and VI transition metals, such as titanium and tantalum. These cells could be constructed in either the charged state or in the discharged state ($Cu//LiTiS_2$). MoliEnergy then replaced the TiS_2 with MoS_2 because the latter occurs as the mineral molybdenite in British Columbia, Canada; these were commercially available for about a decade. Goodenough's team then extended the layered concept to the layered oxides, such as $LiCoO_2$.[3] They recognized that a range of related oxides such as $Li[NiMnCo]O_2$ could be used: "may be constituted by a mixture of transition-metal ions."[4] These cobalt-rich cathodes still dominate most volume-sensitive applications, such as phones and portable computers, and even the Boeing 787 plane. Today, for other applications, because of the cost and child labor issues among others in the Congo, the cobalt has been substantially replaced by nickel, manganese, and/or aluminum. These materials, commonly called NMC, NMCA, or NCA with formulas, such as $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ or $LiNi_{0.9}Co_{0.05}Al_{0.05}O_2$, have a higher energy density than $LiCoO_2$ itself for any given charging voltage. The patented commercial NMC materials[5] used today contain a slight excess of lithium with that lithium being in the phase Li_2MnO_3; it is not clear how essential this second phase is. The early history of the NMC and related cathodes has been reviewed[6] as has an overview of the field in *MRS Bulletin*[7] and will not be repeated here. The structures of some intercalation cathodes are shown in **Figure 1**.

The anode of rechargeable lithium batteries was initially pure lithium metal, but the tendency to form dendrites on charging limited its use. Exxon used a LiAl alloy anode,[8] which was effective for tens of cycles, but eventually crumbled. It was not until A. Yoshino and colleagues at Asahi Kasei Corporation developed the use of carbons that a long-lived and safe anode was available. Basu at Bell Laboratories showed that pure graphite was an effective anode for lithium.[9] It was the combination of the $LiCoO_2$ and synthetic graphitic carbon that allowed researchers at SONY to market the first commercially successful Li-ion battery in the early 1990s. Today, natural graphite is also used in some cells, because of its lower cost.
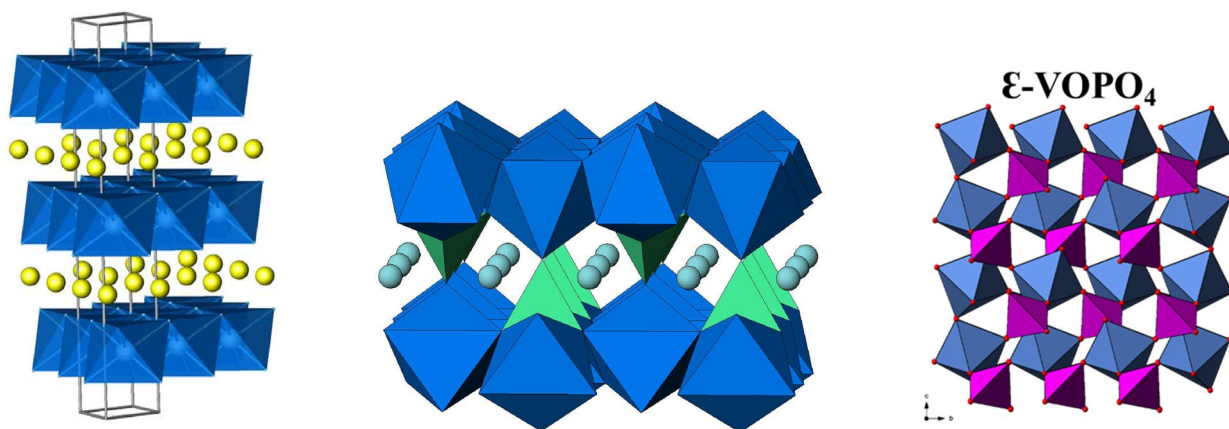
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Figure 1. Intercalation cathodes used in lithium batteries: (Left) layered structure, $LiMX_2$. Examples of M are Ti, V, Mo and S, or Se, or M = Ni, Mn, Co and X is oxygen. (Center) 1D tunnel structure of $LiFePO_4$ and (right) multidimensional structure of Li_xVOPO_4.
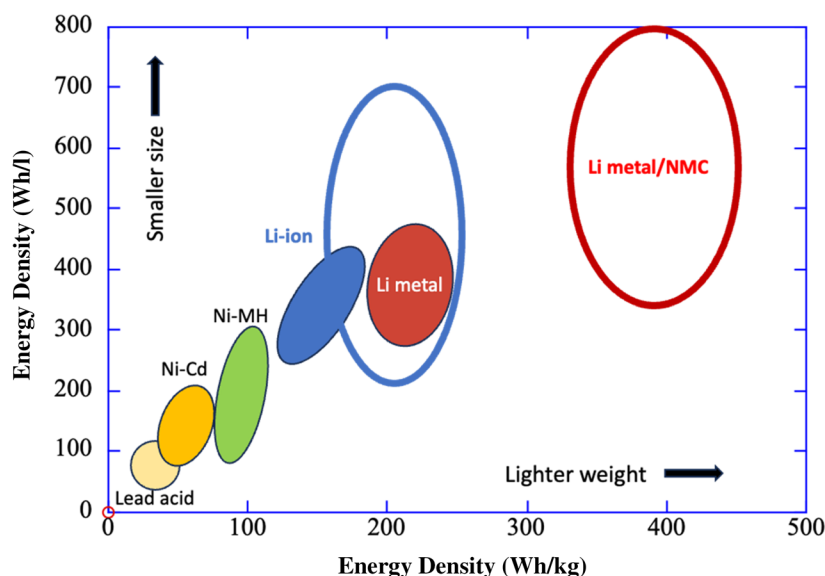
Progress on lithium batteries

Not much has changed in the chemistry of Li-ion cells in the last 30 years, but the energy density of the cells has gradually increased so that today's commercial cells attain around 250 Wh/kg and 700 Wh/l. This significant change is shown in **Figure** 2, which compares the data from 2001[10] with 2023. Another dramatic change is the cell cost which has dropped from around USD$5,000 to USD$100/kWh in large quantities.

However, if the cell energy density is to be further significantly increased then the carbon anode must be replaced, 72 g of carbon are needed to store 7 g of lithium, and the anode takes up half the volume of the cell. Thus, there is much effort targeted at a replacement with the end target being close to pure lithium. The DOE-funded Battery500 consortium has a 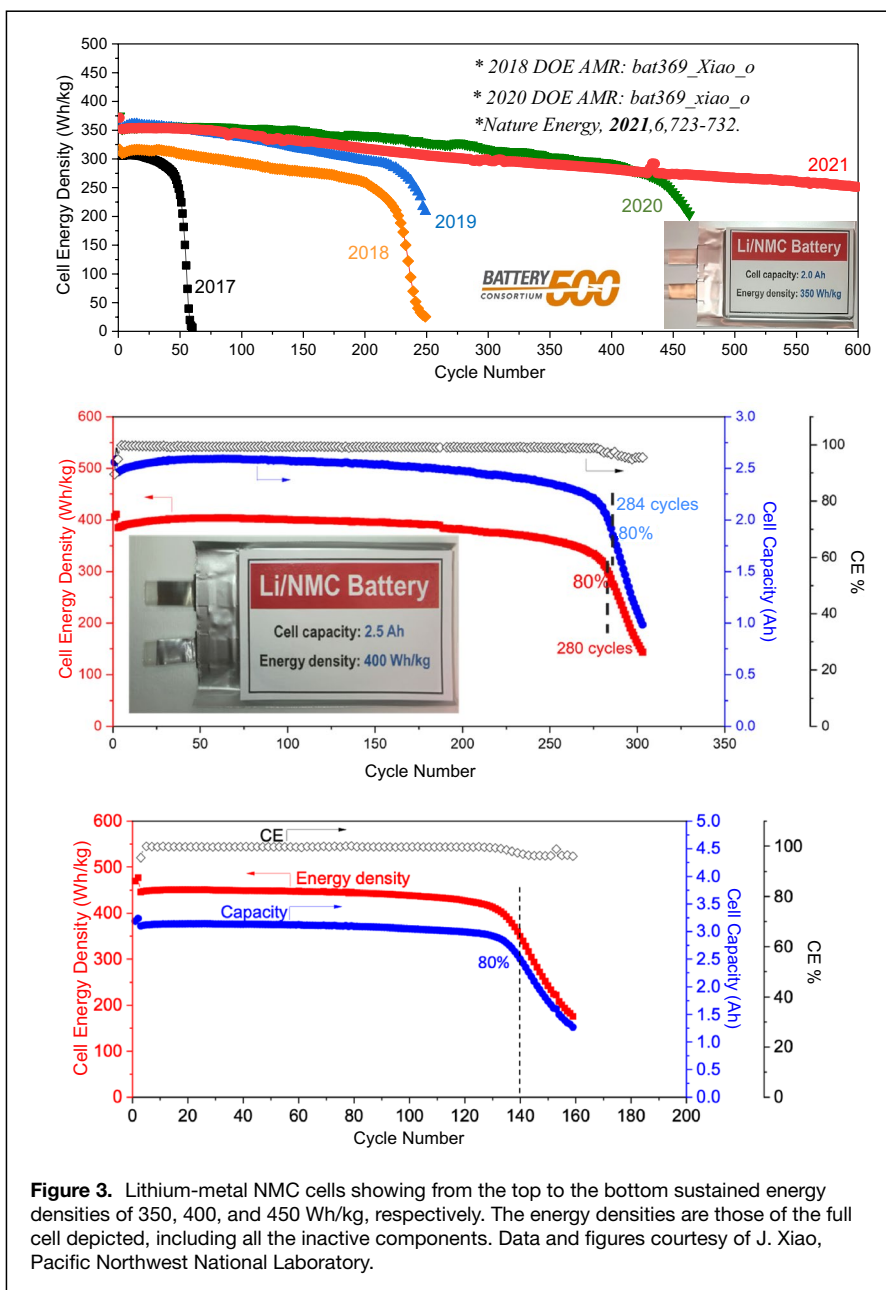goal of achieving cells with an energy density of 500 Wh/kg and approaching 1 kWh/l (these are just 50% of the theoretical energy density of the active materials alone). It is using pure lithium metal as the anode and has achieved over 600 cycles at 350 Wh/kg at the full cell level as shown in **Figure** 3 (top). Progress is being made at achieving the 500-Wh/kg goal, as also shown in the figure for cells with 400 and 450 Wh/kg. Whereas, the 350 Wh/kg was achieved over a five-year period and using the 622 NMC composition, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$,[11] and the higher energy densities used the 811 composition, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$.

Still larger energies will need perhaps even higher nickel contents; this increase in nickel content is also being driven by the desire to minimize the amount of cobalt both to reduce the cost and to ameliorate the child labor issues. Today, most effort is on the 811 composition ($LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$), but as the nickel content is increased, the lattice instability on charging increases making it more reactive with the environment and the electrolyte. This is due to their tendency to evolve oxygen. This may be mitigated by surface coatings and lattice substitution, which inhibits the diffusion of the transition-metal and oxide ions. Niobium, even at the 1% level, can allow the 90% Ni material to be deep cycled for hundreds of cycles without capacity or voltage fade.[12] Similarly, Tesla uses the NCA material, $LiNi_{0.9}Co_{0.05}Al_{0.05}O_2$, where the aluminum provides the stabilization. However, as the Ni content increases there is an increased tendency to release gas, so pouch cells may have to be replaced by hard-cased cylindrical or prismatic cells.

The phosphates such as lithium iron phosphate, $LiFePO_4$–LFP,[13] are making a comeback partly because it contains no expensive cobalt or nickel and because

Figure 2. Comparison of the energy densities of batteries reported in 2001 (closed circles) compared with commercial cylindrical cells today (open circles).

Figure 3. Lithium-metal NMC cells showing from the top to the bottom sustained energy densities of 350, 400, and 450 Wh/kg, respectively. The energy densities are those of the full cell depicted, including all the inactive components. Data and figures courtesy of J. Xiao, Pacific Northwest National Laboratory.

oxidation catalysts, toward the electrolyte must be controlled. Several vanadium oxides have been used in commercial lithium cells and continued under active study, as vanadium is the fourth most abundant transition metal.

The long-term goal is to use pure lithium anodes, but as a transition between carbon and lithium, numerous researchers are exploring the use of silicon-based anodes that can react with more than 4 Li/Si, but are highly reactive with the electrolyte. Another alternative is to use alloy-type materials such as SnCo as commercialized by SONY,[7] but clearly this is too expensive. The compound Sn_2Fe, which is much less reactive than Si, has shown extended cycling capability and high Coulombic efficiency.[17] This shows that a conversion reaction such as a tin alloy can be an effective electrode material. Whatever is used, a means to control the large volume changes of the anode compartment will be necessary. One approach could involve an "inert" open host structure.

Single crystal versus meatball NMC cathodes

The first cathode materials used in Li-ion batteries were single crystals, because their lower surface area reduced their reactivity thereby increasing the safety of the cells. Typical particle sizes for TiS_2 and $LiCoO_2$ are shown in **Figure** 4; the TiS_2 crystals exceed 1 mm in thickness and could be on the order of a millimeter in the basal plane. Today, the NMC morphology are similar to meatballs, with the primary particles around 300–500 nm, and the secondary particles between 5 and 15 mm. This allows high capacity at moderate rates, and as today they are synthesized at lower temperatures than the single crystals, a lower-cost product. Whereas, the NMC "meatballs" tend to crack on extended cycling; Xiao has proposed[18] that the single crystals if smaller than 3 mm will not tend to crack. There is increasing R&D evidence[19] that although single crystals have a lower capacity at low rates due to the slower overall diffusion in the larger particles, at very high rates their capacity exceeds that of the meatballs because the latter become starved of electrolyte. A challenge that is being addressed is how to make single crystals in larger quantities.[20]
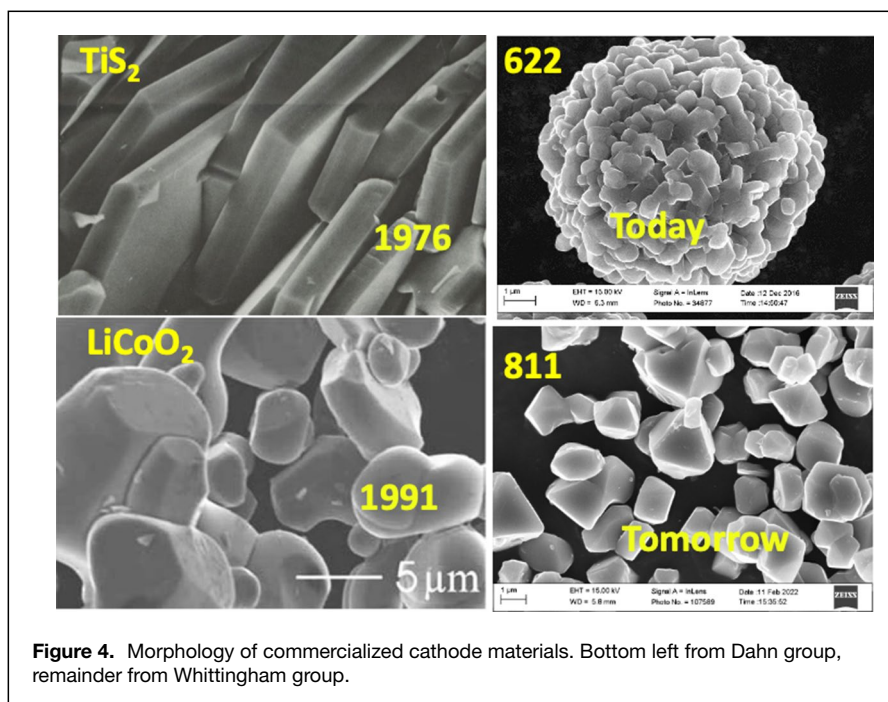
the ingredients are readily available on most continents. Extensive studies have shown that substituted and nano-sized LFP reacts by a single phase-type reaction, rather than the two-phase reaction of larger particles.[14] This results in its high rate capability, unexpected for an insulator; however, it has a low-temperature reaction issue. LFP has significantly lower energy densities than the layered oxides. This can be partially ameliorated by replacing the iron with manganese,[15] leading to a discharge voltage just under 4 V compared to the less than 3.5 V for LFP. Another approach is to intercalate two lithium ions into the lattice, and research is underway on materials such as Li_xVOPO_4 where two lithium ions can be cycled without harming the host lattice structure.[16] However, the reactivity of these vanadium compounds, some of which are used commercially as

Figure 4. Morphology of commercialized cathode materials. Bottom left from Dahn group, remainder from Whittingham group.

The lower surface area of the single crystals reduces the side reactions with the electrolyte giving them a much longer life than the meatballs. They have been promoted as the "million-mile" batteries by Dahn.[21] Their low reactivity and long life should allow them to be used in vehicles that can be tethered to the electric grid while not in operation. This should lead to a more resilient grid and provide local power backup when the grid is not available.

Alternative systems/approaches

It is going to be at least five to ten years before any alternative technologies can compete on cost with lithium-ion technology. Li-ion is the lowest cost high energy density battery on the market. They are also readily mobile, even those used for grid storage as they are in shipping containers. For example, the LFP system in Binghamton, which enabled the shutdown of the co-sited peaker coal power plant in 2009, was moved to Ohio by the AES utility after the 2011 Susquehanna flood. Subsequently, it was moved to Puerto Rico to help stabilize the grid after the Irma and Maria hurricanes. This demonstrates the mobility of containerized Li-ion storage systems. In addition, with no moving parts they do not need on-site expertise and can be readily controlled from a distance.

There is a lot of interest in *solid-state batteries*, where today's liquid organic electrolyte and polymeric separator are replaced by a solid electrolyte or combination of solid electrolytes. There are many challenges. These include the large volume changes of the electrodes during cycling that can break the interfaces between electrode and electrolyte leading to voids, and the need to allow the transport of ions between the active cathode particles. The latter is solved today by either the addition of a few drops of a liquid electrolyte

to the cathode, which enables lithium ions to get from particle to particle and across the electrode/electrolyte interface or by mixing some of the solid electrolyte into the cathode powder. The former is sometimes called a hybrid cell. Neither is perfect, the former will increase the side reactions and the latter increase the cost and reduce the energy density. Some of these contact issues are being "solved" using extreme pressure on the cells, which is not realistic in commercial cells. There are mainly three classes of solid electrolytes being considered: polymers, ceramic oxides, and sulfides. Only the first are in commercial operation. They have been commercialized by the French company Boloré/Blue Solutions, using poly(ethylene oxide) (PEO), a lithium anode, and most recently LFP in small cars, grid storage, and for buses. These are operated around 70°C because of the low conductivity of the PEO. However, recent bus fires in Stuttgart and Paris have cast doubt on their safety. Among the ceramics, the oxide lithium lanthanum zirconium oxide has been the most studied in the last decade and is under active development by a number of companies mostly for automotive applications. Here again, a lithium-metal anode is being proposed and much effort is being focused on high-nickel NMCs.

The sulfides, being a soft material, are expected to form better interfaces, but it is not clear that they are stable to lithium metal, so a dual electrolyte could be needed or pure lithium metal will need to be replaced by a lower thermodynamic potential lithium source. Significant progress is being made, but a number of materials improvements are still needed to make large solid-state cells viable. It is possible that we will see hybrid cells in which a ceramic separator is used with a liquid electrolyte before a fully solid-state cell is available. In all cases, the potential formation of dendrites and their mitigation must be addressed by researchers. In summary, for solid-state batteries, the following critical questions need answering:

1. Can a solid electrolyte stop dendrites from forming and if not is thermal runaway less of an issue with a solid than with a liquid?
2. Can the interfaces between the electrodes and the electrolyte be married to allow high rates of reaction with only practical pressures being applied, or must another soft phase such as a liquid be used to "grease" the interface?
3. If dual solid electrolytes are used, to ensure thermodynamic stability with both the lithium anode and the cathode, what is the manufacturing and cost penalty?

4. Can thermal management be eliminated for a solid electrolyte battery pack? This would eliminate the 20% round-trip efficiency lost on Li-ion grid systems.
5. Is there a limit on the thickness of ionically poorly conducting solids such as the NMC?
6. Can a Li/S cell be made to work? This would allow for the highest gravimetric energy density and possibly the lowest cost. What would such a cell look like?

A number of alternative systems based on *sodium intercalation electrodes* are being investigated. These will only be successful if they are of lower cost and can meet the other target goals. There are at least three chemistries being actively studied: layered oxides, Prussian blue/white, and several vanadium phosphates. The first demands that they have less Ni/kWh than Li/NMC, the second that there is no chance of cyanide release after extended cycling, and the third that the vanadium reactivity with the electrolyte can be controlled. Their safety also needs to be thoroughly studied. Even though, there is evidence that sodium cells can be shipped in the completely discharged state making transportation safer, sodium metal melts at around 100°C compared with the 180°C for lithium, which could lead to major issues in a thermal runaway event. However, sodium-based cells are unlikely to find application in volume-sensitive systems such as portable electronics and high-performance vehicles because of their lower energy densities. Cells based on potassium are only of scientific curiosity to get a better understanding of alkali cells. Potassium will diffuse slower than either Li or Na and have a much more slopy discharge curve because of its larger size. In addition, melting around 60°C it will be extremely hazardous in the metallic form.

Lithium (or sodium) sulfur cells offer the highest gravimetric energy densities for alkali metal rechargeable cells. They have been under study since the 1960s, but there are many major scientific challenges. These include the very poor conductivities, both electronic and ionic, of sulfur and lithium sulfide. Could a solid electrolyte make such cells viable, but have all the research efforts expended on beta alumina exhausted potential solutions?

Flow batteries with their moving parts are more complex. They also tend to be more corrosive leading to lifetime challenges. However, it is to be expected that flow batteries will find a niche for long-duration storage. The chemistry used is still wide open.

Beyond the cell chemistry

Moving on from the materials themselves, there are a number of other big concerns that the materials researcher should be worried about given that Li-based battery technology will stick around for some time to come. These revolve about how the world makes, uses, and then disposes of these energy-storage devices. Much more materials R&D is required in the areas described next.

Manufacturing of materials

It does not make sense that manufacturing technologies have not changed in 30 years. We need to reduce the 40–80 kWh of electricity equivalent it takes to produce a 1-kWh battery. We have got to find new manufacturing technologies and minimize transportation costs from the mine through processing to the final product. This entails a thorough analysis of the initial materials processing from the mine to the product. Where along the line can one extract the desired material and how pure does the starting material have to be? We did this at Exxon for the formation of battery-grade TiS_2 by two large European companies and basically take the gaseous $TiCl4$ and react it with H_2S in place of the air used to make paint pigment. There was no need to convert it to the metal or a metallic salt as an intermediate step, as shown in **Figure** 5.

NMC is still commercially made using a batch process, involving several steps. First, an NMC hydroxide is precipitated from an aqueous sulfate solution and then the precipitate is fired with either LiOH or Li_2CO_3. LiOH is used for high Ni NMC where the firing temperature is too low to decompose the carbonate and the firing must also
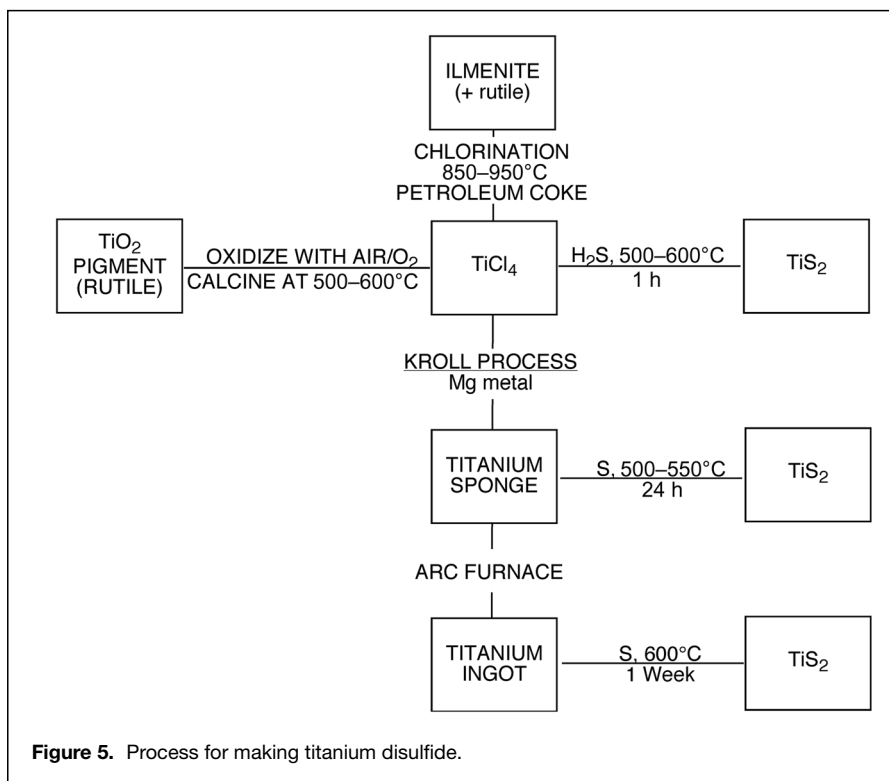

Figure 5. Process for making titanium disulfide.

be done in an oxygen environment to maintain the oxygen content. For low nickel, a higher temperature is used that is sufficiently high to effectively decompose Li_2CO_3. Coatings and or substituents can be added either at this stage or in a subsequent step. A single-step continuous process would be much preferred and even ideally one where an effective protection layer can also be formed.

LFP is commonly made by the carbothermal reduction of ferric salts at elevated temperatures; alternatively, it has been manufactured by hydrothermal methods that use much lower temperatures with their lower cost but the starting material is often the more expensive ferrous salt. Often, here again, part of the iron is often substituted by other ions to give preferred morphology and phase behavior (single-phase Li_xFePO_4) when operational.[14]

The graphitic carbon anode used today comprises either synthetic carbons formed at extreme temperatures or from natural graphite that often has to be purified using hydrofluoric acid. The latter is restricted in most countries. The clean flake graphite must then be spheroidized, which results in a low yield of usable product. Spheroidization allows for an improved packing density over the flat flakes. A combination of both natural and artificial carbons may also be used.

Sustainability: Supply chain, cell manufacturing, and recycling

The present *materials supply chain* for batteries is broken. It is not sustainable nor is it environmentally clean. Many of the materials are shipped tens of thousands of miles from the mine to the finished battery pack. We need regional supply chains, and we need to look at clean mining with clean energy. This needs materials research to determine the feasibility of switching from carbothermal reduction of ores to electroreduction using renewable energy. It could mean that different battery chemistries will be favorable on each continent. It is clear to all that the heavy metals, such as cobalt, must be eliminated. It is only a matter of time before there will be pressure to reduce the amount of nickel in batteries. The same approach needs to be taken for the formation of the lithium to be used in batteries. For example, massive amounts of water are needed to release the lithium from the salts in South America. Can we be more selective and just pull out the lithium rather than evaporative techniques?

The *manufacturing* of the cathode electrode today uses the toxic organic solvent *N*-methyl-2-pyrrolidone (NMP) to make the paste that is to be coated onto the electrode, typically carbon-coated aluminum foil. Not only does this present a hazard in the workplace, it also requires much more energy to be evaporated from the coated electrode than say an aqueous paste does (as used for producing the carbon anode electrode). Moreover, it must be recovered again at great expense. All signs are pointing to its banishment in Europe, and the rest of the free world is likely to follow suit. So, an alternative is needed. Cyrene was investigated for NMC, but elevated temperatures had to be used to dissolve the PVDF binder, so it

is not likely to be practical.[22] Much more research is needed on replacing both the solvent and the PVDF binder. The binder is a fluorine-containing material, a potential source of PFAS, so is likely to become a candidate for elimination. Similarly, the fluorine-containing salts and solvents in the electrolyte need replacing by lower cost and environmentally friendly materials.

Recycling technologies are critical to a sustainable world. In addition to PFAS, unacceptable levels of lithium are being found in drinking water in the United States, according to a recent data set from the US Environmental Protection Agency.[23] Thus, it is incumbent on all to recycle all batteries. Gigafactories are a major source of battery waste and perhaps the easiest to handle, because the materials are in essentially a virgin state. The material needs to be recovered with minimal processing and fed right back into the front end of the facility. End-of-life batteries are more difficult to recycle, with their damaged materials and certainly less lithium than the virgin material. In addition, the chemical compositions of the materials are likely to change over the lifetime of a battery, so that process upgrading will be required. Brute force burning of spent cells is not acceptable as fluoride and other toxic materials will be emitted into the environment. It will be essential that batteries can be effectively separated by chemistry at the front end of the recycling process. AI could be of help here, but it will be important that each battery is permanently accompanied by its pedigree. One approach is for each cell/battery to have an equivalent of a passport,[24] and this is mandated in the EU from February 2027. The conversion of spent battery material into so-called "Black Mass," from which the metals must then be separated does not seem to be an optimal process approach. We need to be smarter, to be sustainable and to help build localized supply chains so that the export of black mass between continents does not occur.

Safety and the exploding battery nightmare

Batteries store energy and therefore are inherently unsafe, and should be treated with the same respect petroleum products are. I have said, "We can't have cheap junk out there."[25] A number of batteries sold today are counterfeit, that is, pretending to be something they are not. Some years ago, when we were studying the SONY Nexelion battery, two-thirds of the cells we purchased online were not the genuine SONY article. Many fatal e-bike fires reported this year in New York City are believed to be due to counterfeit batteries and/or chargers or just to low-cost systems. If we are to have a safe and growing electric economy, all systems should be certified, just like household appliances are today.

However, all safety issues are not associated with counterfeit batteries. There have been many instances of badly designed batteries/systems from the leading manufacturers creating issues, from the A123 LFP EV cells for Fiske, the Avestor/AT&T PEO solid-state batteries, SONY's laptops, Samsung's phones, and Boeing 787 $LiCoO_2$ batteries[26] to LG/GM's recent Bolt recall. Materials scientists and engineers have a responsibility to overdesign safety into the materials and the whole

system. We need to understand better why dendrites grow on charging in both liquid and solid electrolytes and to find a way to mitigate them. With much attention being paid these days to the possibilities of solid-state batteries, some claims for the technology are overblown, that is, solid-state chemistries are not necessarily safer than liquid chemistries and carry their own risks, given they incorporate far more lithium. Another example, LFP is not necessarily safer than NMC as exemplified by the bus fires in Stuttgart and Paris. We need to understand better all the gases that can be evolved on thermal runaway and/or a fire so that firefighters and other first responders can be forewarned. And we need to develop ways to permanently extinguish fires and not let them burn to extinction as is common today.

Summary

Electrical energy storage is crucial for the effective proliferation of an electric economy to all and for the implementation of many renewable energy technologies. Transformational changes in battery science and technology have occurred in the last 10 years that have allowed higher and faster energy storage at the lower cost and longer lifetime that are allowing deep market penetration. However, much more is needed to make the manufacturing, deployment, and reuse more sustainable, environmentally friendly, and safer.

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