Nobel Prize in Chemistry 2019*

A Ramanan

The Chemistry 2019 Laureates – The Quest for Excellence

The 2019 Nobel Prize in Chemistry was awarded to M. Stanley Whittingham, John B. Goodenough and Akira Yoshino for "the development of lithium-ion batteries" that created a rechargeable world. It honors the three scientists (*Figure* 1), whose pioneering work in the chemistry of novel cathodes and anodes led to the explosive growth of lithium-ion batteries (LIBs). The technology has not only revolutionized portable electronics but are also making a greater impact on electric vehicles and grid storage. Interestingly, the three men worked independently on different aspects of chemistry, physics and engineering of electrode materials, which eventually led to LIB. Stan Whittingham investigated the intercalation of different ions into complex metal oxides and explored its use as potential new electrodes along with his advisor Peter G. Dickens in the Inorganic Chemistry Laboratory, University of Oxford, UK. Though the field of solid-state chemistry research was dominant in Europe in the 1960s and 70s, the young graduate took up a postdoctoral position with Robert A. Huggins at Stanford University, US, to work on the electrochemistry of various solid compounds. While his work at Oxford was mostly confined to synthesis and structures, the materials laboratory at Stanford was more interdisciplinary in nature and provided the opportunity to investigate detailed properties. Exxon employed him to initiate work on alternative energy storage systems for electric vehicles. At Exxon, Whittingham laid the foundation for LIB by creating an innovative cathode based on layered titanium disulphide that could intercalate lithium ions achieving a potential over two volts by combining with lithium anode. However, the reactive lithium anode hampered the viability of a commercial battery.

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At 97 years of age, Goodenough is the oldest Nobel Laureate till date. After earning a doctorate in physics from the University of Chicago in 1952 under Clarence Zener (inventor of the diode), he moved to Lincoln Laboratory, MIT to start his independent career. He gained vast experience working in various labs including the Digital Computer Development Group, the Magnetism and Resonance Group and the Electronics and Materials Group. He made immense contributions to the fundamental aspects of solid-state chemistry. Notables are the two classic books, *Magnetism and the chemical bond* (1963) and *Metallic oxides* (1973), and the formulation of fundamental rules of magnetism, now known as the Goodenough–Kanamori rule (1955) that rationalized magnetic properties of a wide range of materials on a qualitative level. John Goodenough moved to Oxford, UK in 1976 where he discovered that a layered cobalt oxide instead of sulphide as the cathode, coupled with lithium intercalated graphite anode led to a cell with four volts. This was an important breakthrough and would lead to much more powerful batteries.

Akira Yoshino graduated with a master's in petrochemistry from Kyoto University and took up an industrial position at Asahi Kasei Corporation in 1981. He began research on rechargeable batteries using polyacetylenes – the electroconductive polymer discovered by Hideki Shirakawa, who would receive the Nobel Prize in Chemistry in 2000. With Goodenough's cathode as a basis, Akira Yoshino used lithium intercalated petroleum coke, a different carbon material as anode and assembled the first commercially viable lithium-ion battery in 1985.

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Early History of Batteries

Benjamin Franklin was the first to use the term 'battery' in 1749 when he was conducting experiments with electricity using a set of linked capacitors. In 1786, an Italian doctor and scientist named Galvani attempted to dissect a frog using two different metal tools (iron and bronze). The frog's leg started dancing, and Galvani promptly declared that he had discovered 'animal electricity'! In

Figure 1. Nobel Laureates Chemistry – 2019.

1800, Alessandro Volta, an Italian physicist, assembled the first battery with stacked discs of copper and zinc metal sheets, separated by a cloth soaked in salty water. Over the centuries, primary batteries (non-rechargeable) have been constantly redesigned, improved and miniaturized. Towards, the end of the 19th century, the first zinc rechargeable battery was invented, based on liquid zinc bromide. And in 1901, Thomas Edison had the first patent on Zn-Ni battery. The lead-acid battery, invented in 1859 (Gaston Platé), is still in use to start most internal combustion engine cars. In the second half of the 20th century, zinc rechargeable batteries would be increasingly outdone by other, higher performance systems such as lead-acid (car batteries, stationary storage), nickel-cadmium (power tools, toys), nickel-metal hydride (cellphones, toys, hybrid electric vehicles) and of course, the ubiquitous lithium-ion (portable electronics, electric vehicles, storage). Today, batteries come in a wide range of sizes – from large Megawatt capacities, which store the power from solar farms or substations to guarantee stable supply to entire villages or islands, to tiny batteries like those used in electronic watches.

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Basics of an Electrochemical Cell

Batteries are based on different chemistries (*Table* 1), which generate basic cell voltages typically in the 1.0–4.0 V range. The

Battery type	Cathode (forward reaction during discharge)	Anode (forward reaction during discharge)	Electrolyte	Cell potential (Volts)
Lead-acid	$PbO_2 + 4H^+ + SO_4^{2-} + 2e^- \rightleftharpoons PbSO_4 + 2H_2O$	$Pb + SO_4^{2-} \rightleftharpoons PbSO_4 + 2e^-$	aqueous H_2SO_4	2.05
Ni-Cd	$Cd(OH)_2 + 2e^- \rightleftharpoons Cd + 2OH^-$	$Ni(OH)_2 + OH^- \rightleftharpoons NiOOH + H_2O + e^-$	aqueous KOH	1.30
Ni-M hydride	$NiOOH + H_2O + e^- \rightleftharpoons Ni(OH)_2 + OH^-$	$MH + OH^- \rightleftharpoons M + H_2O + e^-$	aqueous KOH	1.31
Lithium-ion	$Li_{1-x}CoO_2 + xLi^+ + xe^- \rightleftharpoons LiCoO_2$	$Li_xC_6 \rightleftharpoons 6C + xLi^+ + xe^-$	$LiClO_4$ in propylene carbonate	3.80

Table 1. Chemical reactions in selected rechargeable cells.

The electrolyte resides between the anode and the cathode and enables the transport of ions rapidly from one electrode to the other.

stacking of the cells in series increases the voltage, while their connection in parallel enhances the supply of current. This principle is used to add up to the required voltages and currents, all the way to Megawatt power. A battery (*Figure* 2) is a transducer that converts chemical energy into electrical energy and vice versa. It consists of three active components – the anode, the cathode and the electrolyte. The anode is the positive terminal of the cell, typically a metal. The cathode is a negative terminal, normally a halide or chalcogenide-based compound. The electrolyte resides between the anode and the cathode and enables the transport of ions rapidly from one electrode to the other. To obtain a high energy density, i.e., a high cell voltage, one needs to look at a reaction that has high free energy changes during the anode/cathode reaction or the oxidation/reduction.

Metals have long been recognized as a suitable material to serve as battery anodes. They are readily oxidized from their metallic state to produce ions and electrons. The electrons liberated are conducted through the metal, the negative terminal of the cell. The fact that metals are physically strong and easily produced
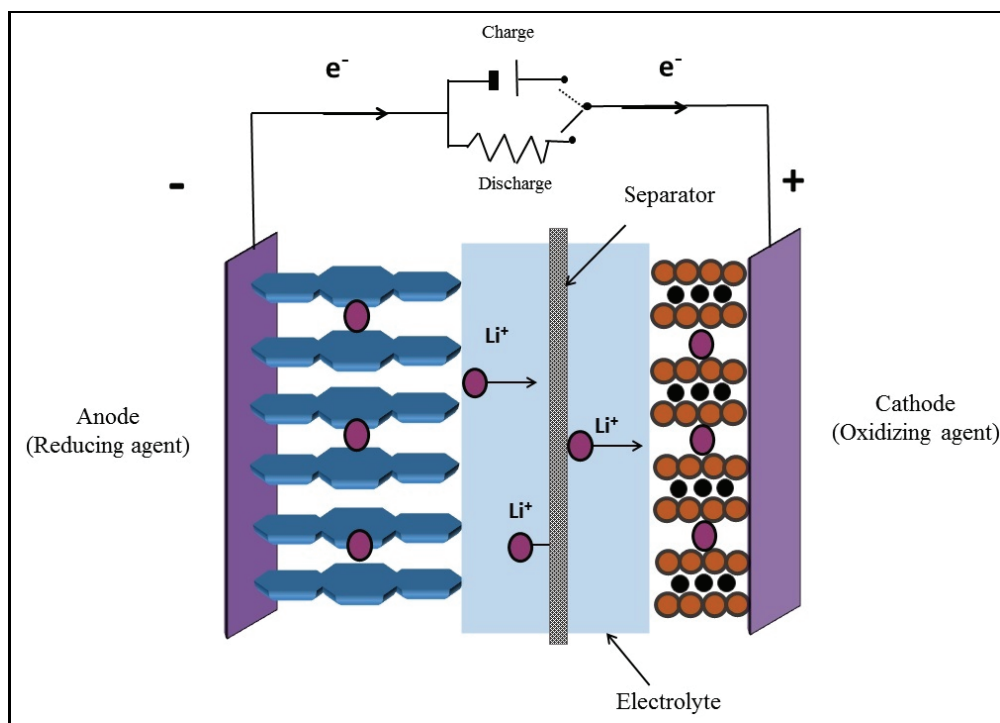
into any desired shape further makes them attractive to function as anodes. The standard reduction potential is a measure of the ease with which the ion is reduced back to the metallic state. The quantity of electric charge produced per gram of metal in the anode half-reaction, referred to as the electrochemical capacity, is an important property of the metal for practical applications. In many cases, alloys such as LiAl are preferred due to its stability at higher temperatures of operation. The cathodes are generally made of electronegative elements like sulfides or oxides of metal in higher oxidation state.

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the cell discharges, the oxidation half-reaction at the anode is, $Li(s) \rightarrow Li^+ + e^-$. The electrons thus produced are driven through the external circuit with a load and return to the cell at the cathode. The solvated lithium ions migrate through the cell to the cathode where they undergo a chemical reaction. A battery electrolyte also separates the anode and the cathode, to prevent any direct reaction between them. Additionally, it must allow ions to pass between the electrodes. This movement of ions constitutes the flow of charge inside the battery. For a lithium battery, the electrolyte must allow lithium ions to migrate from the anode to the cathode. Unlike other batteries employing aqueous electrolytes, a lithium battery necessitates the use of nonaqueous and aprotic solvents since it reacts violently with water, by producing hydrogen gas. The solvent needs to be sufficiently polar to dissolve lithium salts and produce a solution with high ionic conductivity. A few organic solvents that meet these criteria include acetonitrile, dioxane, γ-butyrolactone, methyl formate, 1, 2-dimethoxyethane, propylene carbonate, dimethylsulfoxide and tetrahydrofuran. The lithium salts such as $LiClO_4$, $LiPF_6$, $LiBr$, $LiBF_4$, $LiCF_3SO_3$, and $LiAlCl_4$ with low lattice energies (small lithium ion combined with a large anion) are highly soluble in these solvents and also show appreciable ionic conductivity. Primary batteries based on lithium metal have been known for a long time. Matsushita, Japan developed the first $Li/(CF)_n$ battery to use it in fishing floats. Though lithium fluoride and carbon are the final reaction products, the cell potential of 2.8–3.0 V suggests a different electrochemical reaction. It was assumed that lithium initially intercalates the carbon monofluoride lattice and subsequently undergoes a dissociation reaction, $Li + (CF)_n \rightarrow Li_x(CF)_n \rightarrow C + LiF$. In spite of considerable works by several others, the reaction could not be made reversible. Meanwhile, Sanyo developed one of the earliest lithium batteries with the Li/MnO_2 system that was employed in solar rechargeable calculators.

Development of Lithium Ion Battery

In the 1960s, chemists in Europe were investigating the intercalation chemistry of lithium into layered disulfides and diselenides (MX_2) where M is a transition metal atom. The ready chemical formation of intercalation compounds under ambient condition, their inherent reversibility and modification of their physical properties such as electronic and ionic conductivity on reaction made them natural candidates for electrochemical devices, such as batteries. The interest in the possibility of using an alkali-metal ion as the mobile electrolyte cation, led Brian Steele, UK, to suggest the use of TiS_2 as the cathode of a lithium battery, in analogy to the insertion of H^+ into layered NiOOH with an aqueous electrolyte. A primary lithium battery (non-rechargeable) of long shelf life that used an organic-carbonate liquid electrolyte was already available in the market. In 1967, Kummer and Webber of the Ford Motor Co. reported fast sodium ion diffusion at 300^oC in partially occupied Na and O layers of spinel-related sodium β-alumina and eventually assembled a sodium-sulfur battery operating above 300^oC. Their cell had molten sodium as the negative electrode (anode) and carbon felt in molten sulfur as the positive electrode (cathode); the molten electrodes were separated by the solid electrolyte. This was in contrast to other electrochemical cells, wherein solid electrodes are kept apart by a separator containing the aqueous electrolyte.

The oil crisis in the early 1970s stimulated extensive interest in cation transport in solid electrolytes and alkali-ion batteries. Under the leadership of Robert Huggins at Stanford, his student Gamble and Whittingham investigated the intercalation of a range of electron-donating molecules and ions into layered dichalcogenides, in particular, tantalum disulfide, with an objective to enhance its superconducting properties. Whittingham studied the transport properties along with the thermodynamic and kinetic aspects of the different synthesized solids identified as solid electrolytes; in particular, he studied 'β-alumina', which showed significant ionic conductivity. He also worked on the transport properties of intercalated bronzes that revealed high diffusivity of mono-

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Exxon's efforts to commercialize the LIBs failed due to safety issues arising out of the lithium anode and the flammable organic electrolyte. During charging, non-uniform deposits of lithium on the anode resulted in the growth of lithium dendrites or whiskers owing to the different nucleation energy across the metal surface; the protruding dendrites punctured the separator containing the flammable organic-liquid electrolyte, thereby, creating an internal short-circuit followed by frequent explosions.

valent alkali and silver ions inside the channels. A continuation of their previous studies on dichalcogenides resulted in the formation of the hydrated alkali-metal intercalates of tantalum disulfide. The stability of the hydrates, $K_x(H_2O)TaS_2$, was attributed to their salt-like behavior, in contrast to the metallic-like behavior noted in graphite, KC_8. They also noted that such intercalation reactions could be accomplished in an electrochemical cell either by electrolyzing dissolved salts or by inserting ions from the anode. Further, the group at Exxon found the lighter titanium disulfide to be more suitable as an energy storage electrode. Titanium disulfide has a hexagonal close-packed sulfur lattice with the titanium ions in octahedral sites between alternating sulfur sheets. The TiS_2 sheets are stacked directly on top of one another, giving the sulfur anion stacking sequence ABAB (*Figure* 3a). They later realized that being a semi-metal, the material did not require additional conductive diluent in the cathode structure. More importantly, the electrochemical reaction yielded a single phase over the entire composition range of Li_xTiS_2 for $0 \leq x \leq 1$, and all the lithium could be removed reversibly. Thus the first rechargeable Li-ion battery with a cathode of layered TiS_2 and anode of metallic LiAl alloy was reported by Whittingham in 1976. Exxon marketed the button cells with LiAl anodes and TiS_2 cathodes for watches and other small devices between 1977 and 1979. The LiAl anode provided better safety of the cells over pure lithium anode. Exxon exhibited the lithium single cell at the Electric Vehicle Show in Chicago in 1977. Exxon's subsequent efforts to commercialize the LIBs failed due to safety issues arising out of the lithium anode and flammable organic electrolyte. During charging, non-uniform deposits of lithium on the anode resulted in the growth of lithium dendrites or whiskers owing to the different nucleation energy across the metal surface; the protruding dendrites punctured the separator containing the flammable organic-liquid electrolyte, thereby, creating an internal short-circuit followed by frequent explosions. Within a few years, Exxon abandoned the project for safety reasons.

Meanwhile, many groups were experimenting with lithium as the

positive electrode (anode) which posed considerable problems – the system was highly flammable and prone to short-circuiting. In the same period, reversible intercalation in graphite and cathodic oxides was examined by Jürgen Otto Besenhard, Samar Basu and Rachid Yazami independently. Battery cells built in that time, however, would deteriorate rapidly with each recharge. Yazami tackled this problem in the early 1980s through his research on the reversible electrochemical intercalation of lithium in graphite. They also noticed that Li got plated onto the surface of the carbon more rapidly than the insertion of lithium ions into carbon. This, however, did not deter Yazami and P. Touzain to come out with the reversible graphite-lithium negative electrode for electrochemical generators.

During the oil crisis, Goodenough explored oxide materials towards energy conservation and electrical energy storage. His group synthesized several new framework solids that supported fast sodium ion transport, a significant example being sodium zirconium silicon phosphate, known as NASICON. When he moved to Oxford, UK in 1976, he and his group started to examine the dual problem of replacing the metallic lithium negative electrode with an intercalation host and a cathode which provides a larger voltage vis-à-vis Li. They rationalized that the voltage attainable with a layered sulfide would be limited to ~2.5 V since the bottom of the Ti(IV)/Ti(III) conduction band of TiS_2 is only 0.2 eV above the top of the S-$3p$ bands. The oxides would offer O-$2p$ bands at a lower energy. Earlier studies on a large number of solid electrolytes led Goodenough to identify that lithium ions in close-packed oxygen arrays are likely to be mobile. He considered the 1:1 ordered rock-salt based $LiMO_2$ series possessing a comparable layered structure to that of TiS_2 to be the most likely candidates for delithiation (*Figure* 3b). His group systematically explored the extraction of Li from the layered oxides, $LiMO_2$ (M = Cr, Co, Ni). The disproportionation reaction, $3Cr^{IV} = 2Cr^{III} + Cr^{VI}$ with the Cr^{VI} occupying interlayer tetrahedral sites eliminated $LiCrO_2$. The group, however, succeeded in reversibly extracting Li from $LiCoO_2$ and $LiNiO_2$ at a voltage ~ 4.0 V versus

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Figure 3. **(a)** Crystal structre of TiS$_2$. **(b)** Crystal structure of LiCoO$_2$.
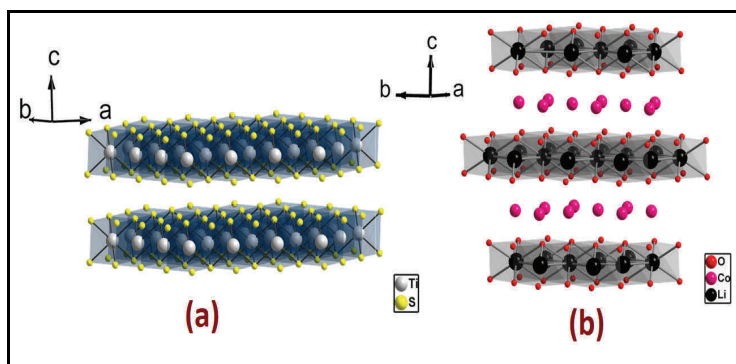
Li. Till then, the battery community was familiar with the fabrication of only charged batteries. The battery industries of Europe, England, and the US remained skeptical of the construction of a discharged cell without specification of the discharged anode.

Akira Yoshino from Japan initially examined the viability of organic conductors like polyacetylene as possible negative electrodes. This experience enabled him to extend the concept of intercalation, and he arrived at the right composite material after studying a series of carbonaceous materials. The secondary battery thus fabricated based on this new combination, enabled stable charging and discharging, over many cycles for a long period. This invention (1985) led to the construction of a new secondary battery using Goodenough's LiCoO$_2$ as the positive electrode and lithium intercalated petroleum coke (a carbon material) as the negative electrode. The first Li-ion cell with Li$_{1-x}$CoO$_2$ as the cathode and petroleum coke as the anode was assembled in the discharged state. Both carbon anode and LiCoO$_2$ cathode are stable in air, which is highly beneficial from the engineering and manufacturing perspectives. This battery design facilitated large-scale manufacturing of LIBs in the early 1990s. It should be highlighted that Yoshino also carried out the first safety test on LIBs to validate their enhanced safety features, by dropping iron lump on the battery cells, in contrast to that of metallic lithium batteries which caused fire and explosion. Yoshino's success is widely considered the beginning of modern commercial LIBs and

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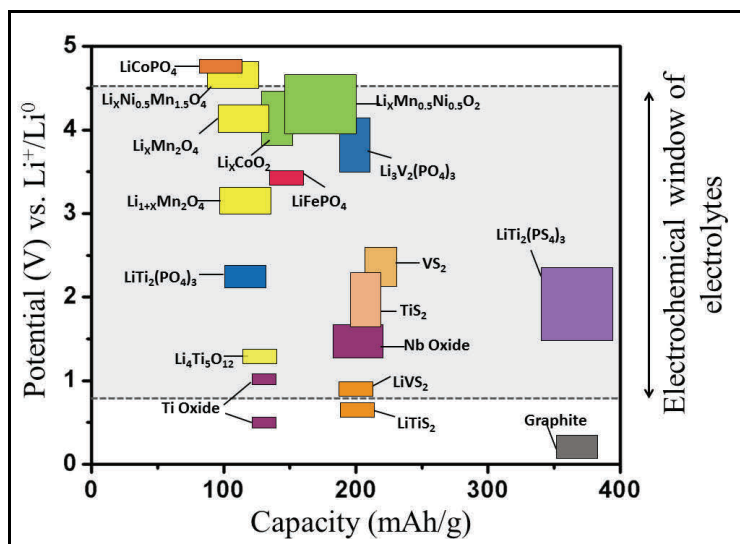
Figure 4. Voltage versus capacity of several cathode materials relative to the window of the organic electrolyte (e.g. 1M $LiPF_6$ in an organic carbonate). [Adapted from J. B. Goodenough and Y. Kim, *Chem. Mater.*, Vol.22, p.593, 2010.]

eventually, Sony commercialized it in 1991. It was a tremendous success and supported the revolution of personal mobile electronics. Goodenough, Yazami, and Yoshino were awarded the 2012 IEEE Medal for Environmental and Safety Technologies for their pioneering contribution to the development of LIB. The original lithium cobalt oxide has been modified by incorporating additives to stabilize the crystal structure and increase the capacity. *Figure* 4 illustrates the significant contributions by Goodenough and others in the chemical tuning of several cathode materials that include $LiCo_{1-x}NixO_2$, $LiMnO_2$, olivine based $LiFePO_4$. Apart from different forms of graphitic carbons, lithium titanium oxides and nanostructured stabilized tin alloys were also developed as alternative anode materials.

Future LIBs and Their Sustainability

The $LiCoO_2$/carbon or $Li_{1-x}(Co_{1-y-z}Ni_yAl_z)O_2$/carbon cell remains the principal LIB that powers today's portable electronic devices and power tools. The flammable liquid electrolyte in a LIB is a major scientific challenge, apart from issues of scalability and cost, in comparison with a fossil fuel source wherein these

factors are mitigated or redeemed by their inherent higher energy density. With a focus on safety, solid-state batteries consisting exclusively of solid components are being developed. These solid-state batteries focus on low volume and niche applications and can offer higher energy density at a lighter weight, without the thermal issues presently associated with LIBs. A significant scientific effort is further required to develop new battery chemistries that would achieve battery performance for electric vehicles and fulfil the expectations of those driving cars with internal combustion engines.

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Since their inception, more than one hundred years ago, electric vehicles have been promising a cleaner and healthier alternative to gasoline-powered cars. The commercialization of LIBs in 1991 and subsequent improvement of battery range, cost, and durability have set up a feasible platform for the growth of electric vehicles. The advances in battery performance have made it possible for electric cars to have the range (around 240 miles per charge) required to make them viable and competitive to ICE vehicles. Notable ones are the early generation electric cars like Tesla's Roadster and Nissan's Leaf. The LIBs provide 25–50% more energy density than nickel metal hydride (NMH) batteries that were employed in the first modern hybrid electric vehicle, the Toyota Prius. The electrified transport faces severe challenges as we still lack sustainable and economically viable means of recycling end-of-lifecycle LIBs. There are also serious concerns about environmental hazards pertaining to the disposal of the waste. Currently, available incumbent Li-ion recycling technology often uses a pyrometallurgical (smelting) approach that limits the recovery rates and can emit harmful pollutants into the atmosphere.

Composite cathodes with two or three $3d$ metals and polyanions are highly promising, and cathode chemistry may require new electrolytes as well. Battery management systems to enhance the safety of huge battery packs in vehicles is the need of the hour to expand the market of LIB powered vehicles and ensure wide acceptance of electric vehicles. Nonflammable LIBs based on aqueous electrolyte or ceramic electrolyte and all-solid-state bat-

teries are other possibilities. Next-generation LIBs may use high voltage (5V) cathodes and high capacity anodes such as Si- or Sn-based. It is still challenging to develop electrode materials with a low carbon footprint. The possible alternatives are biologically derived organic or inorganic electrodes using aqueous electrolyte or organic electrodes that will not be easily dissolved by the electrolyte. The impact of the present materials on the environment is still uncertain. Mass production of LIBs for electric vehicles is expected to produce large volumes of contaminated waste which can create serious problems around the mine sites.

There are still many issues to achieve next-generation LIBs. Apart from new battery chemistry, safety and sustainability are still the major factors to go beyond LIBs in the future. The chemistry to replace the existing LIB technology is still challenging. To increase energy density of LIBs, it is required to find electrode couples with high specific capacities and high operating cell voltages, where Si- and Sn-based anodes could be potential materials. While preparation of Si nanomaterials on a large scale with low cost is a daunting task, Sn-based anodes suffer from the issue of poor cycling performance. Si-/Sn- based composites are also possible. $LiCoO_2$ is expensive and highly toxic. The increasingly popular $LiFePO_4$ has a low capacity. Though NiCoMn-based cathodes developed by Argonne National Laboratory are highly attractive, the specific capacity is only moderate. Also, both Co and Ni are expensive and toxic. From the environmental perspective, it is desirable to avoid Co, Ni or other toxic elements. Additionally, the ideal cathode should be able to reversibly insert/extract more than one electron per $3d$ metal suggesting materials containing Mn and/or Fe.

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> With an objective to develop batteries with higher energy, Li-air and Li-sulfur have attracted considerable attention. Although Zn-air and high-temperature Na-sulfur batteries have been known for some time, rechargeable $Li-O_2$ and Li-S batteries are yet to cross technical barriers.

Beyond LIBs

With an objective to develop batteries with higher energy, Li-air and Li-sulfur have attracted considerable attention. Although Zn-air and high-temperature Na-sulfur batteries have been known for some time, rechargeable $Li-O_2$ and Li-S batteries are yet to cross

technical barriers. On the anode front, the issue of Li- electrolyte interface in the development of Li-O_2 or Li-S batteries is being revisited. Sodium-ion batteries (SIBs) are being considered for large-scale stationary energy storage systems. Though sodium (2.7V for Na/Na$^+$) has a lower potential in comparison to lithium, electrolyte degradation and safe electrolyte with low decomposition potential are being considered. Other potential cathodes that are being studied are carbon-coated $NaVPO_4F$, $Na_{0.44}MnO_2$, $Na_xCo[Fe(CN)_6]_{0.90}2.9H_2O$, layered $Na_{0.71}CoO_2$ and Na_2FePO_4F. Unlike lithium, graphite is not suitable for reversible incorporation of sodium. However, selected carbon nanomaterials show promising Na storage. Mg-ion batteries have the advantage of negligible dendrite formation and offer high-energy density at a significantly lower cost. Better safety features in Mg-ion batteries makes them a step closer to commercialization. Al-ion batteries, with three- electron transfer, could achieve three times higher energy density than the LIBs, but the use of expensive ionic liquids limits their application. Rechargeable Zn-air batteries could be more promising than Li-air batteries.

The success of commercial LIBs was a result of intensive research and contributions by many great scientists over the past few decades. The three scientists receiving the Nobel Prize in Chemistry deserve the recognition for their fundamental contribution in the area of solid-state chemistry/electrochemistry/engineering, identifying key issues towards the realization of a rechargeable battery.

The success of commercial LIBs was a result of intensive research and contributions by many great scientists over the past few decades. The three scientists receiving the Nobel Prize in Chemistry deserve the recognition for their fundamental contribution in the area of solid-state chemistry/electrochemistry/engineering, identifying key issues towards the realization of a rechargeable battery. The three laureates are still active, sparkling with new ideas and unbound enthusiasm. "I am in the lab every day. I am still working", told 97 years old Goodenough who is currently a Professor at the University of Texas, Austin, USA. Recently, he has developed the prototype of a solid-state battery along with his collaborator Professor Braga from Portugal, employing alkali (Li, Na) barium oxychloride glass as a solid electrolyte. Mike Stanley Whittingham, the Director of NorthEast Centre for Chemical Energy Storage and Professor of Chemistry from Binghamton University, USA comments, "I think it passes on the message that we can have a cleaner and nicer world, and perhaps start to solve the

climate problem". Teaching, in the formal sense, is relatively new for Akira Yoshino who is a Professor at Meijo University, Japan. His advice to the future generations of students is, "When a child becomes interested in something after being given the right stimulus, they will naturally find their own way of pursuing it."

Suggested Reading

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[13] For a video of how a lithium-ion battery is made, watch: https://youtu.be/nZCRLGegZZY

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