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Materials Towards the Development of Li Rechargeable Thin Film Battery

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Abstract
The present work gives an overview of materials towards the development of Li rechargeable thin film batteries. Conventional Li rechargeable battery faces issues related with large volume, safety issues due to the presence of liquid electrolyte. These issues are proposed to resolve by developing these batteries in thin film form. The main drawback of these batteries is finding an appropriate inorganic material to be used as electrolytes. Other issue is related with design of appropriate cathode material which should be cost effective and is able to provide better electrochemical performance compared to competitive counterparts. In this review, a brief description of lithium lanthanum zirconate as a solid-state electrolyte and Co free Ni rich layered oxide has been provided to overcome these issues. Strategies for optimizing these materials for designing a stable, safe and cost-effective thin film batteries are also elaborated.

Keywords- Li rechargeable batteries, Cathodes, Solid state electrolyte, Dendrites.

1. Introduction

Recent years are focused towards the optimization of energy storage devices in order to complete them with existing energy devices (Koohi-Fayegh and Rosen, 2020; Mitali et al., 2022). Some of these devices are rechargeable batteries (Winter and Brodd, 2004; Liang et al., 2019;), fuel cells (Winter and Brodd, 2004; Fan et al., 2021) and capacitors (Winter and Brodd, 2004; Sharma et al., 2019). Figure 1 shows Ragone plot for these devices which intimates that energy capability of these devices is still inferior compared to combustion engines or gas turbines. Thus, the development of these devices for better performance is essential and need of time.

Among various devices, rechargeable batteries are seen as a better alternative that can replace existing combustion engine-based devices (Li et al., 2018, Li et el., 2020a, Sun et al., 2022). Though many rechargeable batteries such as Lead acid, Ni-Cd, Ni-metal hydride (Ni-MH), Li-ion and Li-metal are developed so far but Li ion rechargeable battery (LIB) has emerged as victorious in terms of specific power and specific energy (Figure 2). Thus, numerous studies have been carried out on LIB focussing on the electrochemical performance for its use on wide range of applications. An LIB based on lithium nickel cobalt manganese oxide as cathode, and Li metal ion as the anode is shown in Figure 3. This battery utilizes a liquid electrolyte as a medium for Li ion propagation. A commonly used liquid electrolyte is 1M LiP_6 in a 1:1 mixed solvent of ethylene carbonate (EC)/dimethyl carbonate (DMC) (Jian et al., 2020). Cathode may have variable compositions of NCM, however, Ni-rich NCM cathodes are preferred now-a-days.

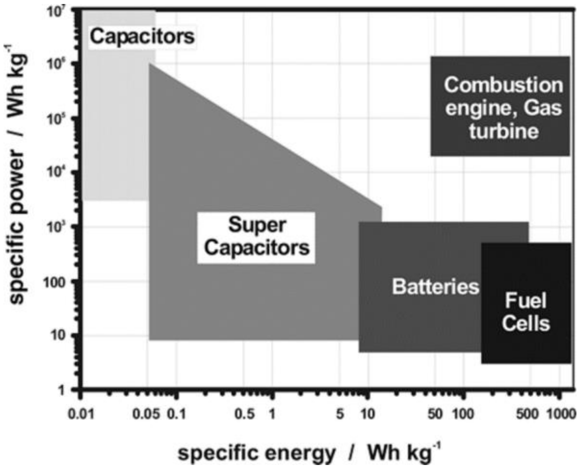

Figure 1. Simplified Ragone plot of the energy storage domains for the various electrochemical energy conversion systems compared to an internal combustion engine and turbines and conventional capacitors. Reprinted with permission from Winter and Brodd (2004).

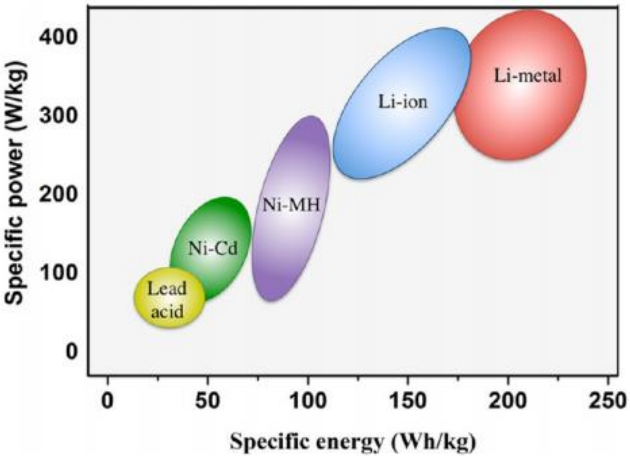

Figure 2. Ragone plot for various rechargeable batteries showing superiority of the Li-based battery systems as the desired energystoring devices. Reprinted with permission from Meesala et al. (2017).

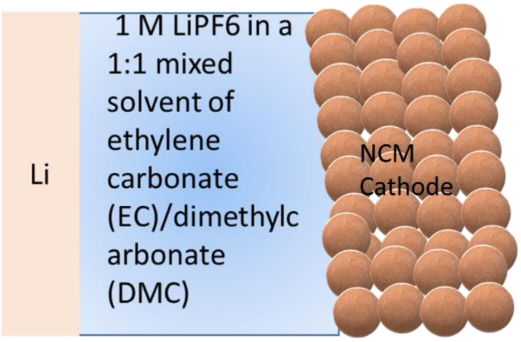

Figure 3. Schematic of Li ion battery based on a NCM cathode and a liquid electrolyte.

2. Li Rechargeable Thin Film Battery

Though seen as an alternative of combustion based energy devices, LIB faces several issues based on their components. The main concern is related with safety which is due to the presence of liquid electrolyte (Yu et al., 2023). For safety reasons, replacement of a liquid electrolyte with a solid state electrolyte (SSE) is considered an effective approach (Guo et al., 2022a). In addition to this, scientific community is rigorously working towards miniaturising energy storage devices. Thus, the development of appropriate SSE will pave the way of solid state batteries (Fang et al., 2023) or thin film batteries (Wu et al., 2021) which will not only be safe but also persists as a way of miniaturisation of energy storage device. A simple schematic of thin film rechargeable batteries is shown in Figure 4.

These batteries persist the way towards utilization of these storage devices for the applications depicted in Figure 5 (Jones et al., 2012). The use of these batteries for electrical vehicles (Deoff et al., 1995; Turcheniuk et al., 2021) and grid storage (Zhu et al., 2022) applications are other points to develop these batteries. Owing to these applications, tremendous growth is observed in the thin film growth market (Figure 6) along with the research activities related to the growth of SSEs (Figure 7).

Though, origin of thin film battery can be traced back to decade of 70s with the publication of reports related to SSE (Kennedy, 1977; Raleigh, 1967), however, their actual development could be realized in the mid of 90s with the growth of Li ion rechargeable thin film battery (Bates et al., 1994). In this context, the design of LIB based on $Li_2Mn_2O_4$ as the positive electrode, $Li_4Ti_5O_{12}$ negative electrode, and $Li_3PO_{4-x}N_x$ electrolyte is reported by Nakazawa et al. (2014). This battery shows a stable electrochemical behaviour upto 1000 cycle. A stable long-term cycling effect on thin-film lithium batteries (Li/LiPON/$LiCO_2$) of thickness 10 µm is also investigated. This battery leads to a capacity decline of 27% over 1040 cycles (Wang et al., 2022). The batteries consist of cells with crystalline $LiCoO_2$ cathodes and glassy lithium phosphorus oxynitride ('Lipon') electrolyte that can deliver up to 30% of their maximum capacity between 4.2 and 3 V at discharge currents of 10 mA/cm^2, (Bates et al., 2000). Madinabeitia et al. (2022) deposited a high-voltage, lithium-ion thin-film battery composed of $LiNi_{0.5}Mn_{1.5}O_4$ cathode, a LiPON solid electrolyte, and a lithium metal anode on low-cost stainless-steel current collector substrates. The gravimetric and volumetric energy densities of this battery were reported to be 333 Wh kg^{-1} and 1,212 Wh^{-1}, respectively. Aribia et al. (2022) reported growth of Li/LiPON/ $Li_{1.6}Ni_{0.8}Mn_{0.1}Co_{0.1}O_2$ (NMC811) thin film battery on a stainless-steel battery (Figure 8). These efforts of researchers pave up a way to design a thin film battery based on NCM cathode with a compatible SSE and with optimized interfaces (Hemmelmann et al., 2021).

Despite many efforts, commercial use of these batteries is still limited because of poor electrochemical performance. Poor performance of thin film battery is because of poor ionic conductivity through SSE (Yu et al., 2017; Wang et al., 2021), interface formation at cathode/electrolyte interface (Wood et al., 2018, Li et al., 2021, Zahiri et al., 2021) and formation of dendrites in electrolytes (Yu et al., 2021). Compatibility among the SSE and cathode is another major concern in these batteries (Gellert et al., 2018; Chen et al., 2021). These several issues hinder the actual development of thin film battery even though the efforts are still underway (Nagao et al., 2013; Hikima et al., 2022).

As mentioned earlier, LIB with NCM cathode seems to be most successful for electrical vehicle applications (Park et al., 2021a; Namkoong et al., 2022), however, it still lacks development in the form of thin film battery. Thus, researchers are giving specific attention to develop an SSE with impressive Li ionic conductivity (Uddin and Cho, 2018; Materzanini et al., 2021; Yang and Wu, 2022) and optimized cathode-SSE interfaces in Li ion thin film rechargeable battery (Banerjee et al., 2020; Lim et al., 2022). Though, progress towards the development of electrolyte thin films and interface optimization is receiving great attention but the least attention is given to design an appropriate cathode thin film. Hence, the proposed work is intended to design Co free Ni rich thin film cathode for LIB to work for long life cycle using radio frequency (RF) sputtering. Efforts are also extended to identify nature of SSE (Famprikis et al., 2019; Kim et al., 2022) and suppression of dendrites in SSE (Ji et al., 2020; Liu et al., 2021a). The development of cost effective and productive Li ion thin film battery technology is another aspect of thin film battery research (Clement et al., 2022). Some of these issues will be discussed in forthcoming sections

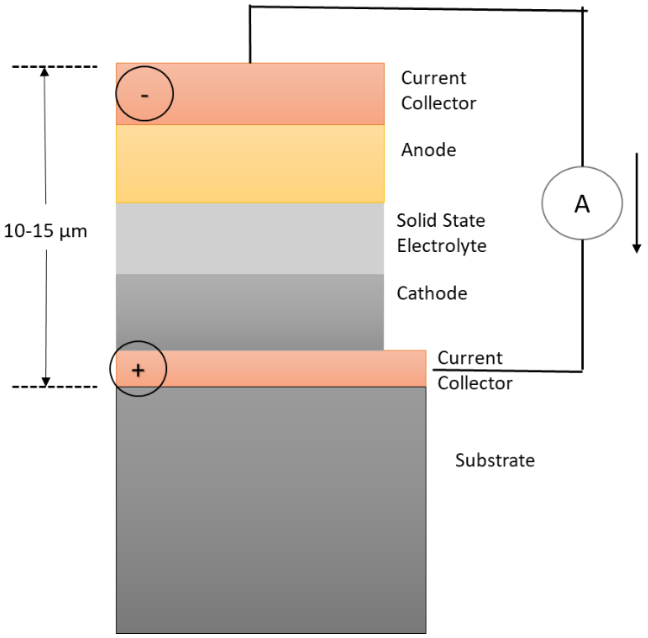

Figure 4. Various components of a rechargeable thin film battery.

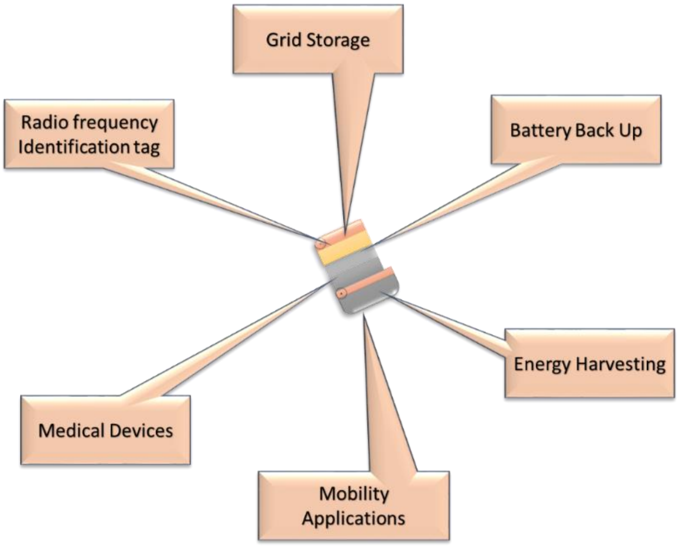

Figure 5. Application of thin film rechargeable batteries. Redrawn from reference Jones et al. (2012).

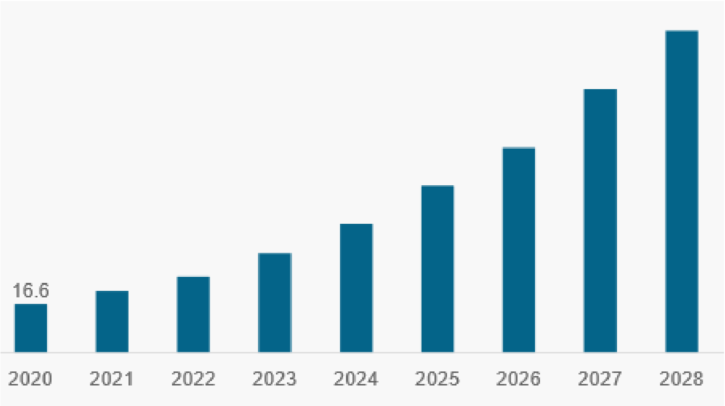

Figure 6. Projected market size of thin film battery by 2028, Applications of thin film battery. https://www.fortunebusinessinsights.com/thin-film-battery-market-106443.

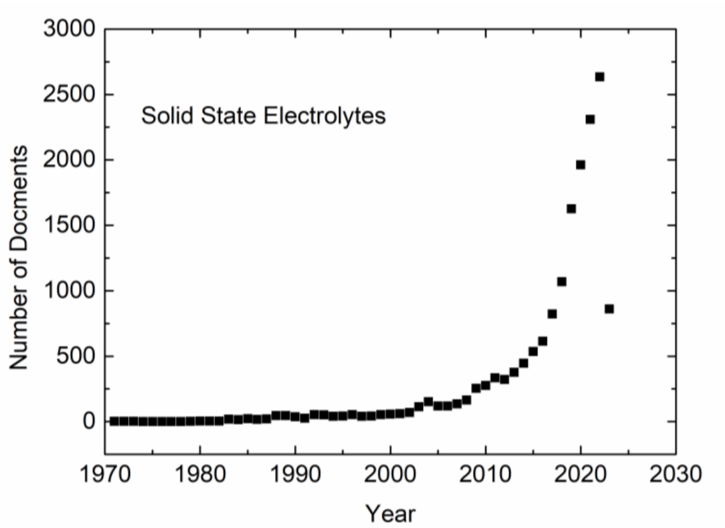

Figure 7. Research activities towards the growth of solid state electrolytes with time. Data were taken from Scopus 22/03/2023.

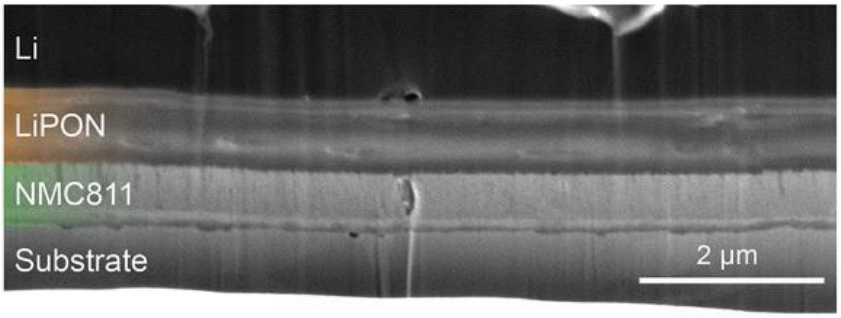

Figure 8. Cross-section SEM image of the all-solid-state battery stack (Li/LiPON/NMC811/Pt/Ti/sapphire); (Aribia et al., 2022).

3. Lithium Lanthanum Zirconate

The importance of thin film battery is reflected from the growth of various Li ion based such batteries by well-known groups as mentioned in the previous section. Though SSEs are able to miniaturise energy devices but they face the problem of ionic conductivity which affects the electrochemical performance of these batteries (Yang et al., 2022). Efforts to optimize the conductivity of these SSE's are under progress for numerous rechargeable batteries including LIB.

Researchers have identified $Li_7La_3Zr_2O_{12}$ (LLZO) as a potential material for LIB based on the work reported in past few years. Low ionic conductivity at room temperature (10^{-8} S cm^{-1}) and wide electrochemical operation window of 0–6 V (Kravchyk et al., 2022) are major reasons for its choice over other SSE's. It's compatibility with NCM structure, is another reason for selection (Roitzheim et al., 2022). Though, it exhibits moderate value of ionic conductivity and activation energy among several SSE's but these parameters are quite low compared to liquid electrolytes (10 mS cm^{-1} at RT). This limits the Li migration through the SSE and affects the electrochemical behaviour of the Li rechargeable thin film batteries.

Thus, numerous efforts are made by researchers to improve Li ionic conductivity of LLZO SSE (Lv et al., 2021). Some of these efforts are made by forming composites (Huang et al., 2019; Li et al., 2019), designing specialised morphology (Wu et al., 2020; Din et al., 2021; Kravchyk et al., 2022) and by changing method of synthesis (Sakamoto et al., 2013; Yang et al., 2020). Among these, doping remains one of the major efforts (Adhyatma et al., 2022; Wang et al., 2022). It is reported that LLZO doped by elements such as Ta (Chen et al., 2018), Ca (Chen et al., 2018), Al (Xia et al., 2016), In (Yan et al., 2021), and Ti (Shao et al., 2017) exhibits improvement in the ionic conductivity (Yang et al., 2017). The ionic conductivity shows a significant enhancement in the conductivity of LLZO with Ga doping (Sastre et al. 2020).

Though Ga ions reflect a higher value of ionic conductivity but it is still too low to compete with conventional liquid electrolytes. Thus, efforts to further improve ionic conductivity are carried out by substitution of dual/element doping (Xu et al., 2023). Ga/Nb ion dual substitutions result in high Li^+ ion conduction in LLZO (Lan et al., 2020). Cubic-phase $Li_{6.05}La_3Ga_{0.3}Zr_{1.95}Nb_{0.05}O_{12}$ exhibits ionic conductivity of 9.28×10^{-3} S cm^{-1} (Abrha et al., 2020). The ionic conductivity value of $Li_{6.65}La_{2.95}Sr_{0.05}Zr_{1.8}Mo_{0.2}O_{12}$ is 6.43×10^{-4} S cm^{-1} (Zhou et al., 2022). The total conductivity and activation energy for $Li_{6.925}La_{2.95}Y_{0.05}Zr_{1.925}Sb_{0.075}O_{12}$ are 3.20×10^{-4} S/cm and 0.30 eV (Cao et al., 2021). In addition to above, multi-element doping strategies are reported to improve significantly the ionic conductivity of LLZO (Shin et al., 2015; Meesala et al., 2019). The total conductivity, relative density and contractibility rate of multi-element doped SSE ($Li_{6.52}La_{2.98}Ba_{0.02}Zr_{1.9}Y_{0.1}Al_{0.2}O_{12}$) are 2.96×10^{-4} $S\cdot cm^{-1}$, 94.19% and 18.61%, respectively (Liu et al., 2021b).

4. Co free Ni rich Oxides

The content of Ni in well-known NCM cathode is favourable towards cumulative capacity (Ryu et al., 2001). Figure 9 shows that variation of cumulative capacity of LIB based on this cathode with Ni content. Compositions of this cathode such as $LiNi_{0.33}Co_{0.33}Mn_{0.33}O_2$ (NCM111), $LiNi_{0.4}Co_{0.4}Mn_{0.2}O_2$ (NCM442), $LiNi_{0.5}Co_{0.3}Mn_{0.2}O_2$ (NCM532), $LiNi_{0.6}Co_{0.2}Mn_{0.2}O_2$ (NCM622) and $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ (NCM811) are shown in the figure. One can see that highest capacity is observed for $LiNiO_2$ (LNO) when there is no Co and the least value is reported for $LiCoO_2$ (LCO). LNO despite the high value of cumulative capacity is reported to exhibit high-capacity fading. Thus, a complete elimination of Co is avoided from NCM as modified cathode exhibits behaviours similar to that of LNO (Sun et al., 2021).

It is reported that increasing Ni content promotes capacity fading along with the reduction in thermal stability (Noh et al., 2013). To overcome these effects, efforts are underway by utilising various strategies such as doping, coating, composite formation and change in morphology (Jiang et al., 2021a; Park et al., 2022b). Doping is considered as an important and effective way to control these factors in Ni-rich cathode oxides (Dang et al., 2022; Guo et al., 2022b; Park et al., 2023).

In NCM cathodes, cobalt is beneficial to promote the electronic and ionic conductivity (Chu et al., 2022). Mn ions are reported for the structural stabilization (Xie et al., 2017). Co ions are also required for structural stability as reported in recent work by Liu et al (2021c). Thus, the presence of Co and Mn ions are pertinent in the NCM structure in order to design stable NCM cathode (Thackeray and Amine (2021). But the use of Co ions is not considered favourable at industrial level as Co has issues related with the abundance and cost (Gourley et al., 2020; Li et al., 2020). Thus, efforts for developing next generation Ni-rich layered oxide cathodes with no Co are underway (Liang et al., 2020; Liu et al., 2022; Yu et al., 2022).

As a matter of fact, efforts are being made towards the understanding the role of Co ions in layered structures (Voronina et al., 2020; Liu et al., 2021d; Gent et al., 2022; Shen et al., 2022a), so that suitable ions for replacement of Co ions may be identified (Park et al., 2022). Considering, abundance and high price of Co (Brow et al., 2022), various research groups around the world are working towards designing Co free Ni rich layered oxide cathode by looking for a suitable ion to replace Co (Mu et al., 2022; Park et al., 2022).

In this context, Cui et al. (2021) reported that substitution of Zn ions enhances the cyclic efficiency of Ni-rich $LiNi_{0.9}Co_{0.05-x}Mn_{0.05}Zn_x$ cathode. Jeong et al. (2022) observed outstanding capacity retention in $LiNi_{0.9}Mn_{0.05}Zn_{0.05}O_2$ (NMZ955) synthesized by the co-precipitation method.

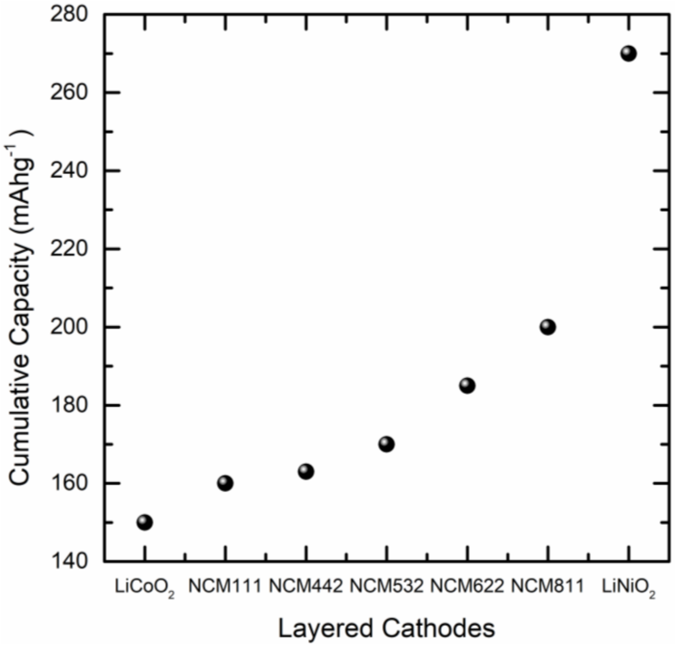

Figure 9. Variation of cumulative capacity with Ni Content. $LiCo_2$ (Blomgren, 2016), $LiNiO_2$ (Koutavarapu et al., 2020).

Mo introduction to Ni-rich layered cathode pave a way to design a Co-free Li-ion battery with cumulative capacity retention of 86% after 1,000 cycles (Shen et al., 2022b). Sattar et al. (2020) reported that 1 wt. %

molybdenum $LiNi_{0.84}Co_{0.11}Mn_{0.05}O_2$ delivers superior initial discharge capacity of 205 mAh g^{-1} (0.1 C), cycling stability of 89.5% (0.5 C) and rate capability of 165 mAh g^{-1} (2 C) compared to those of others. Yang et al. (2022) designed Co-free $LiNi_{0.9}Mn_{0.1}O_2$ with lattice Ge- doping and interface Li_4GeO_4-coating (NMGe). Ge doping reduced the cation mixing, suppress the oxygen loss and stabilize of the crystal structure. The NMGe delivers a high specific capacity of 223.3 mAh g^{-1} at 0.1C and 127.5 mAh g^{-1} at 10C. Tan et al. (2023) designed Co free Ni-rich $LiNi_{0.96}Mn_{0.04}O_2$ cathode material (NM) by doping with Al and Zr. Synthesized material exhibits better thermal stability and structural stability compared to NM material. Lv et al. (2023) reported that replacement of Co with Al and prolongs the cycle life of the Ni-rich Co-free cathode under high voltage (≥4.5 V) as well as high temperature (≥45 °C).

Though, efforts to design a Co free Ni rich oxide cathode material are in process in order to meet future challenges, however no attention is given to develop these cathode materials for thin film rechargeable batteries because of several issues owing to film growth (Patil et al., 2008; Clement et al., 2022) and their optimisation in terms of chemical compositions (Hikima et al., 2022). Considering the need, research developing thin films of NCM cathodes based on theoretical models is recently reported (Hemmelmann et al., 2021), however, growth of films with appropriate cathode/ especially Co free Ni rich cathode could not be reported till date as per the best knowledge of author. As this, requires optimization not only in the form of thin films but also for the sputtering target, thus a lot of efforts are needed. In this context, Jiang et al. (2021b) fabricated highly homogeneous $LiNi_{0.6}Co_{0.2}Mn_{0.2}O_2$ and $LiNbO_3$-coated $LiNi_{0.6}Co_{0.2}Mn_{0.2}O_2$ thin-film electrodes via RF sputtering deposition. Lithium-rich Li-Ni-Mn-Co-O thin film cathodes are deposited onto these modified stainless-steel substrates by non-reactive radio frequency (RF) magnetron sputtering from a ceramic $Li_{1.14}$ $(Ni_{0.37}Mn_{0.1}8Co_{0.32})$ $O_{1.19}$ target (Strafela et al., 2016). Thus, these studies reflect that efforts to develop Co free Ni rich oxide cathodes are continuing, however, no report related to growth of thin film cathode for these compositions are available. Even the fabrication of thin film cathode for Ni – rich oxide is at a very infant stage. Thus, a lot of efforts are needed in this direction in order to design a cost effective, and safe thin film battery for electrical vehicles and high energy applications.

5. Conclusion

Li thin film rechargeable batteries with NCM cathodes are considered suitable for electrical vehicle (EV) and other high energy applications. For a safe battery, SSEs over liquid electrolyte are preferred. However, SSE faces the challenges of ionic conductivity. In last five years efforts are made towards enhancing the cumulative capacity by increasing Ni composition. Cathodes designed with high Ni content are termed as Ni-rich layered oxide cathodes and achieve high value of cumulative capacity. However, considering the Co abundance design of Co free Ni rich oxides is an alternate. Research focussing on designing suitable Co free N cathode, may pave design of such cathodes. Though there is a significant increase in the thin film related research activities around the globe, but growth of appropriate film battery is still lacking. Thus, gap can be filled by providing an optimized way of growing thin film battery based on Li anode, LLZO as SSE and NCM as cathode.

Conflict of Interests

The authors declare no conflict of interest.

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