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A COMPREHENSIVE REVIEW ON LI-ION BATTERY AGEING ESTIMATION TECHNIQUES FOR GREEN ENERGY VEHICLES

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ABSTRACT
Over the last few years, research scientists have proved that lithium-ion (Li-ion) battery can successfully compete as a rechargeable battery for green energy vehicles (electric vehicles or EVs, hybrid electric vehicles or HEVs) because of its higher energy density and lighter weight. However, capacity fade and battery pack failures remain a hindrance to its maximum utilisation. Failure not only leads in huge replacement cost but also prospective safety concerns such as short circuiting or overheating which may induce fire accidents and it becomes aggressive with ageing. That is why ageing estimation, health monitoring in terms of state of health (SoH) and end of life (EoL) estimation ability in battery equipped systems are of major concern. This paper critique novel research and development in the field of Li-ion battery ageing estimation and health monitoring. Various models, techniques and algorithms have been presented along with their pros and cons. The intent of this review and discussion is to prepare an inclusive perspective on progress and challenges in monitoring Li-ion battery ageing.

KEYWORDS: 2 Li-ion battery, ageing, capacity fading, SoH, EVs, HEVs.

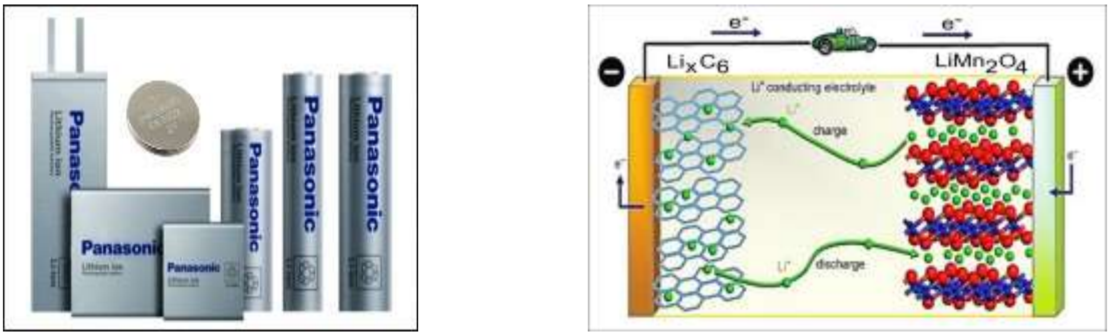
I. INTRODUCTION
Li-ion battery is a preferred choice of green energy vehicles and other automotive applications as an energy source [1]. As the applications are shifting from small-scale consumer electronics to dynamic energy/power application, the cycle life and performance of Li-ion battery are gradually becoming vital issues [2]. To create greater control over the performance, safety and cycle life of a Li-ion battery, smart monitoring for battery ageing is essential. It is specifically required for real-time applications (EVs, HEVs), where a larger power and energy demand along with an extended lifetime is critical [3,4]. Performance losses (e.g. power loss, energy loss, and capacity fade, etc.) over time is an existing problem, and extensive research has been focussing on understanding the ageing mechanism causing these losses. To promote researchers and their efforts to address the ageing issue, the United States Advanced Battery Council (USABC) has kept a goal of 10 years of calendar life of the battery for EVs and 15 years for HEVs [5]. Degradation in the health of the battery is measured in terms of SoH. We can overcome the above problems by applying effective SoH monitoring, prognostic methods and algorithms [6,7]. Li-ion batteries used for EVs/HEVs require more attention due to challenging driving behaviour, variable environmental conditions, unbalanced battery cells, and self-discharging etc. [8,9]. All of the above challenges make Li-ion battery a unique case, and it needs aggressive efforts towards research and development of accurate health monitoring method and algorithm [10,11].
The main objective of this review paper is to analyse different approaches, methods, and algorithms for estimating ageing of Li-ion battery in terms of its state of health. The following points distinguish this review article from the other.

- The authors try to collect and combine information about all models/methods and simulation approaches on a single platform. No other single review article provides all such information.
- Also, this review article reviews some novel approaches e.g. magnetic field probing for monitoring of battery ageing.

Section 2 defines ageing mechanism inside Li-ion battery, its origin and types. Section 3 defines different methods of estimating ageing with their benefits and drawbacks. Section 4 defines various software/tools used for battery diagnosis. Section 5 and 6 define discussion, conclusion and a proposed smart monitoring method for ageing respectively.

II. AGEING MECHANISM INSIDE LI-ION BATTERY

In 1912, G. N. Lewis initiated pioneering research work on the lithium battery. However, it took long to get a commercialised product. In 1991, the Sony Corporation commercialised the first Li-ion battery [12]. Figure 1a shows the different types of geometries of Li-ion battery [13] and Figure 1b shows the schematic representation of the working principle of the battery. During charging and discharging of the battery, Li^+ ions move between



(a) *(b)*

Figure 1. Li-ion battery (a) Different shapes of Li-ion batteries [13] and (b) Functioning.

through the electrolyte, and this process is named as intercalation process [14,15]. After various cycles of charging and discharging, the intercalation process slows down which reflects the ageing of the battery. During a battery lifetime, its health tends to deteriorate slowly due to irreversible physical and chemical changes like internal impendence rise, gassing due to the secondary reaction, loss of active Li^+ ions, internal temperature rise, electrolyte decomposition, loss of active electrode material and mechanical stress in electrodes [16,17], etc. Ageing phenomena occurring at electrodes (positive and negative) differ significantly. Therefore, these are explained individually [18]. Ageing indicators are summarised in the flowchart (Figure 2).

Ageing process at negative electrode

Carbons, lithium alloys, lithium titanate ($Li_4Ti_5O_{12}$ or LTO) and chalcogenides are the most prominent insertion compounds that have been proposed as negative electrode (anode) materials for Li-ion batteries [19]. Solid electrolyte interphase (SEI) layer is a protective layer for rechargeable Li-ion battery, which forms at carbon/graphite electrode [20,21].

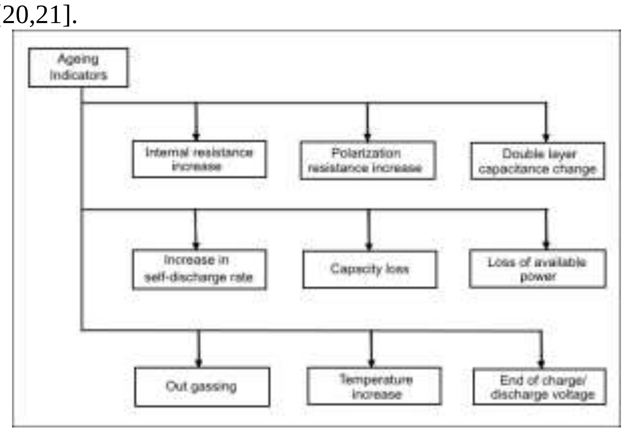


Figure 2. Ageing indicators chart

The deposition of the SEI layer is an important part of the formation process of the battery during the first few charging cycles. SEI layer protects the electrolyte material from further depletion and charged anode from corrosion because electrolyte material reacts vigorously with the anode material during the initial charging of the battery [22,23]. However, there is an initial capacity loss in Li-ion battery during SEI layer formation due to irreversible loss of Li-ions which is approximately 7-10% of the rated battery capacity. The ageing process provokes some parasitic side reaction which results in further loss of active Li-ions and reflects more capacity loss. The thickness of the SEI layer is not homogeneous and rises with age. Figure 3 shows the ageing effect on negative electrode due to SEI layer. It shows how SEI pores block and gas bubbles. In the case of the graphene flakes, the edges are very rough and have many entries in which gas bubbles can be formed, between the graphite

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mass and solid surface species [24]. The thickness of SEI layer rises battery internal impedance, reducing capacity and cycle life of the battery [25,26].

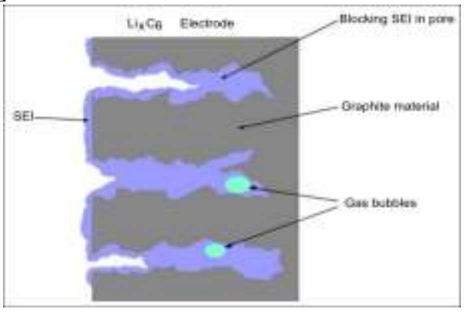

Figure 3. Schematic representation of ageing effect inside negative electrode [27].

Ageing process at positive electrode

Lithium metal oxides like $LiCoO_2$ (lithium cobalt oxide), $LiNiO_2$ (lithium nickel oxide), $LiMn_2O_4$ (lithium manganese oxide), $LiFePO_4$ (lithium iron phosphate), and advanced composite compounds like lithium nickel manganese cobalt oxide ($LiNiMnCoO_2$ or NMC), lithium nickel cobalt aluminium oxide ($LiNiCoAlO_2$ or NCA) are commonly used as positive electrode (cathode) materials in Li-ion batteries [28]. Cathode materials significantly affect both performances as well as calendar life and cycling of Li-ion batteries. Some current publications focus on the ageing of Li-ion batteries including cathode materials [29,30]. Mostly, capacity fading of positive electrode material can initiate from three processes: *chemical decomposition/dissolution reaction, structural changes during cycling and surface film alteration*. All these processes are described in Figure 4.

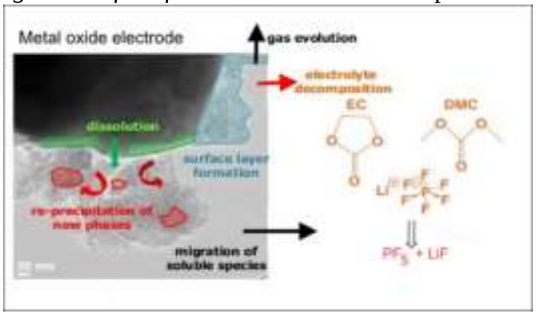

Figure 4. Schematic representation of ageing effect inside positive electrode [29].

Similar to the negative electrode material, degradation of positive electrode material also depends on the cycling conditions and other operating conditions [31,32]. So finally, the major consequences observed on an aged positive electrode are wear of active mass, electrolyte degradation, electrolyte oxidation and the interaction between positive electrode element dissolved within the electrolyte [33,34]. Table 1 summarises the root causes of ageing phenomena in the Li-ion battery.

Table 1. Summarised causes of Li-ion battery ageing.

Ageing Indicator	Cause 1	Cause 2	Cause 3
Energy loss	Active material transformation in inactive phase	Ohmic polarisation	SEI layer thickness growth
Power loss	Impedance growth		
Capacity fade	Li-ion loss/Li-metal corrosion	Side reaction (irreversible capacity loss) thermodynamically unstable lithiated carbon	Li-metal plating, clogging micropores, reduction of active surface area, and Gas evolution
Overpotential	Non-homogeneous electrode surface	Loss of active surface of electrodes	

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Self-discharge rate rise	Separator degradation	High SoC, and High temperature	Oxidation at cathode against electrolyte at high temperature

Beginning of Ageing inside Li-ion battery

Two distinguished ageing situations are:

- Calendar life on storage (i.e. on rest)
- Cycle life while in-use (i.e. on cycling)

Battery ageing occurs during storage due to self-discharge and impedance rise. It affects the calendar life of the battery. Ageing on storage mainly depends on storage time and storage conditions like temperature and state of charge (SoC). The ageing process during storage can be monitored by capacity fading, change in potential, internal resistance rise with SoC fade [35,36]. Sometimes, controlled charging/discharging cycles improve the capacity fade and recover a fraction of percentage faded capacity.

On the other hand, cycling process usually damages the materials' reversibility, the interaction between the electrolyte and active materials, that results in loss of cyclable Li-ions. Throughout cycling, one can measure the capacity fade, impedance rise and overpotential that influence the charge/discharge curves [37,38].

III. BATTERY AGEING ESTIMATION METHODS/MODELS

The ability to diagnose and identify an ageing mechanism for the Li-ion battery is an important and most challenging goal. Methods for measuring its state of health mostly rely on the chemical and physical parameters like battery potential, in/out current, battery temperature and internal impedance etc. These methods are categorised as below:

- Empirical and analytical modelling
- Equivalent circuit modelling
- Statistical model approach
- Direct measurement methods
- Electrochemical equation modelling
- Invasive methods

Empirical and analytical models

An analytical method is based on previous experimental data and predicts the future behaviour of Li-ion batteries. Empirical models can be parameterised without a comprehensive understanding of the electrochemical cell structure. Empirical models have the advantage of simplicity. The main disadvantages of these models are the inaccuracy of the measurements and not being able to produce a prediction of internal cell behaviour.

(a) Coulomb counting: V pop et al. [39] define Coulomb counting approach, in which the current flowing in and out of a battery is measured and integrated over time to determine the capacity of the battery. Capacity is an important parameter which decreases with ageing of the battery. Kong Soon Ng et al. [40], proposed enhanced coulomb counting method and evaluated the SoH as the ratio of the maximum releasable capacity (Q_{max}) to the rated capacity (Q_{rated}) of the battery i.e. SoH = Q_{max}/Q_{rated}. The SoH is calculated at two conditions: when the battery has been completely discharged and when the battery is fully charged. Larry W. Juang et al. [41] applied Coulomb counting method for the diagnosis of lithium iron phosphate battery. They have also included temperature parameter in the model for accuracy improvement. This approach can be used in BMS due to its simplicity and facility to apply with other methods. However, it requires a recalibration at regular intervals which is a difficult task in real time.

(b) Electrochemical impedance spectroscopy (EIS): It is a very powerful way to gain health information about energy/power sources e.g. batteries, fuel cells, and supercapacitors [42]. After various battery cycles the composition of the electrodes' active materials changes due to secondary reactions. Such change of material is reflected in battery impedance. The model can determine SoH as a function of the battery impedance. The electrochemical impedance of a battery characterises its dynamic behaviour. Impedance and battery ageing relationship vary with battery temperature, electrode polarisation, and corrosion of electrodes [43,44]. The advantage of EIS method is that it can be applied easily to different battery chemistries. At present this method has not been applied for on-line estimations e.g. in EVs and HEVs as it requires laboratory setup or significantly advanced hardware.

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(c) Kalman filter (KF): The KF approach is an optimal state estimator for the linear systems. An extended Kalman filter (EKF) is used for the nonlinear systems. In References [45–47], Professor G. L. Plett represented a complete solution for Kalman filter and extended Kalman filter theory for battery state estimation. The KF and EKF techniques are model-based techniques, if a suitable battery model is available then it leads to higher accuracy. One disadvantage of this approach is the computational complexity. Lack of stability is another issue that comes with EKF approach when the system is nonlinear [48]. Another improved approach as sigma-point Kalman filter (SPKF) is given by G.L. Plett

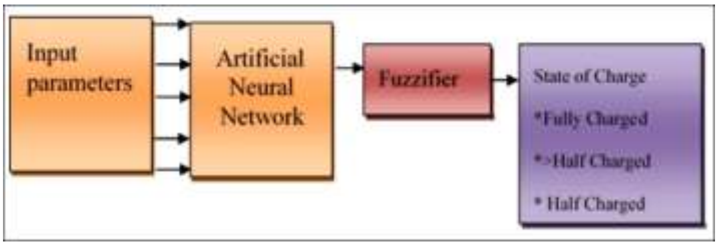

Figure 5. State of battery analysis using ANN and Fuzzy logic using five input parameters [56].

[49,50]. The sigma-points Kalman filter approach uses deterministic sampling points to capture the mean and the covariance of the estimated state vector. The mean and the covariance of the state vector can be estimated better by SPKF than by the EKF method.

(d) Fuzzy logic and Artificial Neural Network (ANN): The fuzzy logic and ANN approaches provide a powerful method of modelling complex and non-linear physical systems. The fuzzy logic approach considers the battery as a black box and simply maps the input characteristics of the battery to its output characteristics. It does not include any physical description of the fundamental physicochemical processes [51,52]. This technique's usage can help in using complete information about battery performance in order to derive its more accurate state of health estimation. The authors [53,54], applied the fuzzy logic methodology to Li-ion and Ni-MH batteries with EIS data. This data was pre-processed to bring out parameters that were used to develop precise fuzzy logic models for predicting SoH of the battery pack. The disadvantage of this approach is that it is not realistic for EVs and HEVs because the EIS data collection is not possible in a realistic environment. Neeta Khare et al. [55] show the capability of effective ANN system which is used to simulate the battery model integrated with the fuzzy logic approach. The author [56] shows that ANN training with five preferred parameters gives greater than 99% accuracy whereas the accuracy reduces with fewer number of parameters, leading to the conclusion that all five parameters are essential as shown in Figure 5. Mohammad Charkhgard et al. [57] applied a combination of neural network (NN) and EKF to estimate Li-ion battery states. The main drawbacks of NN approach are computational complexity and need to train a large amount of data.

Equivalent circuit-based models

These battery models are based on equivalent circuit theory and employ various methods to estimate model parameters [58–60]. For estimating battery ageing, model parameters are internal battery parameters like battery impedance, temperature, current, and voltage. These models can be implemented readily on low-cost microcontroller inside BMS. At present, the BMS for large Li-ion battery packs use the flavour of equivalent circuit-based models. The main issues of these models are the extraction of the parameters using experimental or manufacturer data and low accuracy as the battery becomes old. Liao Chenglin et al. [61] applied the following dynamic equivalent circuit model for $LiFePO_4$ based Li-ion battery (Figure 6).

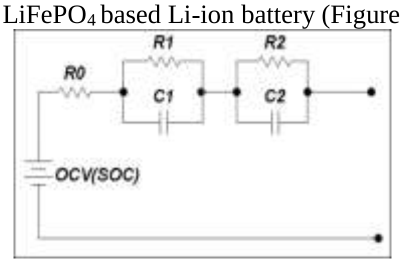

Figure 6. An example of dynamic equivalent circuit model for Li-ion battery state analysis.

(a) Support vector machine (SVM): The method of SVM was originally introduced by Vapnik [62]. SVM is a supervised machine learning algorithm which can be used for both classification and regression tasks. However, it is mostly used in classification problems. It performs classification tasks by constructing hyperplanes in a multidimensional space. Adnan Nuhic et al. [63] applied this method to build a battery health estimation model for EVs and HEVs. Yongqiang Chen et al. [64] proposed another method of weighted least squares support vector machine (WLS-SVM) to create the relationship of the state of charge with the cell potential, current and temperature. A good SVM regression model needs sufficient collection and tuning of empirical parameters, which

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is a time-consuming process. Hong-Zhong Huang et al. [65] applied SVM to estimate remaining useful life (RUL) of a battery. One of the major limitations of SVM method is the inadequacy of probabilistic outputs.

(b) Relevance Vector Machine (RVM): The RVM is a Bayesian form representing a general linear model of the SVM and applied for RUL prediction [66]. The fundamental idea is to build a probability density function (PDF) of the state, depending on all existing battery information. A comprehensive discussion of SVM and RVM is available in reference [67]. Bhaskar Saha et al. [68] have developed a model using battery internal parameters derived from an electrochemical model based upon RVM regression approach. The theory was that the internal parameters, e.g. electrolyte resistance and charge transfer resistance, would change steadily as battery deterioration proceeded and RVM model tracked the deterioration process accurately. The main advantage of RVM is that it is more generalised as compared to SVM and has low computational cost. The disadvantage of RVM is that the SVM parameters influence the results prediction.

(c) Particle Filter (PF): Particle filters are a type of non-linear filters that combine Bayesian learning methods with sampling and can contribute good state estimation while maintaining the computational load controllable [69]. M. Dalal et al. [70] proposed a battery model based on PF. The authors used parameters like non-linear open circuit potential, internal temperature, current, cycle numbers. A statistical estimate of the system noise and the expected operational conditions are processed to provide estimated RUL. Its advantage is high accuracy in short-term prediction and good adaptive ability but suffer from sample degeneracy.

Statistical models

Statistical models need an extensive data set. These models do not use any chemical or physical formulation and do not require any analytical information on the ageing mechanism [71].

Autoregressive Integrated Moving Average (ARIMA): This technique consists of two components: a disturbance component and a self-deterministic part. James D. Kozlowski et al. [72], represent the application of Autoregressive Moving Average (ARMA) for battery health estimation. Inputs are fed into a 2nd order ARMA model to calculate the battery prognostics. The precision of an ARMA model relies on representativeness and comprehensiveness of the past data used. So, it is not suitable for EVs applications, where past data is usually inadequate. Recursive model training and updating are essential for data, to make a realistic estimation. Yapeng Zhou et al. [73] applied ARIMA model along with empirical model decomposition (EMD) technique to predict the RUL of the Li-ion battery. The authors found that their model is more accurate than RVM method to predict battery ageing.

Direct measurement (Non-invasive) methods

Non-invasive methods use real-time sensors to measure the battery parameters. These battery parameters are related to SoC and SoH of the battery. Some of the non-invasive methods are:

Quantum magnetism (Q-Mag) and magnetic field probing (MFP): A method developed by Cadex that measures the SoC of the battery named as Q-Mag. It uses the measurement of susceptibility or magnetic field [74]. The battery is polarised by an external source, and the resultant magnetic field is measured. Figure 7 shows a relation of SoC and magnetic field for LA battery [75]. Similarly, in Li-ion battery intercalation/deintercalation process alters electrode structure thereby, modifying its magnetic field susceptibility [76–79].

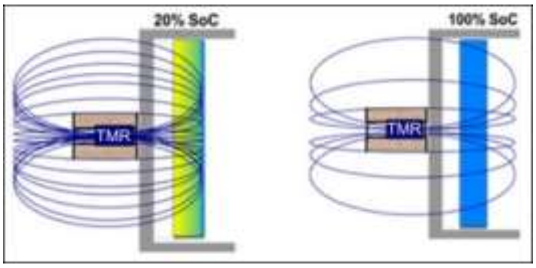

Figure 7. SoC estimation by magnetic field response courtesy of Cadex.

The sensor developed by Magna-Lastic Devices Inc. USA measures the variation in the magnetic field due to change in SoC and also monitors the SoH of the Li-ion battery [80]. Neeta Khare et al. [81] used an approach based on induced electromotive force (emf) in a secondary coil as a measure of battery behaviour under an applied magnetic field. The variation in secondary coil voltage indicates the battery behaviour and its internal health when the battery is subjected to an applied AC magnetic field. The main advantages of this technique are low cost, non-invasive, contactless and robust.

Electrochemical models (physical models)

Electrochemical models (physical models) provide complete information about battery parameters on performance and conditions e.g. voltage, current, temperature, electrolyte's concentration, corrosion, etc. They need specific knowledge of the physical and chemical properties of the battery e.g. electrolyte volume, density

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and porosity of the active materials [82]. The advantage of electrochemical models is that they include all internal battery behaviour parameters inherently for more precision. These models are too complex to use, and still, their implementation on an embedded system for BMS is questionable. Electrochemical models are further categorised as below:

Phenomenological approach

Physical models for battery health estimation started about twenty years ago vary in terms of complexity and computational requirements. Professor John Newman at the University of California published the first mathematical models capable of simulating the performance of Li-ion battery at the beginning of the 1990s. These models were based on well-proven electrochemical and thermodynamic concepts, and they described the processes that take place in the battery during operation. Few models based upon phenomenological approach are as follows:

(a) Single-particle models (SPM): The SPM includes the effects of transport phenomena in a simple way. A model of electrolyte diffusion and ion intercalation inside a single electrode particle was developed [83]. In this model, a detailed distribution of local concentration and potential in solution phase are ignored to increase computational run time without compromising accuracy. The SPM is very simple and fast in the simulation. The only drawback is its validity for limited conditions, like thin electrodes and low rates.

(b) Pseudo-two-dimensional models (P2D): The P2D model includes diffusion in the solid-phases and electrolyte as well as Butler-Volmer equation. A P2D based model was developed on concentrated solution theory to define the internal behaviour of a Li-ion battery consisting of porous electrodes with current collectors and a separator [84]. The model is represented by coupled nonlinear partial differential equations (PDEs) that may take from seconds to minutes to simulate and is based on the theories of electrochemistry, transport phenomena, and thermodynamics [85]. Ali Jokar et al. [86] provide an exclusive review on electrochemical modelling approach (SPM and P2D).

(c) Multiphysics Models: This is a complex approach used in system modelling. It includes multiscale, multidimensional and multiphysics electrochemical coupled models. Generally, these models accurately define all of the essential phenomena that take place during the operation of an electrochemical multiphysics system like battery [87–89]. A 2D thermal-electrochemical coupled model is presented by Long Cai et al. [90] for Li-ion battery that considers the effects of local heat generation. This paper also covers the battery discharging performance at different operating temperatures.

Atomistic and molecular approach

The approach uses essential properties of electrodes and electrolyte materials. These models are harder to design and recognise. Models based on atomistic and molecular approach have been developed and are as follows:

(a) Kinetic Monte Carlo (KMC): It is a random methodology used to analyse the discharge behaviour of Li^+ ions during the process of intercalation. KMC based models have been applied in [91,92] to simulate the diffusion of Li^+ ions within an electrode to understand how different crystal structures affect Li^+ ions mobility. It is also used to predict thermodynamic properties of the material [93]. Ravi N. Methekar et al. [94] applied KMC method to simulate the parasitic growth of the SEI layer over the surface of the electrode particle to analyse the capacity fading mechanism. The authors found that at slow discharge rate KMC calculations become expensive.

(b) Density functional theory (DFT): DFT calculations can be used to achieve analytical result about the structure and function of the battery electrode materials. Ken Tasaki et al. [95] used DFT-based calculations to define the electronic structure and lattice properties of graphite within LiC_6 electrode. Ki Chul Kim et al. [96] applied DFT calculations to examine the effect of lithium binding and Quinone property for positive electrode stability. One of the greatest challenges of physical models is there solving and convergence complexity.

Invasive methods

In situ investigations of Li-ion batteries for battery ageing estimation have been proved to be extremely insightful but have some challenges like the electrochemical cell must be completely compatible with testing method conditions. In situ techniques are very attractive because they provide continuous data during electrochemical cycling and avoid the problems of relaxation and contamination. Significant progress has been made with more advanced, new in situ techniques in the past few years [97,98].

The functioning of batteries is a complicated electrochemical system where various physical and chemical processes occur e.g. change in volume, phase transitions, parasitic side reactions, etc. These processes can be monitored by in operando and in situ measurements and therefore allow to link these processes directly to the electrochemical response of the battery. In operando process represents a special case of in situ research, where the battery is in operation, i.e. is being discharged during the process, allowing measurements of non-equilibrium states. Herein, a comprehensive overview of in situ methods for studying and diagnosing Li-ion battery's ageing is provided which emphasises on recent developments and stated experimental highlights.

X-ray techniques

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(a) X-ray diffraction (XRD): In XRD method, a scattering of X-rays due to sporadically spaced atoms produces a diffraction pattern. This pattern helps in collecting information about crystal's structure. During battery diagnosis, XRD technique is used to observe the structural evolution in an electrode as the electrochemical processes take place. To conduct XRD measurements during the battery operation, an X-ray translucent window needs incorporating into the design. This window allows the X-rays to reach the electrodes under investigation [99]. This setup allows incident X-ray beam to penetrate and is also vulnerable to air and moisture contamination. Misra et al. [100] applied XRD on the delithiation mechanisms in Si nanowires and also analysed parameters which reduce the cycle life performance of Si nanowire electrodes. Figure 8 shows a general view for in situ X-ray techniques. XRD method is used for post-mortem analysis of the battery.

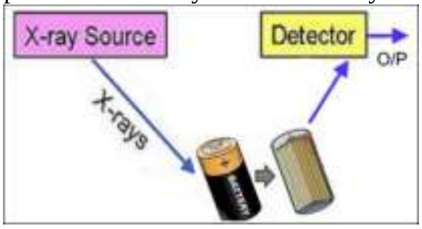

Figure 8. A general view for in situ X-ray techniques.

(b) X-ray absorption spectroscopy: This technique helps in analysing the structure and geometry of material, associated with the redox reactions that take place in batteries. Alexander W. Brownrigg et al. [101] applied XAS technique to analysis cathode material for Li-ion battery. Authors observed the stability and other ageing parameters for the Li_2FeSiO_4 cathode material. P.T. Kristiansen et al. [102] employ XAS to study redox behaviour of Li_2MnSiO_4 cathode material during cycling for better capacity performance.

Some advanced forms of X-ray microscopy are also used to investigate Li-ion batteries e.g. TXM [103] and X-ray tomographic microscopy [104]. These techniques are used at nanometre scale visualisation.

Magnetic resonance methods

(a) Nuclear magnetic resonance spectroscopy (NMR): This method is based on the nuclear element's magnetic resonance properties. NMR is an essential tool to probe the structural changes that occur in electrode materials. Sample electrode of the battery is placed inside NMR tube, and a strong static magnetic field is applied. Battery diagnosis setup is shown in figure 9. Minor variations in the monitored resonance frequencies provide elaborative information on the electronic environment around the sample electrode's nucleus and yield information about the lithium environment and electrochemically induced structural changes of electrodes during or after cycling [105]. The metal parts normally present in a battery such as casings, current collectors and metallic coatings in cells, shield the electrodes from the applied field. Plastic and cylindrical cells have been successfully employed to avoid this problem [106]. NMR has shown to be an adequate technique to investigate anode, cathode and electrolyte materials for ^{7}Li [107,108]. Rangeet Bhattacharyya et al. [109] were able to identify the mass of dendrite/moss lithium micro-scale structure developed during cycling process and monitoring lithium dendrite formation using NMR. Rémi Castaing et al. [110] applied NMR spectroscopy for SEI layer interphase for aged Li-ion battery and analyse parameters for capacity loss.

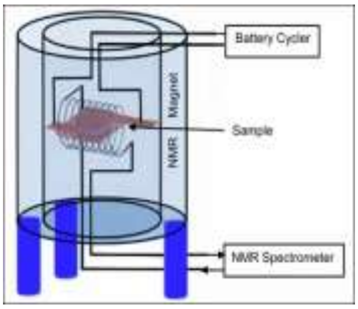

Figure 9. NMR spectroscopy setup for in-situ battery diagnosis (modified from [105]).

Nowadays, several in situ NMR studies have been conducted, measuring Li-ion concentration profiles [111,112]. S. Chandrashekar et al. [113], also measured a number of deposited Li microstructure during electrochemical cycling.

(b) Mössbauer spectroscopy (MS): Mössbauer spectroscopy is a nuclear interaction method that works as a function of gamma rays. Theoretically, several isotopes relevant for Li-ion batteries can be investigated by MS, but most research has been restricted to ^{57}Fe and ^{119}Sn Mössbauer experiments. The authors [114,115] used MS technique for in-situ operando studies of Li-ion batteries for the characterisation of electrode materials and the analysis of electrochemical reactions.

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Probe microscopy

Atomic force microscopy (AFM): The AFM is a suitable tool for Li-ion batteries ageing analysis because high-resolution images of the electrode (cathode) surface are obtainable [116]. This approach is based on the deflection of a cantilever with a high-pitched tip to examine surfaces. By scanning the interface between the electrode and the liquid electrolyte, a distribution pattern is obtained. Therefore, changes in volume and SEI formation during cycling can be visualised [117]. The critical challenge of an in situ AFM design is the access of the cantilever into the battery. All measurements are often performed under inert atmosphere inside a glove-box to avoid contamination. A detailed discussion about in situ AFM cells can be found in [118]. Jonghyun Park et al. [119] proposed a design in which the Li metal auxiliary electrode was used inside the electrochemical cell and the cell contained two electrodes as shown in (figure 10). Similar to AFM, researchers also used other adopted forms of probe microscopy for advanced study of battery ageing like electrochemical strain microscopy (ESM) [120] and scanning ion conductance microscopy (SICM) [121].

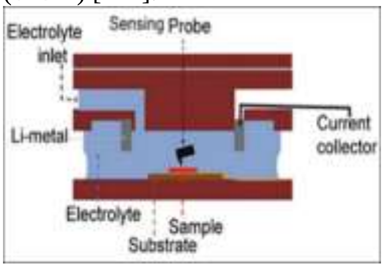
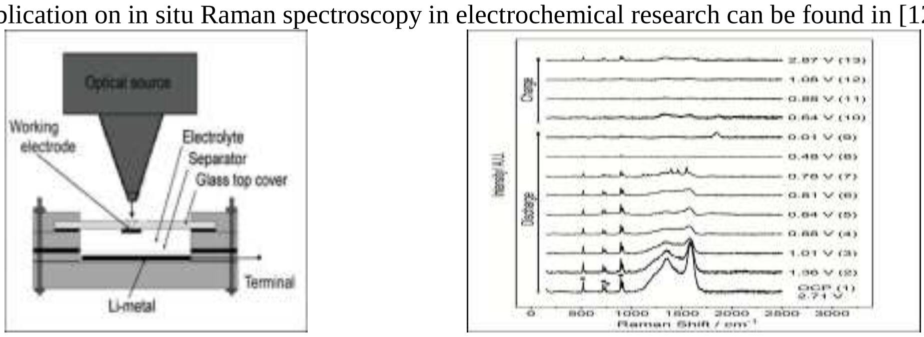


Figure 10. A detail view of AFM technique (modified from [119]).

Optical methods

(a) Raman spectroscopy: Raman spectroscopy is a non-destructive characterisation technique that is able to detect structural variations on the atomic level. This method is founded on inelastic scattering phenomenon of monochromatic light when it interacts with the sample. An optical pathway is to be created for laser light to react with the electrode. Usually, an opening window with a piece of thin glass in the outer casing is designed in the in situ cell as shown in figure 11a, by that the laser light may go through. By Raman spectroscopy, researchers can analyse different crystalline information and structural changes of electrodes during the cycling process. An extensive publication on in situ Raman spectroscopy in electrochemical research can be found in [122].



(a) ***(b)***

Figure 11. Raman Spectroscopy details for in situ electrochemical cell (a) Schematic view (modified from [122]) and (b) Result for in situ Raman spectra of ZFO-C electrode collected at different stages of the discharge and charge cycle in LiPF6 in 1:1 EC/DMC electrolyte [123].

Laura Cabo-Fernandez et al. [123] applied Raman spectroscopy to study the SEI formation and evolution for Li-ion battery during its cycling process, and the result is shown in figure 11b. It represents Raman shifts/peaks associated with electrolyte. These peaks represent the lithiation/delithiation process during charging and discharging profile.

(b) Fourier transform infrared spectroscopy (FTIR): This method is based on infrared light absorption instead of scattering and is similar to Raman Spectroscopy. Its drawback is surface's sensitiveness. Therefore, this is commonly applied to study the interfacial reactions between a working electrode and the electrolyte in the reflective mode. It is also used in identifying gas products formed during the reduction or oxidation of the electrolyte in the transmission mode. Another drawback is positioning the IR-window closest possible to the working electrode in order to limit the absorption of light by the electrolyte between the electrode and window. FTIR research has been described extensively for different battery setups in [124,125]. Some advantages of optical in situ methods are required low-cost instruments, equipment and easy data processing.

Electron microscopy

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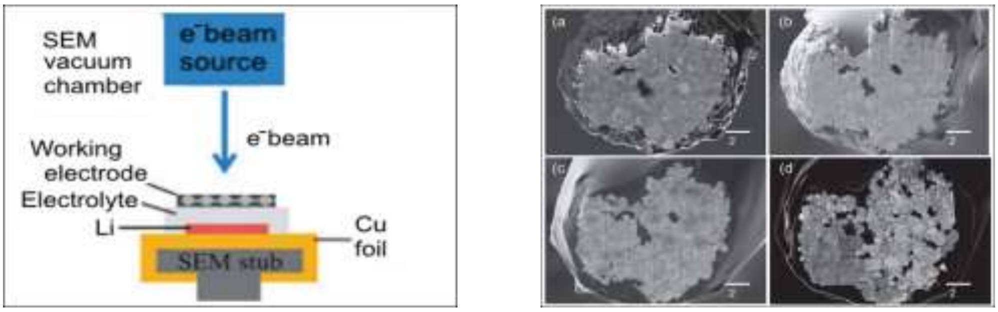
(a) Scanning electron microscopy (SEM): In SEM technique, an electron beam focuses on a specimen and either the secondary electrons or the back-scattered electrons emitted by the available atoms get detected (figure 12a) [126].



(a) ***(b)***

Figure 12. SEM microscopy details (a) Schematic view of SEM microscopy (modified from [126]) and (b) SEM images showing the progression of particle fracture and fragmentation as a function of cycle [127].

The technique offers an excellent spatial resolution to investigate morphological changes during battery operation such as electrode volume expansion/contraction, electrode cracking, etc. In [127], Dean J. Miller et al. applied SEM method to diagnose Li-ion batteries and results are shown in figure 12b. The figure shows the particle fracture progress after discharging/charging cycles.

(b) Transmission electron microscopy (TEM): This is similar to SEM. With TEM, electrons that transmitted through the electrodes and electrolyte are detected and build a nanoscale image. So, only nanoscale batteries can be investigated for the structural changes that can be monitored during battery operation (Figure 13).

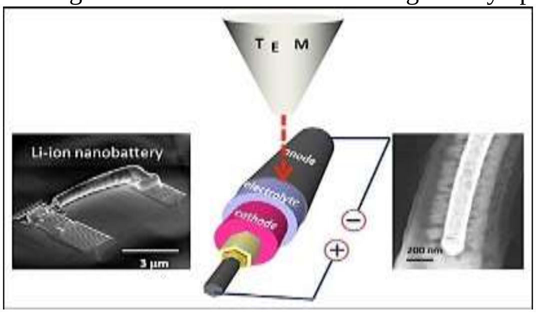


Figure 13. Schematic view of TEM methodology (modified from [128]).

Raymond R. Unocic et al. [128] applied TEM in situ method for SEI formation and evaluation for a liquid Li cell. The authors observed the dynamic self-healing nature of the SEI layer with changes in cell potential. Patricia Abellan et al. [129] used in situ TEM for analysing degradation mechanism inside electrolyte solution for Li-ion battery.

(c) Electron holography: In electron holography, the sample is exposed to an electron beam whose phase will be modulated by the electrostatic potential and magnetic field across the sample [130]. The signal coming from the sample and a reference signal interfere with each other, which results in a hologram (interference fringe pattern). The authors [131–133] applied this electron holography technique to map the electric potential distribution through the cathode/solid electrolyte interface during charging/discharging process as well as tracked anode side reactions.

Neutron-based methods

(a) Neutron reflectometry: Neutron reflectometry inculpates directing a highly focused neutron beam onto a surface and observing the intensity of the reflected radiation in terms of neutron wavelength or function of angle. It permits the thin film growth monitoring such as SEI formation and volumetric changes that battery operation induced [134,135]. In [134] Jeanette E. Owejan et al. determine the depth profile of the scattering length density (SLD) by adjusting the strength of reflected neutrons. Figure 14 shows the SLD as a function of depth for the SEI deposited on Cu. It also indicates the growth of the SEI thickness. As a result of neutron's large penetration depth, conventional coin cells and commercial cylindrical batteries can be used for in situ reflectometry research.

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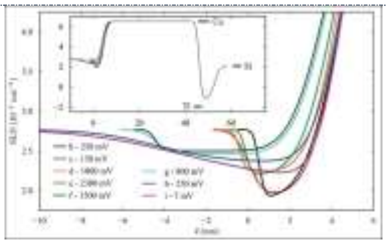

Figure 14. Neutron reflectometry for measurement of SEI thickness in terms of SLD [134].

(b) Neutron depth profiling (NDP): This method is based on bombarding the sample with thermal neutrons of energy about 25 mev. Lithium material, which has a high neutron absorption cross-section, absorbed these thermal neutrons resulting in alpha and triton particles emission. The authors [136,137] used in situ NDP for Li-ion batteries diagnosis. The study illustrates NDP as a powerful source to probe the concentration of lithium ions throughout thin film battery.

(c) Neutron diffraction: This technique is similar to XRD. Its disadvantage is the weaker interaction of neutrons, so larger penetration depth is required for better results. Another disadvantage of this technique is this being very sensitive towards the hydrogen existing in liquid electrolytes and the separators of commercial batteries giving rise to large background signals from the neutrons which are incoherently scattered [138]. Prabeer Barpanda et al. [139] used neutron diffraction technique for cathode material study.

IV. SIMULATION TOOLS USED FOR MODELLING

The main simulation tools used for simulating the Li-ion batteries ageing models are:

- DUALFOIL software uses Newman's based subroutine [140]. Which uses a finite difference method (FDM) to simulate electrochemical systems for more than four decades. It is freely available open source tool based on FORTRAN code.
- Microsoft Excel and MATLAB (Simulink and Simscape) software are used to simulate the empirical or analytical based models [141]. Maple, Mathematica, and Mathcad tools are also used [142,143].
- COMSOL Multiphysics and Battery Design Studio [144,145] software implement the FDM/FEM in an easy to use interface and include a package that implements the P2D battery model and Multiphysics model.

V. DISCUSSION

From electrochemical to direct estimations, different types of models/methods present to monitor the level of battery ageing and each of them has its pros and cons.

Methods comparison: Direct measurements do not require battery hypothesis since this is a direct measurement based estimations. Methods such as electrochemical models and equivalent circuit models perform well under the limited range but cannot be extended directly to other batteries' technology. In addition, both equivalent circuit-based models and electrochemical-based models rely on the accuracy of models. Direct measurement methods also miss electrochemical information about battery ageing due to the absence of appropriate probing. With the constantly improving technologies and new research findings, innovative technologies are frequently coming in the market to improve the probing methods. As yet, Electrochemical and equivalent circuit models fail to adopt the new technology with significant changes in the base model. However, physical and electrochemical models are dominant tools to recognise the ageing phenomenon under the limited range.

On the other hand, statistical models are easily adaptable to different batteries' chemistry, and they provide ageing results in real time. However, these models require a significant amount of data for an estimation of battery ageing which is a time-consuming process to collect data for entire battery life. In situ/invasive methods provide deep analysis about Li-ion battery's electrochemical behaviour during its cycle life. However, these techniques require complicated and expensive laboratory setup.

In general, there is a large requirement of a non-invasive, on-line method that can provide direct measurements for battery ageing diagnosis. It raises a demand of novel probing signal that can 'see through' the degradation of electrodes and electrolyte, loss of Li-ions, SEI layer thickness, electrode surface phase changes, out-gassing and passivation layer formation due to secondary reactions. Such method certainly needs a direct measurement of electrochemical parameters. MFP looks promising and fits with above requirements. Table 2 gives a classification of these methods on the basis of five major aspects.

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Table 2. Comparison of Li-ion battery ageing estimation methods/models performances on five prime aspects.

Methods, models	Adaptation	Accuracy	Real-time performance	Run without data	Prediction
Empirical and analytical modelling	Very poor	Good	Poor	Poor	Poor
Equivalent circuit modelling	Very poor	Fair	Good	Good	Fair
Statistical modelling	Fair	Good	Good	Very poor	Good
Direct measurement methods	Excellent	Excellent	poor	Excellent	poor
Electrochemical modelling	Very poor	Excellent	Fair	Fair	Fair
Invasive methods	Very poor	Excellent	Very Poor	Excellent	Good

VI. CONCLUSION

Since Li-ion batteries are the common and leading energy sources for green energy vehicles especially used for EVs, their performance highly effects the performance and economical proposition for EVs and HEVs. Hence, the battery manufacturers are looking for a breakthrough in both battery technology, and battery diagnosis approaches. Chemical processes in the battery are highly dependent on the operating conditions. Therefore, the degradation of a battery may differ in different operating environments. As a result, it is critically essential to have a precise estimation of the battery health and the time window where battery works properly. In this review paper, we have presented novel research and development in health estimation and deep ageing diagnosis on Li-ion batteries. Different methods, models, algorithms and techniques were discussed for battery ageing estimation. We hope that this paper will be helpful for engineers in need of a general overview of different diagnosis tools; towards getting a wider perspective on both challenges and progress of Li-ion battery health monitoring. The system engineers working for green energy vehicles may also gain a broad knowledge from this review article as following:

- A quick glance at the ageing mechanism of Li-ion battery and its causes
- Use of mathematical modelling in BMS making it smarter to monitor battery ageing
- Compare the various ageing monitoring techniques to re-design the existing BMS

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