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Composite Nanofibers as Advanced Materials for Li-ion, Li-O_2 and Li-S Batteries

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ABSTRACT

The quest to increase the energy density and improve the cycle life performance of lithium ion batteries (LIBs) and beyond has led to the development of various suitable and alternative materials for energy storage and conversion. The morphology and electrode architecture in advanced battery materials have been re-designed into nanofibers and composite nanofibers. Nanofiber-based separators and electrodes have demonstrated to improve the energy density and cycling performance of LIBs. The improvement in the structure and morphology of nanofibers in LIBs such as LiSi and LiSn, have once again ignited the interest in these Li:M alloy anodes as alternative anode and cathode materials. The major challenges that confront this new frontier have the lack of scalable method among the various techniques and designing nanofibers with good structure and morphology that could prevent dendrite penetrations. However, there seems to be a solution in sight with the advent of mass production techniques such as electrospinning and Forcespinning® that have recently been developed. In this paper, the use of nanofibers and composite nanofibers as electrode and separator materials for lithium ion, Li-O_2 and Li-S batteries is reviewed. The discussion focuses on the performance characteristics of these nanostructured electrode and separator materials and methods used to improve their performances.

Contents

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1. Introduction

The use of nanostructured materials for energy storage devices has been the trend in recent years. Nanostructured fibers made from metals, metal oxides, polymers, ceramics, and composites have been developed for use in various energy storage devices such as high-performance rechargeable lithium (Li) batteries (e.g. Li-ion, Li-sulfur, and Li-air batteries), sodium (Na), magnesium (Mg), aluminum (Al) and zinc (Zn) batteries and supercapacitors[1–18]. The popularity of these nanofibers stem from many attributes such as; controllable fiber diameter, high surface area- to-volume ratio, low density, and high pore volume [6,19–21]. These properties make the nanofiber structure more advantageous when used in LIBs as they deliver superior electrochemical properties; stable cycle performance, enhanced capacity, and better low temperature performance [21–24], when compared to their powder, crystal, nanowire, thin film, etc. counterparts[25–29]. The sterling properties/performance of nanofibers have seen wide applications in lithium ion batteries (as electrode and separator material), supercapacitors, and chemical sensors [30–33]. There are several methods employed to produce these nanofibers, with the most common including; electrostatic charge (electrospinning), external heated air jets (melt-blowing), bicomponent fiber spinning, phase separation, template synthesis, self-assembly, chemical vapor deposition, wet chemical synthesis, Nozzle-Free centrifugal or rotary jet-spinning, and the emerging Forcespinning method that was developed recently by Sarkar, Lozano and coworkers at UTPA (now UTRGV) [34–43].

Non-woven fibrous mats that have high surface area are typically in the range of a few microns to about 200 nm in diameter. Many efforts have been made to further reduce the fiber diameter into the nano scale [44]. This has been demonstrated through methods such as electrospinning, centrifugal spinning, and Forcespinning® (Fig. 1a) that can produce fibrous mats with thickness of 10–20 μm and fiber diameters that are typically less than 5 μm [36,38,45–48]. These fiber processing methods have been successful in giving the nanofibers their high labyrinth-like porous structure. These methods have significantly increased their applicability and ability to scale up for mass production of nanofibers due to their simplicity, high efficiency, low cost, high reproducibility andcomparatively environmentally benign [35,46,49]. There is considerable work on the use of structured composite nanofibers with high surface and interfacial areas to improve the performance of electrodes and separators in lithium ion batteries [1,2,4,6,10,25,50–53]. While there are several topics discussed in this article on the application of structured nanofibers, the main thrust of this review is to discuss the use of nanofibrous materials as electrodes and separators and their electrochemical performance in the lithium ion batteries.

2. Nanofibers (NFs) Production Techniques

Non-woven fibrous mats are typically thick (i.e. 100–200 μm), however, in effort to reduce this thickness, recent fiber manufacturing technology such as electrospinning(Fig. 1b),

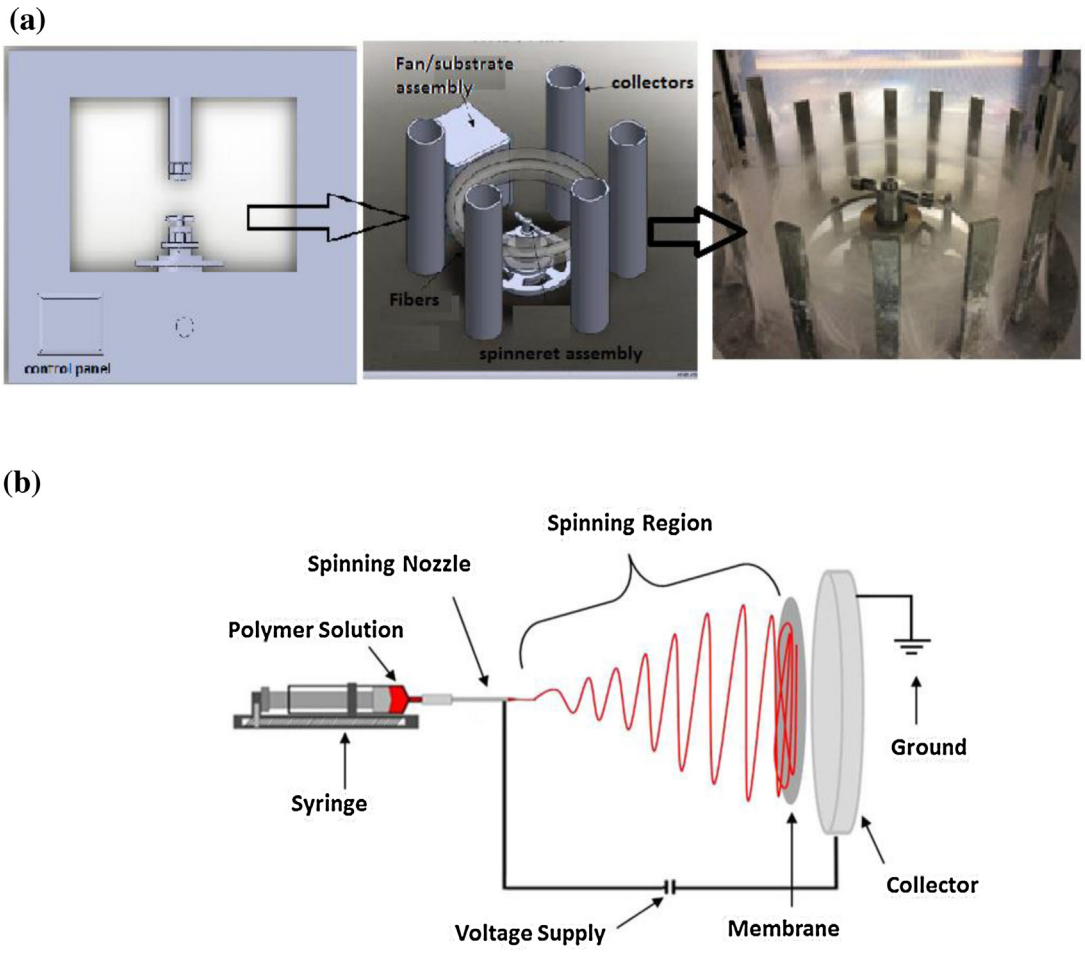

Fig. 1. a: A pictorial view of the Forcespinning machine and a schematic drawing of a typical setup inside the machine showing the collectors, the spinneret and the substrate/fan assembly for fiber production. (b) A schematic drawing of nanofiber making process using electrospinning.
Reproduced with permission from Journal of Materials Science [51].

centrifugal jet spinning, and Forcespinning® have emerged as promising methods to produce fibers with thickness of 10–20 μm and diameters that are typically less than 5 μm [54–60]. These fiber processing methods have been successful in giving the non-woven nanofiber mat their high labyrinth-like porous structure [57]. Presently, the most commonly used methods for making these nanofibers employ either an electrostatic charge (electro-spinning) [57,61–63] or external heated air jets (melt-blowing) bicomponent fiber spinning, phase separation, template synthesis, and self-assembly [64–69]. These methods are often complex and for the past decade they have been used only to make nanofibers from limited types of polymers, often lab-scale in nature.

Electrospinning is also by far the method of choice for producing nanofibers (NFs), however it faces several drawbacks such as high electric field requirements, solutions with better dielectric properties, and many other safety related issues. Many polymeric materials can be electrospun into fibers; however, electrospinning may not be suitable for mass production of certain materials and faces several restrictions regarding safety, especially for melt electrospinning where a high current is applied to the melt to form the fibers followed by stretching, structuring and cooling [48,70–76]. However, the large scale production challenge of electrospinning is set to see an end in sight, with Jiangxi Advanced Nanofiber Technology Co., Ltd mass producing polyimide nano-fiber separators for advanced lithium-ion in a scale of 3000 m^2/day [patent is available at: http://www.faqs.org/patents/app/20130164629]. Forcespinning (FS) has been used to prepare fibers from a wide range of materials due to the fact that FS can produce fibers from melt and solutions without the need of applying an electric field during processing. Furthermore, the FS method has proven to be suitable for the mass production of nanofibers for biomedical, energy storage, defense and aerospace-related-applications [39,55,77–80]. In the FS method (Fig. 1a), the electric field used in the electrospinning process is replaced by centrifugal forces to overcome the polymer/material surface tension and therefore stretching the fibers [38,39,77,81–84].

The production of NFs by a FS lab scale system is higher than 60 grams/hr which is much higher than the yield obtained by electrospinning (approximately i.e. 0.1 grams/hour) [46,85]. Several NFs systems and polymer composites have been recently produced by the FS system [38,77,82–84,86]. In these NFs systems, the fibers were produced with average fiber diameters in the nano to submicron range. In this category, there is another method known as the centrifugal spinning, which has some similarities with the Forcespinning method. The in-house built centrifugal spinning system has been used quiet extensively to produce nanofiber for biomedical, energy storage, and other electronics applications [43,80].The in-house built centrifugal system was specifically designed to produce fibers at the lab scale in a similar manner to the FS and centrifugal systems [43,46,60,85,87]. However, the in-house built centrifugal spinning system is limited for the production of micro and nano fibers at high rotational speed and it has no capability at this time for melt spinning. [88–91]. It is worthwhile to note here that the centrifugal spinning method was originally developed by Hooper back in 1924 to produce artificial silk threads from viscose or equivalent substances by applying centrifugal forces to a viscous material [87]. Hence, this method has been used in the fiber production of various materials since it was introduced by Hooper in the beginning of the 19th century.

The Forcespinning technique on the other hand, has several features such as a fiber management system that allow tunable fiber deposition to ensure accurate cross directional coating uniformity, and also adaptable to substrate web widths. In addition, the FS system has the capability for dual material feed, thus allowing the continuous materials feed system for melt and solution processing with no material dielectric requirements[46]. The system (i.e. FS) has an almost 100% yield and solvent-free processing for melt spinning with melt temperatures up to 350 °C. This eliminates the direct operating expense and environmental burdens.

3. Carbon Nanofiber (CNFs) anodes

Recently, there have been extensive research activities focusing on the development of nanostructured anode materials in the view of enhancing the capacity, energy density and specific power of rechargeable lithium-ion batteries [92–97]. In nanofiber processing such as electrospinning and Forcespinning, a polymer precursor is usually used to produce nanofibers that will be transformed into carbon nanofibers after different thermal treatments (carbonization). The produced carbon nanofiber mats can be very often brittle. This can be avoided by carrying out a stabilization process in air (i.e. oxidative stabilization) on the polymer nanofiber precursor prior to carbonization. The yield of carbon nanofibers depends on several factors including the polymer-type used during nanofiber spinning. In general, the behavior of polymeric materials is time, temperature, and pressure dependent [98–100], therefore, the nanofiber morphology and structure are significantly dependent on the polymer concentration and the chemical structure of the polymer used to prepare the nanofiber precursor.

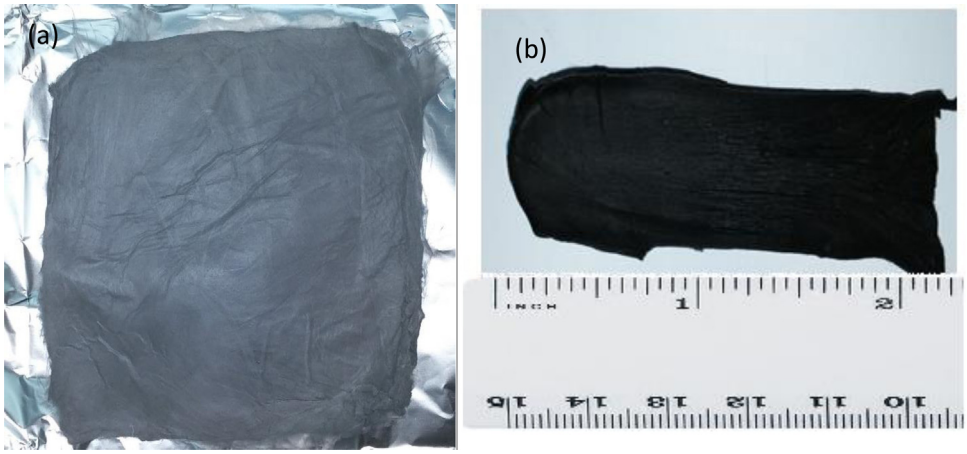

Fig. 2. A typical Pictorial view of the As-forcespun Sn/PAN precursor nanofiber (a) and the carbonized Sn/C at 800 °C for 3 hrs (b).

Carbon nano fibers (CNFs) have been widely used as anode material to replace commercial graphite particles for LIBs [23,101–104]. Graphitized stacked-cup carbon nanofiber, highly porous CNFs, and ultra-long thin align carbon fibers have been produced by electrospinning as alternate anode materials [25,28,105–108]. Carbon nanofibers can be prepared by carbonization of different types of precursor polymer nanofibers, such as polyacrylonitrile (PAN), polyimide (PI), and pitch [52,77,109]. The carbonization temperature can be adjusted to control the carbon crystalline structure and, hence, the electrochemical properties of carbon nanofibers Si/C or Sn/C composite nanofibers (Fig. 2).

The CNFs produced from these precursors have smaller diameters and larger surface area to volume ratio [23,101,103]. A typical morphology and structure of the CNFs produced through the Forcespinning technique is shown in Fig. 3. A large amount of defects in CNFs such as lattice and surface defects along the fiber length are expected to promote fast Li intercalation/de-intercalation. These physical properties of the CNFs promote fast ion transport, high accessibility of electrolyte, and shortens the Li^+/electron diffusion path that is often attributed to the large surface area of nanofibers and their large pore volume [23,102]. There is often a correlation between these physical properties of CNFs with the electrochemical performance of CNFs-based electrodes in lithium ion batteries.

Several studies report a specific reversible capacity between 400–600 $mAhg^{-1}$ and excellent cycling performance after 50 cycles for CNFs anodes [31,101–103]. Comparatively, the electrochemical performance of the CNFs anodes is much better than that for commercial graphite anode with a theoretical capacity of 372$mAhg^{-1}$. However, the loose structure of these nanofibers result in a low tap density, thus most of CNFs materials do not show much advantage compared to the graphite anode (~10 micron in particle size) when based on the volumetric energy density of the electrode. In fact, the graphite is so far the best anode material to use in commercial LIBs due to its long cycle life, low cost, and good capacity retention, even after hundreds of charge/discharge cycles, though its charge/discharge capacity is lower than ceramic composite nanofibers. There is no other anode material that can compete with graphite at this time, although extensive research has been carried out to improve the performance and cycle stability of Si/C or Sn/C composite anodes. Theoretically, Si or Sn based anodes give a high capacity and might replace graphite anode in commercial LIBs in 50 years or so. This needs more work and effort to be done to improve the cycle stability and capacity retention of Si/C composite NF anodes. The good electrochemical performance of CNFs anodes have often been the driving force for the huge interest among researchers. However, often the carbon nanofiber mats produced using some of these common techniques are not flexible after the carbonization process. It is therefore a common practice to grind the brittle CNFs with conductive carbon black to increase conductivity and adding binding agents to produce a slurry for the electrode making process [5,71,110–112]. The grinding of the rather uniform nanofiber compromises the fibrous nature of the nanofiber and its intended purpose. Another challenge of CNFs is the low fiber yield (i.e. 0.1 grams/hr for electrospinning) produced by most of the techniques, thus limiting the scaling up production of these CNFs [71,112–119]. There is however an end in sight for the low fiber yield challenge with the recent introduction of Forcespinning into the nanofiber production techniques. The Forcespinning has the capability of producing fiber yield greater than 60 grams/hr, is less expensive process, and is environmentally safer [38,46,77] compared to electrospinning.

The successes of CNFs and their associated electrochemical performance have boosted the research in the area of lithium alloy-based electrodes for lithium ion batteries. Several research activities have been reported on composite electrodes comprising various metals and metal oxides and CNFs as electrode and separators for LIBs [107,109,120–126]. It is still early for these composite nanofibers to be used as electrodes in commercial lithium ion batteries. The loose structure of composite nanofibers

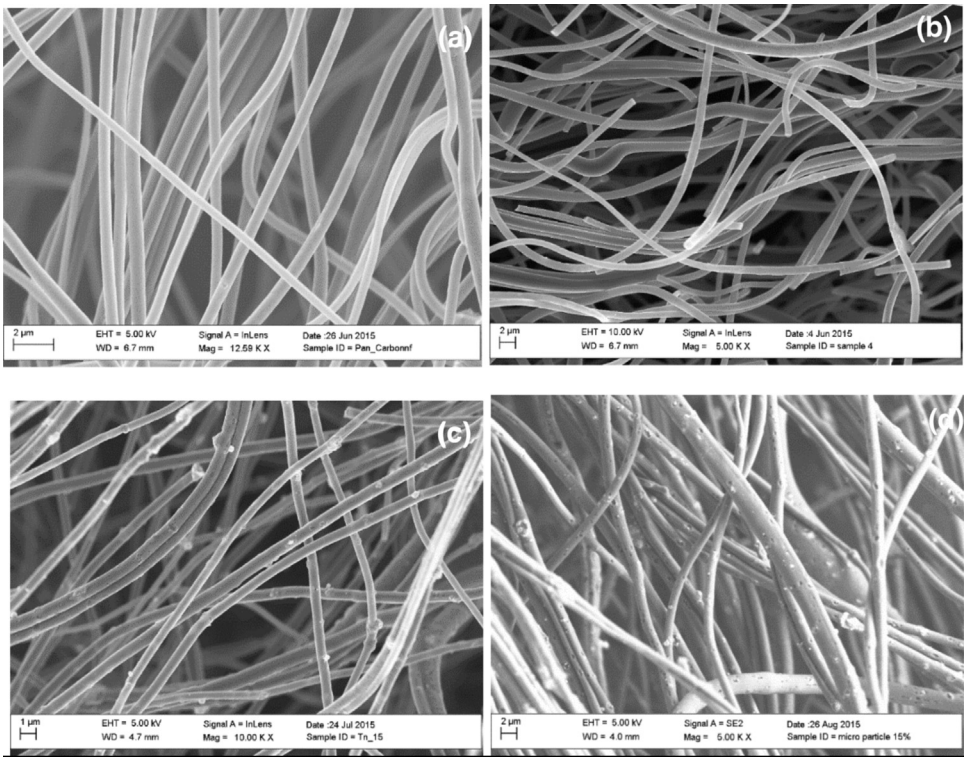

Fig. 3. SEM micrographs showing the morphology of the baseline as forcespun PAN fibers (a) and the carbinized PAN CNFs (b), the forcespun Sn/PAN fibers(c) and the Carbonized Sn/C nanofiber (d) nanofibers showing pores on the fibers strands.

itself, is an undesired property for lithium ion batteries due the low tap density. The ensuing discussion will focus on the use of CNFs as separators and electrodes for lithium ion batteries. Generally, the capacity and energy density of lithium ion batteries depend largely on the cathode material capacity. However, noticeable improvement of the overall capacity and energy density of the cell is often observed when using an alternate anode material having a capacity of the order of 1000 $mAhg^{-1}$ which is much higher than that of carbonaceous electrode (372 $mAhg^{-1}$) material.

The driving force for such a replacement of a carbonaceous material lie in the fact that the potential of the anode vs Li/Li^+ should be close to 0 V and not necessarily an intercalation type of electrochemical reaction as pertaining in the graphite anode. Alloying Li^+ with several viable metals and metal oxides such as Si, SiO_2, Ge, Fe, Fe_2O_3, Ag, Pb, Sn, SnO_2 etc. are potential candidates for use as anodes in LIBs [96,113,127,128]. The Li_xM (where M = Sn, Si, FeO_2O_3, etc.) alloys show a much higher Li:M ratio at the end of the charge cycle therefore allowing a greater Li^+ accommodation. Therefore these lithium alloys have higher capacity and energy density than commercial graphite anode. However, the major challenge for these Li:M alloys is that they are known to degrade rapidly from their high theoretical specific capacity of 998–3600 $mAhg^{-1}$ due to several factors including high volume change during the alloying/de-alloying process which can lead to pulverization and poor cyclability [129–131]. Nanofiber metal/metal oxide composite anodes have been an emerging trend where research have employed metal/carbon composite nanofibers in a bid to either curb or minimize the high volumetric change of these Li: M alloys [5,132,133]. The fibrous structure of the nanofiber electrode provides ample room to accommodate the volume changes during the alloying/de-alloying process. We discuss below recent progress and development reported on the use of metal/CNFs as anodes for LIBs.

4. Lithium alloy-CNFs anode materials

Lithium alloys and carbonaceous materials are two classes of anode materials for lithium-ion batteries. Both materials have shortcomings (e.g., low charge capacity for carbon and low cycle life for lithium alloy) that limit the performance of lithium-ion batteries. Metal and metal oxide/carbon nanostructured composite such as Sn/CNFs, Si/CNFs, SiO_2/CNFs, SnO_2/CNFs, TiO_2/CNFs and Ferriferous oxide/CNFs have been widely used as anodes for LIBs [1,25,134–137]. Some of these anodes are discussed in the following section.

4.1. Tin/Carbon composite nanofibers

Tin (Sn)-based derivatives such as tin oxides, tin sulfides, and stannates have become attractive anode materials for LIBs [5,138–140]. The popularity of these Sn-based lithium alloys stem from their properties such as; the inhibition of solvent co-intercalation and significant improvement in performance over the commercial graphite anode in LIBs [112,115,141–143]. In addition, Sn/carbon composite nanofibers have been studied for use as anodes in LIBs because they are easy to process and they exhibit lower potential hysteresis compared to other transition metal oxides [144]. Sn/CNFs composite are capable of hosting a higher amount of lithium ions in tin crystal structure, i.e. about four (4) atoms (i.e. Li_xSn, $0 < X \leq 4.4$) compared to carbonaceous anode (i.e. $LixC_6$, $0 < X \leq 1$) thereby giving Sn/C composite a higher lithium storage capacity.

Sn/carbon composite nanofibers show excellent reversible capacity and cyclic performance, even at a higher current rate of 200 mAg^{-1} to 1 Ag^{-1} [5,138,139]. This performance has been found to depend largely on the carbonization temperature, which tend to have a direct impact on the fiber morphology, structure and the pore distribution [5,139], as shown in Fig. 3. There are varying reports on the influence of Sn particles size on the overall composite Sn/CNFs electrode performance. While some of these anodes show a higher reversible capacity with a Sn particle size in

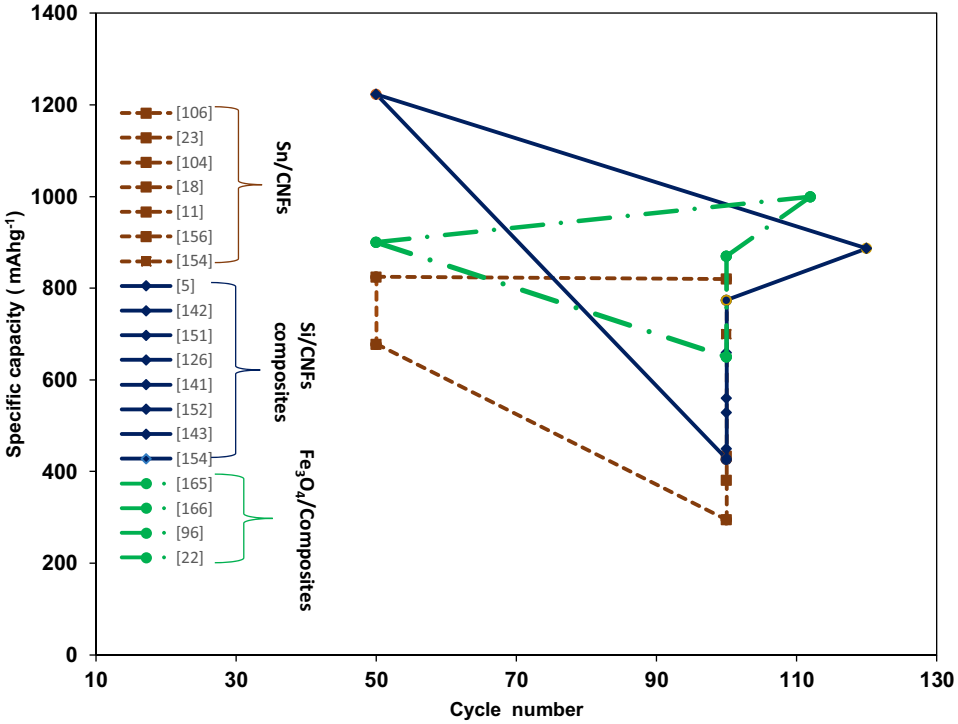

Fig. 4. A plot showing typical cycling performance of the base CNFs electrode, Sn/CNFs composite electrode and Si/CNFs composite electrode cells. Si/CNFs anodes show capacity fading because of the volume change.

the nano range (> 5 nm) [102,137], other studies reported no significant difference in the particle size influence on electrochemical performance [145–147]. A smaller particle size largely will generally offer a higher surface area and aspect ratio, which offer the electrode more reactive sites for electrochemical reaction; however nano-size particles are equally prone to agglomeration which could affect their uniform dispersion in the polymer matrix.

4.1.1. Sn/C composite nanofibers

Ternary composites of Sn/CNFs incorporating or containing other metals and their oxides have been designed to further improve the conductivity, the cyclic and rate performance of LIBs. SnO_2/ZnO heterogeneous nanofiber [104,148], Nitrogen doped CNFs with Sn nanoparticles [123], Co-Sn alloy carbon nanofibers [149], and Fe_3O_4/SnO_2 coaxial nanofibers [150] have been investigated. The N-doped Sn/CNF's for instance, provide fast and versatile electrolyte transport and act as efficient electron transport pathways and stable mechanical supports for keeping the structural integrity of the electrodes during cycling and preventing pulverization of the composite electrode [123]. Furthermore, the Sn/CNFs containing cobalt minimize the volume expansion and enhance the electrical conductivity of the electrode [149,151]. The Sn and its composite derivatives (Fig. 4) show a very remarkable cyclic performance when they are used as Sn/CNFs composite electrode. These composites still show a remarkable capacity retention at high current densities (Fig. 5). The CNFs/Sn based composite electrode can inevitably reduce the volume change (i.e. > 260%) that results from alloying/de-alloying processes that sometimes lead to particle isolation and disconnectivity between the electrical conductive particles during cycling (i.e. charge/discharge process). Although Sn/CNFs-based composites are considered good alternative anodes for LIBs, their inconsistent cycle performance and their structural instability is hindering their practical applications in rechargeable batteries. Additionally, Tin by itself is expensive compared to carbon, which is in abundance and can be derived from either petroleum based products or as a derivative of many polymers.

4.2. Si/Carbon composite nanofibers

Silicon is considered as a good candidate to use as anode material in lithium-ion batteries due to its high specific capacity and low discharge potentials. A fully lithiated Si- Li (i.e. $Li_{21}Si_5$) alloy has a large theoretical ~4100 $mAhg^{-1}$ which is 10 times greater than that of graphite. However, the many associated problems of the LiSi-based cells, such as large irreversible capacity at room temperature, [2,6,7,152–154] and poor cyclability, [3,155] are often attributed to the low electronic conductivity and the large volume change (> 300%) during lithium insertion/de-insertion process. This volume change impacts negatively on the Si crystal structure, inducing stresses in Si lattice and thus leading to cracks formation and pulverization of the Si particles. Various approaches have been developed to minimize the drawbacks of LiSi anodes, these include better morphological design [156], synthesis of single-phase LiSi by highly energetic ball-milling [29] using impregnated Silicon nanoparticle assemblies in templated carbon-bridged oriented graphene[157], coating copper on a nano columnar silicon anodes [158], and creating nanosheets of silicon-based electrodes by topochemical reaction synthesis [159].These approaches and techniques help mitigate the drastic reduction of the LiSi anode capacity, but are not sufficient to maintain a good capacity retention and cycle life [160,161].

4.2.1. Si/C composite nanofiber anode

Si/CNFs binary composite electrodes [6,162,163] from nanostructured metal/oxide namely: Ge, SnO_2, MnOx, iron oxides, and TiO_2 [22,164,165] have been developed to improve the electrochemical performances of LiSi electrode. In the Si/metal binary alloy composites [26,27,165–170], the metal/metal oxides act as the

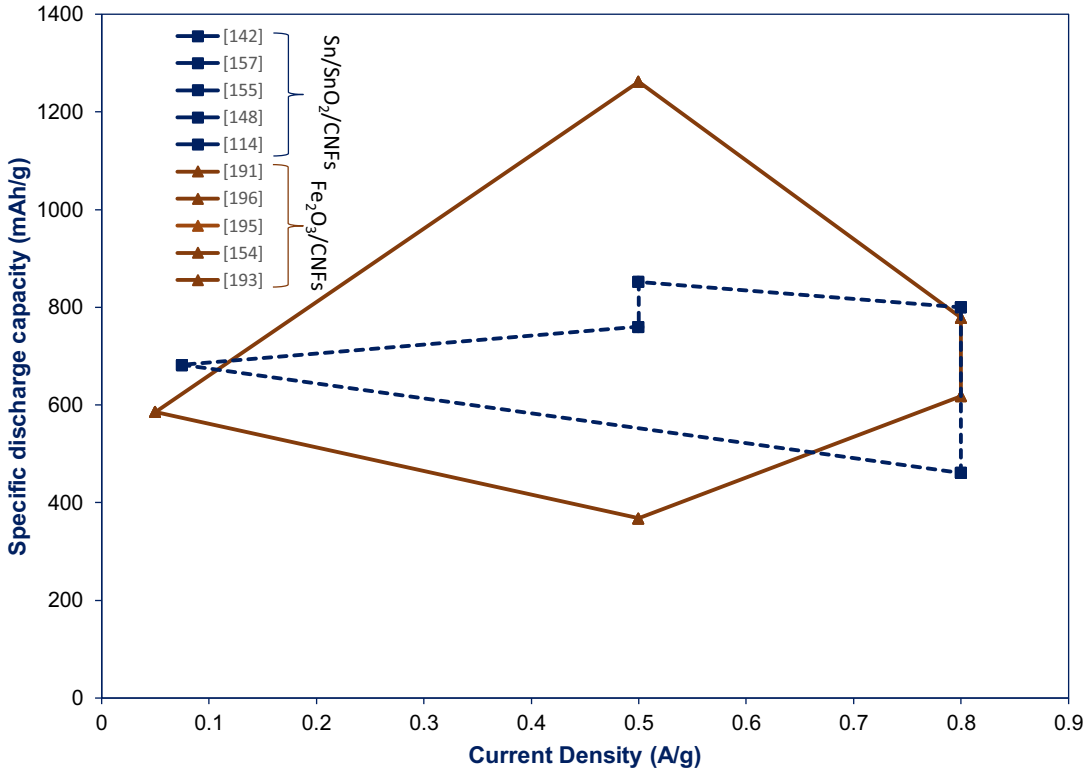

Fig. 5. Rate performance of Sn/C, SnO2/C, and Fe_2O_3/C composite electrode cycled between 0.01- 3.0 V vs Li/Li^+.

unlithiated part of the composite alloy thereby forming a framework that prevents the pulverization of the Si particles [6], improving stability and electrochemical performance of the LiSi electrode. Some of these binary Si/metal/CNFs have been widely reported to delivere a discharge capacity of 1000 mA h g^{-1} over 200 cycles [3,167,171–181] (Fig. 4). The carbon nanofiber in the matrix acts as a buffer for the volume change and improves the electronic conductivity of the electrode. [6,7,182–186]. The Si/CNFs nanostructures can suppress volume changes since the pores act as the structure buffer for the large volume changes during cycling, and hence help maintain the high specific capacity and cycle performance of the electrode above 1500mAhg^{-1} over several cycles [3,7,22,163,167]. Although nanostructured Si/CNFs anodes and their composites have improved electrochemical performance, several problems need to be solved including; dispersion of Si nanoparticles in the carbon matrix, high production cost usually associated with the price of silicon as the based material. Further work is needed to address these issues.

4.3. Ferriferous oxide/Carbon nanofibers anode

Ferriferous oxides (Fe_3O_4)/CNFs are the other class of promising alternative materials that have been widely investigated as anodes [9,25,33,150,187–192]. Honna Ferriferous oxides typically have higher theoretical specific capacity of about 924 mAh/g, relatively inexpensive and environment friendly (non-toxic) [193–195]. However, similar to Si, Ti, and Sn, the bare Fe_2O_3 and Fe_3O_4 have low conductivity and prone to structural pulverization and loss of electrical connection due to large volume changes during lithiation/delithiation, resulting in the poor capacity retention during long-term cycling [9,21,33,188,193]. To overcome these drawbacks of Fe_3O_4 anodes, carbon nanofibers have been employed as matrix to prepare Fe_3O_4/CNFs composites. These Fe_3O_4/CNFs composites have improved electrochemical properties (Fig. 4 and Fig. 5.) due to the excellent electrical conductivity of the carbon nanofibers [33,189,192,196]. The structure of the Fe_3O_4/CNFs, which are either honey-comb like carbon core and Fe shell or hollow [150,197,198], 3D nano-Fe_3O_4 [191],or hollow α-$LiFeO_2$ [196], as shown in Fig. 6.

These ferriferous structures give the composite electrode improved low temperature performance largely on the account of the fast electron kinetics [33,189], and the stability of the porous CNF and its buffering effect [191,196,198]. Other metals like Mg, Ni, and Zn [9,21,190,197] have been used as binary alloy with Ferriferous oxides along with CNFs ostensible to improve the electrochemical performance of the composite electrode. Typically, the specific capacity for these binary composites are in the range of 500 mAhg^{-1} to 700 mAhg^{-1}, which is not essentially better than those reported for Fe_3O_4/CNFs composite anodes (Fig. 7), that have capacities 500 mAhg^{-1}-1225mAhg^{-1} even [25,150,188,193,194] at higher current densities (i.e. 1A/g)[188,191,192,196,198], as shown in Fig. 5. These metals in the binary alloy of the ferriferous also act as a buffer layer that accommodate the large volume changes and reduced polarization [21,190,197] and ostensibly enhancing the kinetics and mass transfer at the electrode interface. The major challenge with the use of these ferriferous oxide electrodes is how to simultaneously design and fabricate composites of Fe_2O_3 combined with CNFs. An improved fabrication technique is highly desirable since this class of alternative anode electrode is highly desired, especially for low temperature performance.

5. Lithium metal oxide-CNFs cathode materials

Electrospinning has been extensively used to produce nanofiber composite cathodes from different precursors such as spinel structured materials; $LiMn_2O_4$, $LiMPO_4$ [199–201], $LiFePO_4$ and Vanadium oxide layered compounds [202–204]. Although $LiMn_2O_4$ is inexpensive and safer than $LiCoO_2$, it has a lower capacity as compared to other cathode materials that form α-$NaFeO_2$ structure [205–208]. This high capacity is mostly

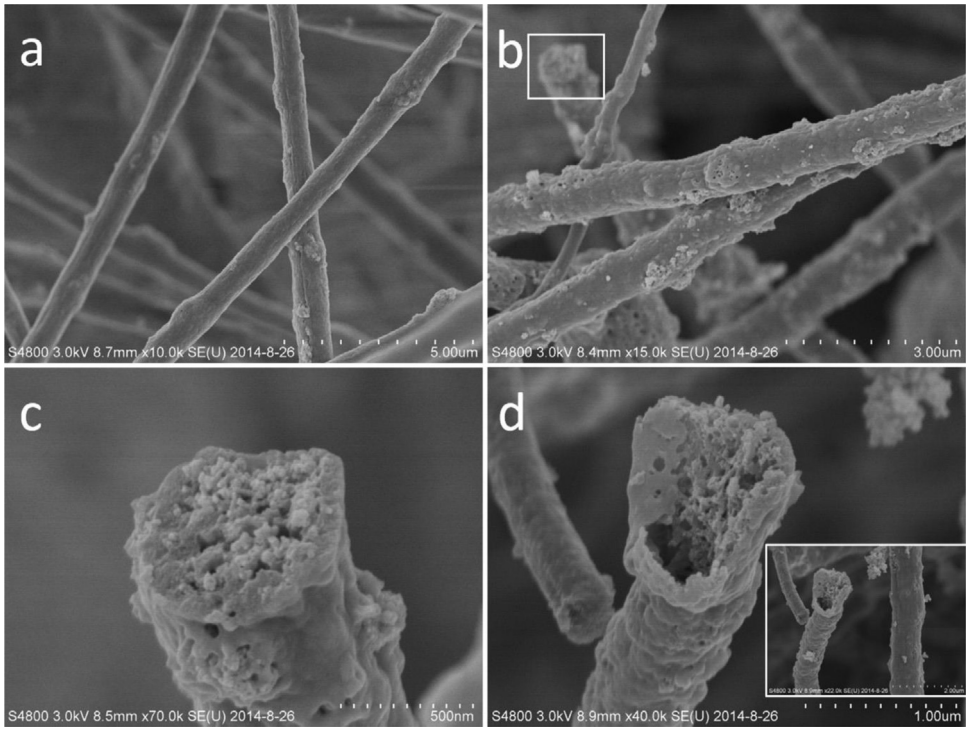

Fig. 6. SEM micrograph of a typical (a) Fe_3O_4/PAN/PS precursor fibers and cross-section of the nanofiber showing the pore structure and hollowness of the finished of Fe_3O_4/CNFs [182].
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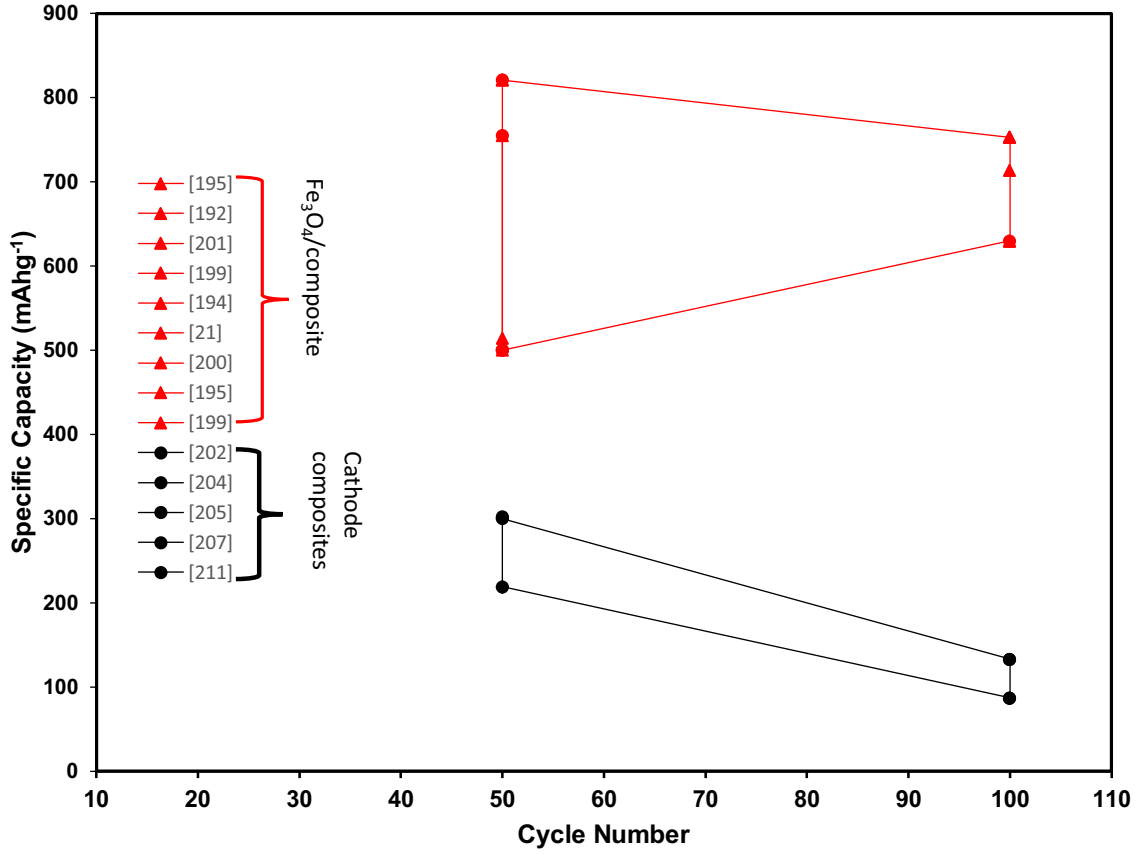

Fig. 7. A plot showing typical cycling performance of the ferriferous oxide/carbon nanofibers composite electrode and cathode materials cells.

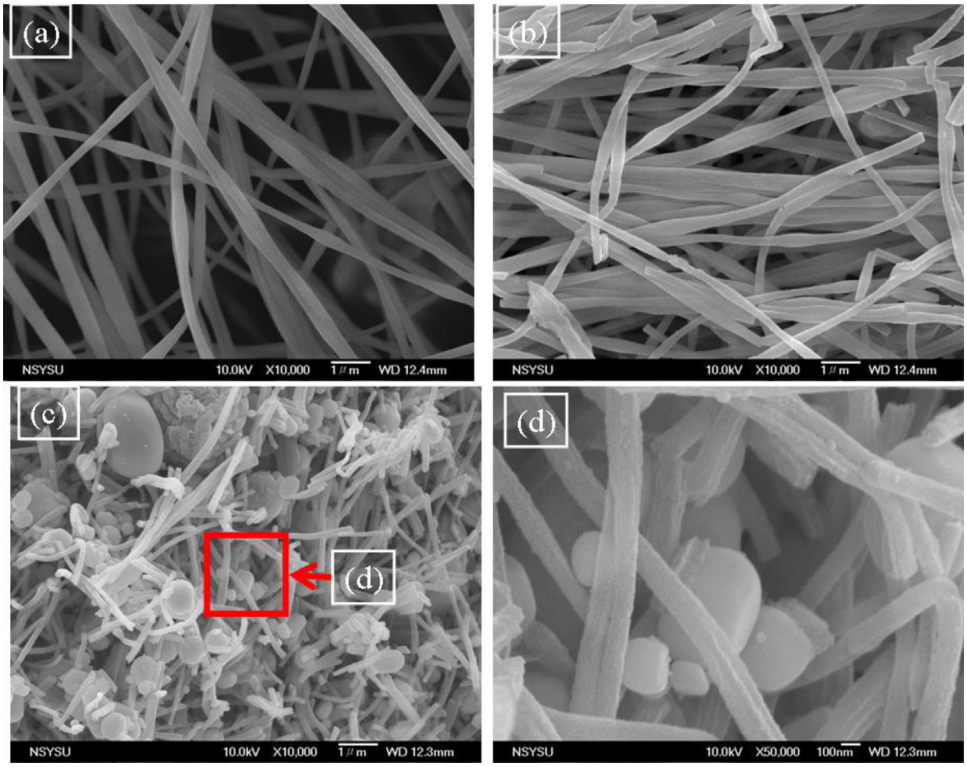

Fig. 8. SEM images (a) as-spun $LiFePO_4$ precursor/PAN nanofibers (b) ASL-PAN after 300 °C heat treatment, (c) $LiFePO_4$/C composite nanofibers after calcination at 800 °C, and (d) high magnification of the region marked with a square in (c)[214].
Reproduced with permission from the Int. J. Electrochem. Sci.

attributed to phase changes that occur during cycling [209–213]. Vanadium based cathodes on the other hand, have high capacities but relatively low voltages (i.e. ≤ 3 V) when compared to cathode compounds such as$LiCoO_2$ and $LiMn_2O_4$ [202,204,214]. The fibrous composite electrodes of V_2O_5/CNFs and $LiMnO_4$/CNFs usually form a one dimensional (1D) nanostructure (Fig. 8). The characteristics of these 1D structured materials are their large surface areas that provide an effective diffusion pathway for both Li ions and electrons. The fast electron transfer rate and the large number of electroactive sites provided by these 1D nanofiber structured cathodes ostensibly improve the electrochemical performance [199,200,202,214]. Typically, these nanofiberous cathode materials show an impressive specific capacity in the range of 240–300 $mAhg^{-1}$ which are much better than those usually used in commercial LIB (Fig. 7). The synthesis of cathode materials into carbon nanofiber structures is relatively new yet promising. The challenge with the synthesis of these cathode/CNFs materials is balancing concentration of polymer-gel solution and cathode material. The right amount of the polymer plays a critical role in maintaining the fiber-structure[215], while the cathode materials determine the electrochemical properties of the composite electrode. Therefore, more work needs to be done to addresses these pertinent issues.

5.1. Composite nanofibers for Li/S battery cathode

Lithium-sulfur batteries have attracted much attention and are considered as good candidates for alternative energy storage systems, largely on account of their ultra-high capacity, high theoretical specific power and energy density, easiness in handling and processing, and environmentally benign [216–223]. A Li-S battery consists of a Li metal anode, an organic electrolyte and a sulfur composite cathode (porous), wherein S (existing as S_8) undergoes a series of electrochemical reactions to give Li sulfide (Li_2S) with each S atom accepting two lithium atoms [216,217]. The application of composite nanofibers has been investigated as an alternative means to improve the microstructure of the cathode electrode in Li/S batteries. The advantage of using these carbon nanofibers with sulfur as cathode for Li/S stem from their unique structures; porous/hollow structures, and high specific surface area. These properties of the carbon nanofiber-sulfur based cathodes form ideal inter wound reservoir-like matrices that accommodate active sulfur [123,153,224–240], while the porous structure ideally confine/trap the active sulfur and its soluble polysulfides, which can greatly reduce the shuttle effect. In addition, the carbon nanofibers improve the mechanical/structural integrity of the composite electrode as well as the conductivity, [232,233,238] that further enhance electron/Li^+ transfer and improve sulfur utilization in Li-S batteries [123,153,224–240] and by extension, improve the batteries electrochemical performance. The challenge however, on the use of these carbon nanofiber-sulfur-based cathodes in Li-S batteries is the inconsistency of the fiber surface area, particularly those produced by electrospinning [225,226,241,242]. A relatively small surface area and a large pore volume size can lead to sulfur being exposed outside the porous structure, which can cause serious polysulfide dissolutions at prolonged battery cycling. Additionally, notwithstanding the fact that the microstructure of these carbon nanofibers provide porous/robust conductive networks, compared to graphite (~10 micron in particle size), their surface area and pore volumes are much lower, which can limit the amount of sulfur loading in the electrode. To countenance these challenges, several morphologies and electrode architectures have been designed including; hollow carbon nanofibers-encapsulated sulfur[243], amphiphilic surface modification of hollow carbon nanofibers [244], highly mineralized chitin-protein fibers as biotemplates [245], bimodal mesoporous carbon nanofibers[246], impregnation of microporous activated carbon fibers with elemental melted

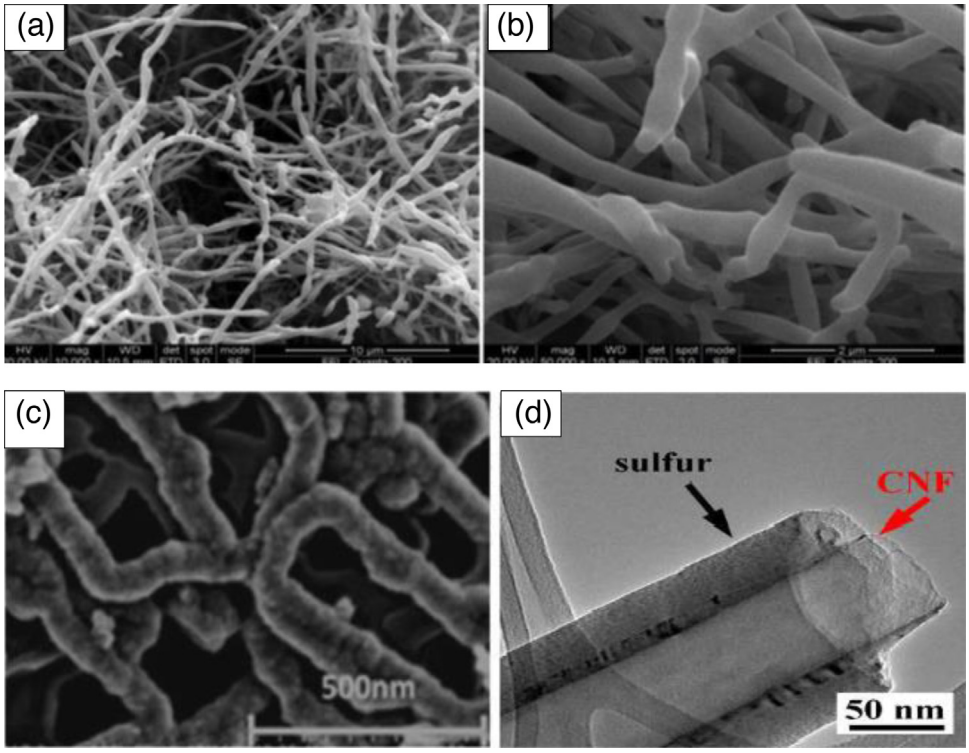

Fig. 9. SEM images of hollow carbon nanofiber-Sulfur composite electrode (a,b) [222], Nitrogen doped carbon nanofiber web/sulfur composite electrode (c)[247] and CNFs with sulfur coating(d) [230].
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sulfur (ACF-S), infiltration sulfur into a microporous CNFs/CNTs composite[237], functionalization of nitrogen-doped porous carbon nanofiber/sulfur composites [247,248] and hollow carbon nanofiber hybrid nanostructure anchored with titanium dioxide ($HCNF@TiO_2$) [239] (Fig. 9). All these electrode architectures and designs are either aimed at improving the crystal structure of the sulfur hosting composite electrode [249,250], minimizing sulfur dissolution, increasing specific capacity or energy density (Fig. 10), reducing cost, or improving safety of Li-S batteries [12,244–246,251–254]. On the separator front for Li-S batteries, there is surprisingly not much attention or interest compared to the active dynamics of cathode research. The few that exist focus on the modification of existing polypropylene and polyethylene commercial separators. Nonwoven PVdF- nanofibers, nanofibers from ceramic precursors have been coated on the mono layer of PP and PE Celgard separators with aim to inhibit the diffusion of polysulfide and its intermediates onto the anode. This will result in reducing the shuttle effects on Li-S battery performance [255–258]. The subtle absence of wholly nonwoven nanofiber separators from polymeric materials is due partly to their low strength and loose and open structure that might compound the plating of sulfur products on the Li metal anode from sulfur dissolution (shuttle effect).

5.2. Composite nanofibers for Li-air battery cathodes

There are very few publications that explore the use of composite nanofibers as cathode material for Li-air batteries. The most common Li-air cathode material designed with carbon is Co_3O_4 [227,259,260] and few reports on RuO_2/CNFs, αMnO_2/CNFs [15,261–265]. The motivation for the use of carbon nanofibers has been the higher surface area as well as the higher pores volume that carbon nanofibers present compared to graphite or other carbon based materials [266] [15]. The porosity and 3-phase structure of air-cathode is one key and critical determining factor for the Li-air battery electrochemical performance (Fig. 11). The higher pore volume and surface area of a typical composite NFs Li-Air cathode, increase the contact area of the electrode/electrolyte interface while the pores provide a high diffusion path for both ions and oxygen necessary for the electrochemical reaction to form species such as Li_2O_2 and Li_2O [259,262,264,265]. These composite nanofiber cathodes record an impressive electrochemical performance over several cycles, far better than the commercial RuO_2, Co_3O_4, and αMnO_2 Li-Air cathode [15,260–264,267], as shown in a typical electrochemical performance plot of composite CNFs electrode for Li-Air batteries in Fig. 12. The downside of the open structure of the carbon nanofibers tends to decrease the conductivity due to occasional discontinuity in the electrical path. This challenge, notwithstanding, the pore structure provides room

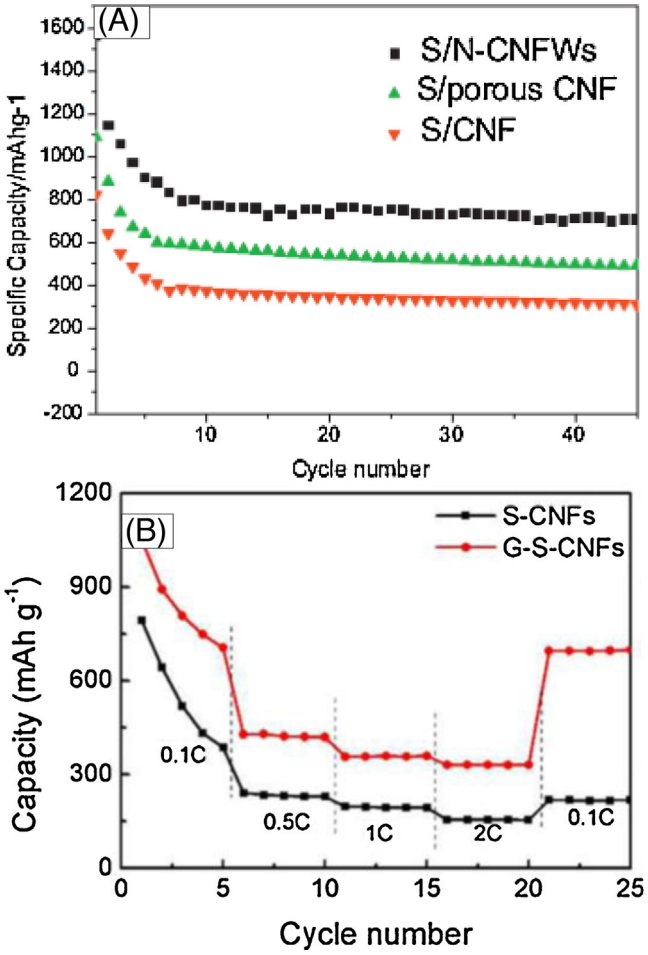

Fig. 10. A typical plots showing the effect of CNFs/S microstructure [247] on cell performance (a) and the rate capability of these of the S-CNF nanocomposite with (G-S-CNFs) composite electrodes [230] (b).
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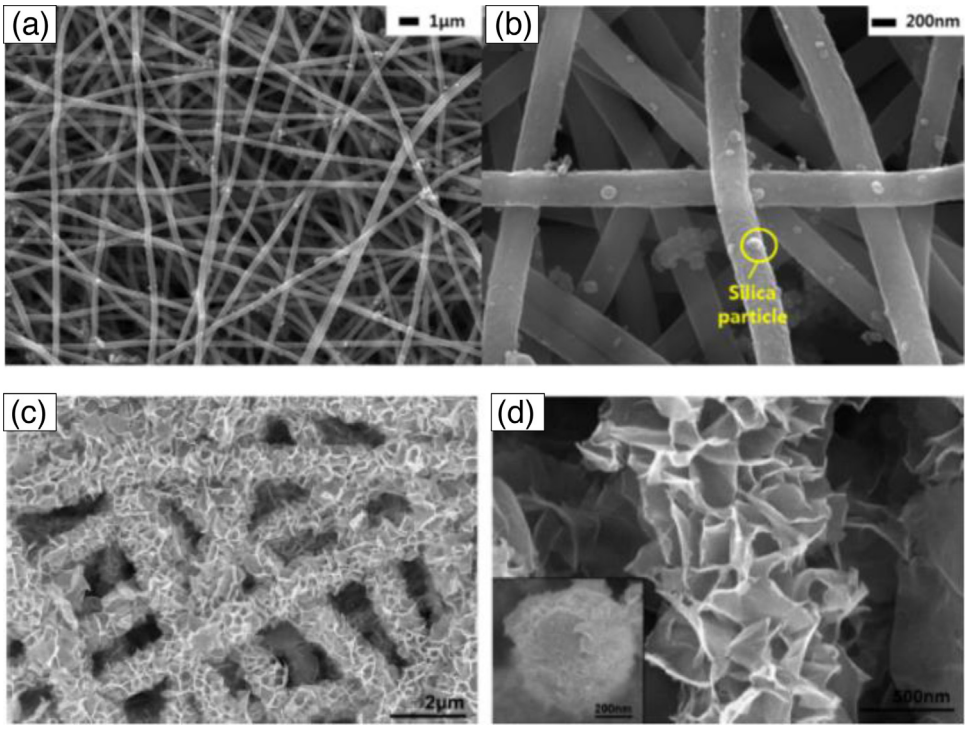

Fig. 11. Microstructure of mesoporous silica coated carbon nanofibers (a,b) [15] and mesoporous flower-like cobalt oxide/carbon nanofiber composites with shell–core structure (c,d) [264] composite electrodes for Li-Air batteries.
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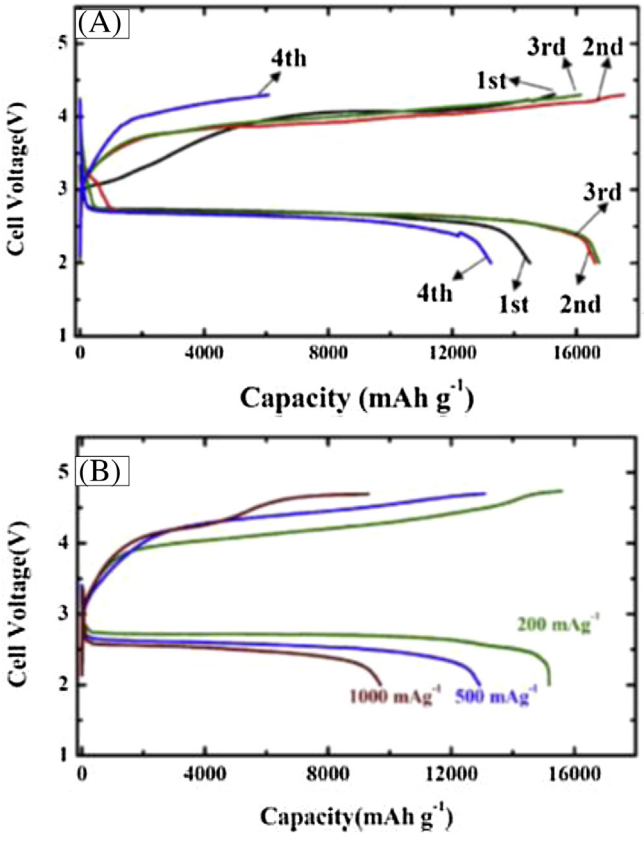

Fig. 12. A typical electrochemical discharge/charge curves at a current density of 100 mA g-1 (a) and rate performance at varying current densities (b) from 200 mA g-1 to 1000 mA g-1 of CNFs/RuO2 composite Li-Air electrode[4].
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for the precipitation of insoluble Li_2O_2 species and insulates it from further access of reactants products such as O_2 and Li^+ [259,260]. The high capacity of the Li-Air batteries chemistry has been established to directly depend on the presence and amount of Li_2O_2 in the cathode structure [227]. Nanofibers as cathode materials seem to be in its infancy and is expected to see a growing interest in the not distance future. Nanofibers and composite nanofibers mainly prepared by electrospinning have been also used in sodium ion batteries as cathodes and separators. The discussion of these NFs is beyond the scope of this review and we refer the reader to reference [13] for more details on the structure and performance of these composite NFs.

6. Polymeric Nanofiber Separators

The commonly used separators in lithium-ion batteries are the microporous membrane separators made from, polypropylene, polyethylene, polyvinylidene difluoride (PVdF) or PVdF-coated microporous polyolefin separators [268–270]. Several companies such as Celgard, Asahi, Toray, Entek Membrane, Ube industries, DSM, etc. have designed and developed commercial separators for the over $4billion lithium ion batteries market [271], these mostly consist of polymer materials such as polyolefin microporous membranes developed by Celgard, DSM, and Asahi and a tri-layer of polypropylene/polyethylene/polypropylene from Celgard and Ube industries. High performance separators must 1) be more porous, 2) be safer to puncture and shorts, 3) have higher melt stability, 4) have a thinner structure to allow for more active material, 5) have a very thin layer to allow for composite structures of ceramic coatings, 6) very thin and porous nonwoven to be filled with polymer electrolyte, 7) High thermal stability, and 8) lower cost for use in hybrid vehicles [268,270,271]. However, several studies [79,270,272–276] have pointed to these polymer-based battery separators to have less impressive key performance indices such as: high interface resistance, low thermal stability, and low electrolyte uptake. The severe thermal shrinkage of polyolefin separators can cause serious internal electrical short circuit leading to a fire disaster or battery explosion when the cells are exposed to abnormal operating conditions. Some battery separator producers such as Celgard Inc., have made several improvements to its polypropylene (PP) mono layer polymer separator by designing a three layer separator comprising of the polypropylene/polyethylene/polypropylene (i.e. PP/PE/PP) thereby improving the deficiency of its mono layer separators Celgard [35,36,38,49,52,68,77]. Nonetheless, the performance of the non-woven mat separators is gradually being considered better alternatives.

6.1. Nonwoven fibrous polymer separators

Lithium ion battery separators most often are classified into three categories; microporous membranes, non-woven mats and inorganic composite membrane separators. In all, they are usually characterized by their thinness, excellent thermal stability and the high porosity that gives them their ion transport characteristics [37,54–57,61–63,277–284]. The increasing vulnerability of the lithium battery to thermal runaway requires a separator with greater mechanical integrity above 130 °C [285,286] to provide a greater margin of safety to the battery. The nonwoven nanofiber separator membrane fibers have shown to have much better stability in the redox environment compared to other polymeric based separators[287] (Fig. 13). The nonwoven fibrous mats such as cellulosic fibers, PVdF, ceramic fibers, nanocomposite polymer have been extensively studied. The PVdF itself has been extensively used as a polymer electrolyte [288–293] by virtue of its appealing electrochemical properties. Generally, the PVdF based polymer electrolytes are anodically stable in the redox environment in the lithium ion battery due partly to the strong electron-withdrawing of the C-F functional groups on the polymer backbone [294,295]. The PVdF category of nonwoven nanofiber separators show improved electrochemical performance relative to the commonly used polypropylene/polyethylene (i.e. PP/PE) polymeric separators (Fig. 14). In the same vein, the cellulosic fibers have never been used successfully in commercial (mostly in laboratory cells) lithium batteries due to their hygroscopic nature of cellulosic papers and films, their tendency to degrade in contact with lithium metal, and their susceptibility to pinhole formation at thicknesses of about 100 μm [54,65,277–279]. This notwithstanding, the cellulosic separators still maintain some level of research interest

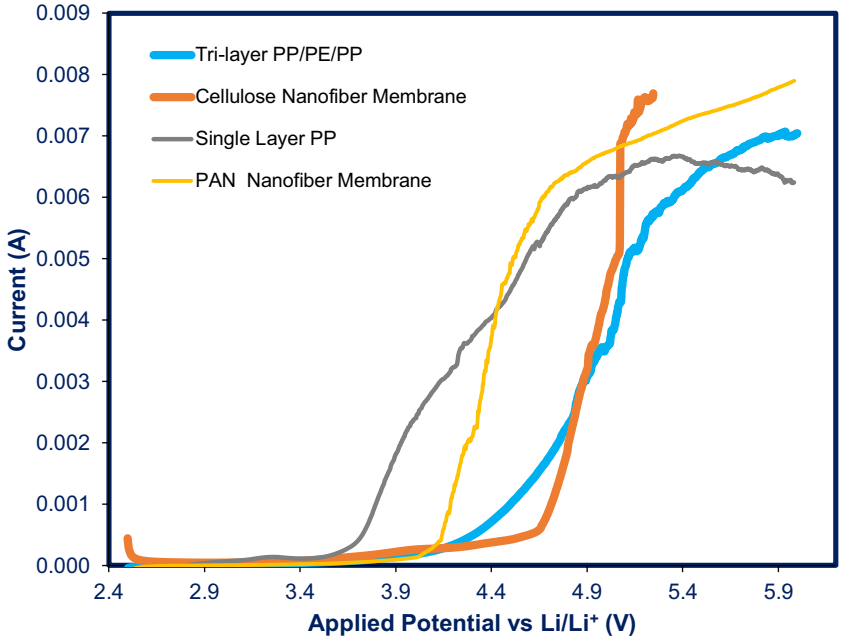

Fig. 13. A plots showing a comparison of the stability of the separator in the redox environment from the cellulose, PAN nanofiber membrane and some polymeric materials like PP and tri-layer of PP/PE.

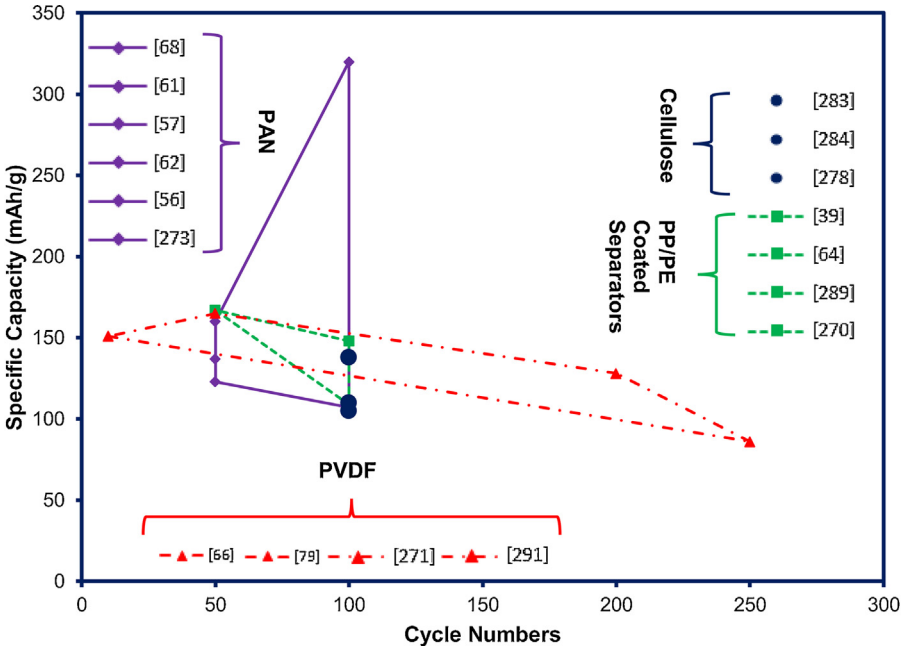

Fig. 14. A plot showing typical cycling performance of the base Cellulose, PAN, PVdF and modified PP/PE coated separators for use in lithium ion batteries.

due largely to the various morphologies of the cellulose membrane (Fig. 15) that offer stability at higher temperatures compared to separators made of polyolefin or can be coupled with the other polymeric separator materials with low melting properties as a polymeric laminate material [277,278].

The non-woven fibrous mats, despite their remarkable properties, have less mechanical strength compared to their PP counterparts[296] and are also yet to be used in commercial lithium ion batteries as separators. The drawback in their application is often attributed to their open structure and their rough surface which cannot effectively prevent micro short circuits and dendrite penetration [278], but mainly used in other battery chemistries such as alkaline batteries such as nickel–cadmium and nickel–metal–hydride batteries. In lithium ion batteries, these non-woven fiber mats are widely used as supporting framework architecture to make gel power electrolyte due largely to their high porosity and large pore size [35,52] as shown in Figs. 15 and 16.

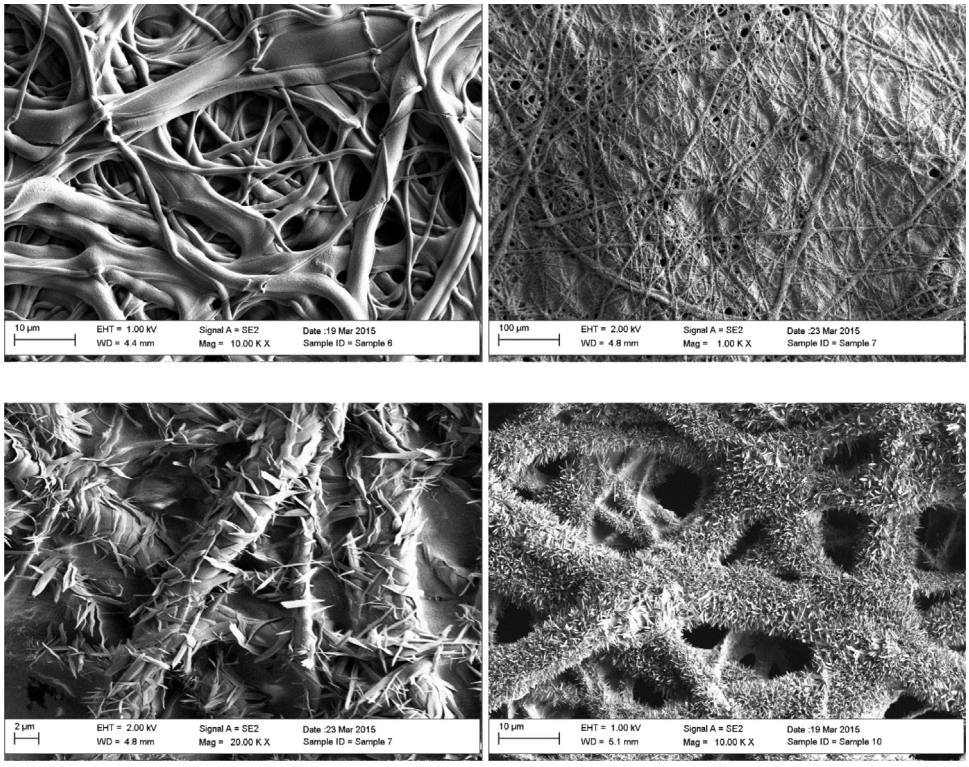

Fig. 15. Various morphologies of nanofiber cellulose membrane separator mats for use in the lithium ion batteries.

6.2. Improvement Trends

6.2.1. Coating separators with ceramic particles

As many researchers identified the short coming of these microporous polymer separators, has paved the way for many research efforts geared towards improving especially the mono layer PP separator for lithium ion batteries, while very viable alternative separators materials are sought. Ceramic coatings such as TiO_2 are deposited on the Tri-layer polymeric separator [270], Al_2O_3 and hydrophilic poly(lithium-4-styrenesulfonate) onto the porous polyethylene membrane [276], and SiO_2 coating on polyethylene [297,298]. These ceramic coating on the polymeric separator not only afford the separator its dimensional stability, but they also improve the porous structure of the separator that allow higher ion transport to improve ionic conductivity and low inner resistance [54,299].

6.2.2. Coating separators with polymer nanofiber (NFs)

The mono layer polypropylene separator has also been improved using various polymer coatings such as polymers and polymer NFs coatings. Among these polymers include the use of poly(vinylidenefluoride-co-hexafluoropropylene), and poly(ethyl-eneglycol) dimethacrylate (PVDF-HFP) to modify the monolayer polypropylene separator (Fig. 17) [35,36,49,52,273,299], ethyl-cellulose and a thermally stable and renewable biomass material also used on the tri-layer (i.e. PP/PE/PP) polymer separator [65]. These coated polymers improve the Celgard mono layer PP separator qualities by acting as pores multipliers, improving the close niche nanofiberous structure and thus significantly reducing the thermal shrinkage of the separator, and improving the electrolyte uptake and its adhesion to the electrode[47,300,301]. To improve both the thermal, ionic conductivity, and interfacial properties with lithium electrodes of these nonwoven fibrous separator mat, inert ceramic oxides fillers such as silicon dioxide (SiO_2), titanium dioxide (TiO_2) and alumina (Al_2O_3) are generally incorporated into the base polymer matrix [54,298,302–306]. The enhancement in the electrochemical properties of these nano-composite separators is generally attributed to the decrease of the polymer crystallinity in the presence of these nanoparticles and the interactions of the ceramic particles with the Lewis-acids in the electrolyte[279]. However, they are often not suitable for high-rate performance and cycle performance class of lithium ion batteries due to their nano-porous structure (Figs. 15 and 16) and the flow property of gel polymer electrolytes at high temperatures [305,307].

For lithium-ion battery applications, microporous polyolefin membranes, and non-woven composite membrane separators should have the ability to absorb and retain electrolyte in order to transport lithium ions between the anode and cathode. Unique techniques such as self-assembly and atomic layer deposition have employed to prepare advanced separators from polymeric materials for use in lithium-ion batteries. Inadvertently, all these techniques have their own drawbacks; hence additional work is needed for the continual development of new separator structures to meet the high performance and energy density of advanced secondary batteries. More details on the discussion of these techniques and methods reported on the current challenges and prospects of electrospun nanofibers used in advanced secondary batteries can be found in these two references [308,309]

7. Conclusions

The development of improved energy storage materials has often been a challenge in meeting the current and future energy requirement. Re-designing the architecture of these materials into nanofiber from various techniques have received attention. Various lithium battery components; electrodes and separators in particular have been produced with nanofiber structure that showed remarkable electrochemical performance. The findings from the various CNFs electrode and separator point to a revolution

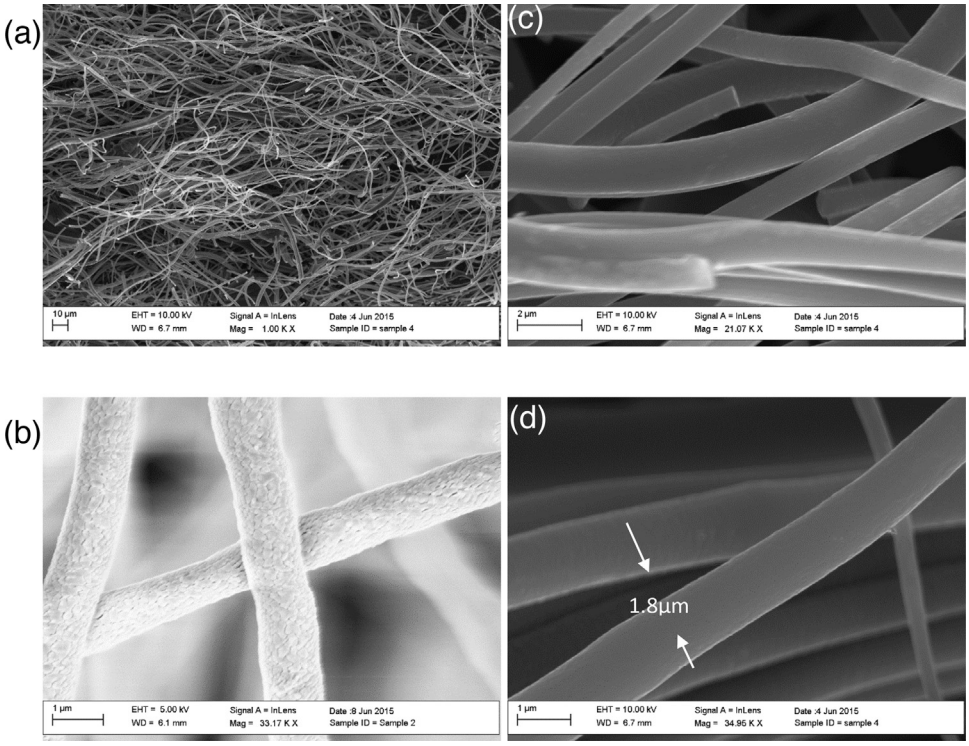

Fig. 16. A typical SEM micrographs showing fibrous separator made from PAN membrane showing a fiber average diameter of <2 µm with fibers with a lot of pores on the fiber strands.

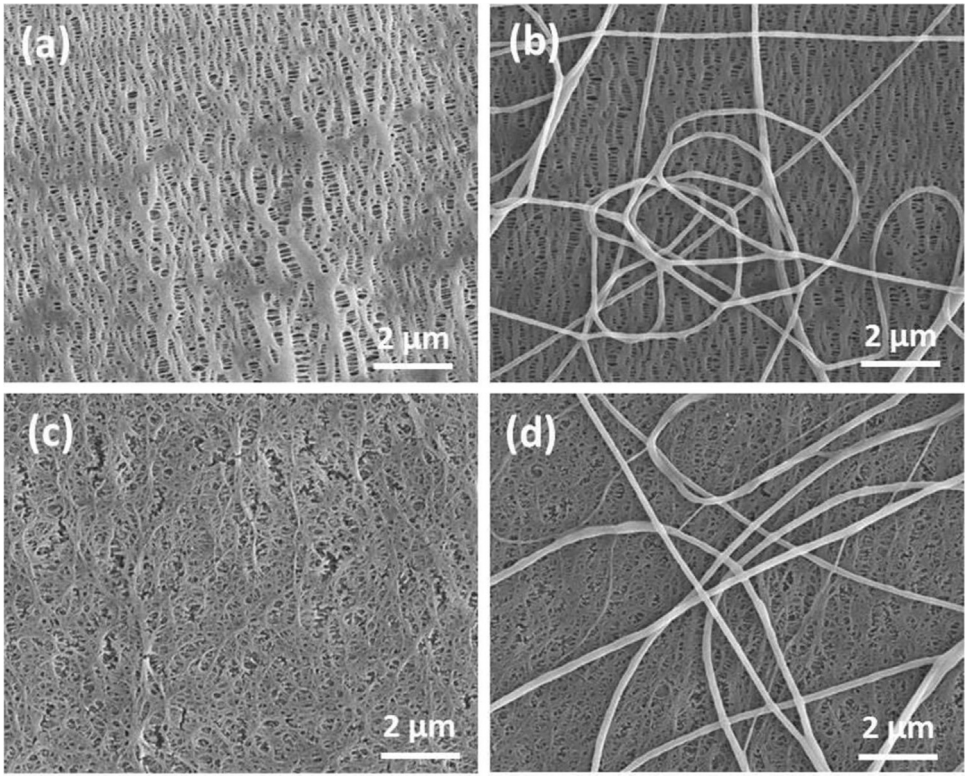

Fig. 17. SEM images of uncoated (a, c) Monolayer polypropylene (PP) separator, (b, d) Monolayer PP coated with PVDF-co-CTFE and PVDF-co-HFP. Reproduced with permission from Journal of Solid State Electrochemistry [49].

in the energy materials with higher energy density and longer cycle life. The major challenges that confront this new frontier is the lack of a scalable method among the various techniques used to make these nanofibers. Most of these methods are lab scale in nature, thus making the commercialization of this new developed materials an illusion. However, there could be an end in sight for this problem with the coming on stream of the forcespinning and electrospinning method with the capability to mass produce nanofibers with good fiber yield from variety of materials. The labyrinth structure of the nanofibers also pose new challenge specially for separator materials as pin-hole size in the materials could be a major headache as these can lead to dendrite penetration leading to an old problem of battery thermal runaway from electrode short circuits. These drawbacks notwithstanding, the nanofiber revolution hold a great promise to the energy storage industry.

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