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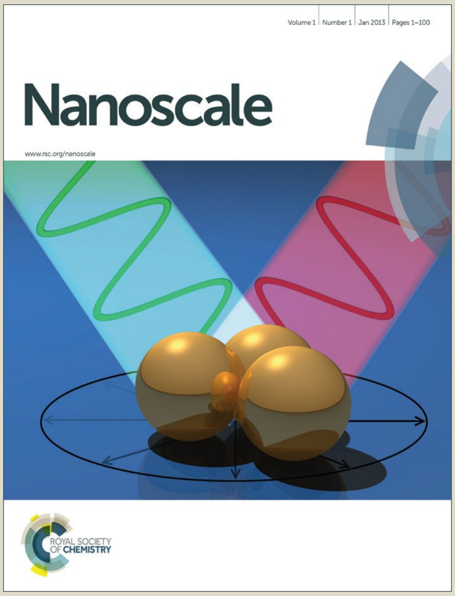

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ARTICLE

Flexible Thin-Film Battery based on Graphene-Oxide Embedded in Solid Polymer Electrolyte

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Enhanced safety of flexible batteries is an imperative objective due to the intimate interaction of such devices with human organs such as flexible batteries that are integrated with touch-screens or embedded in clothing or space suits. In this study, the fabrication and testing of a high performance thin-film Li-ion battery is reported that is both flexible and relatively safer compared to the conventional electrolyte based batteries. The concept is facilitated by the use of solid polymer nanocomposite electrolyte, specifically, composed of polyethylene oxide (PEO) matrix and 1 wt% graphene oxide (GO) nanosheets. The flexible LIB exhibits a high maximum operating voltage of 4.9 V, high capacity of 0.13 mAh cm^{-2} and an energy density of 4.8 mWh cm^{-3}. The battery is encapsulated using a simple lamination method that is economical and scalable. The laminated battery shows robust mechanical flexibility over 6000 bending cycles and excellent electrochemical performance in both flat and bent configurations. Finite element analysis (FEA) of the LIB provides critical insights into the evolution of mechanical stresses during lamination and bending.

Keywords: Flexible lithium ion batteries, polymer electrolyte, nanocomposite, graphene oxide, bendable, solid-state.

Introduction

The worldwide impetus towards flexible electronics, including the ubiquitous touch-screens and wearable sensors, has galvanized research into flexible energy storage devices. [1, 2] Lithium ion batteries (LIBs), based on their superior attributes including high energy density and efficiency, [2-4] are among the leading candidates to be transformed into flexible thin films and effectively integrate with flexible electronics and applications (e.g. can be embedded in textiles/fabrics or directly attached to the biological organs).

The ultimate challenge in this evolutionary phase of flexible LIBs is to achieve mechanical flexibility while maintaining the high electrochemical performance of conventional LIBs including high capacity and cycling stability. Flexible batteries must also yield to a rational assembly process that incorporates a reliable flexible seal.[1] Overall, the next generation LIBs are expected to be lighter, smaller, thinner, and flexible to better facilitate portability and usability. This should be contrasted with conventional batteries that are generally too thick, heavy, and rigid to meet the requirements of burgeoning flexible electronics market. [2-4] In this study, we develop and investigate flexible thin-film Li-ion batteries based on a relatively safer and more stable solid polymer nanocomposite electrolyte (**Figure 1**) that exhibit superior performance with respect to area capacity, energy density and cyclic bending behavior.

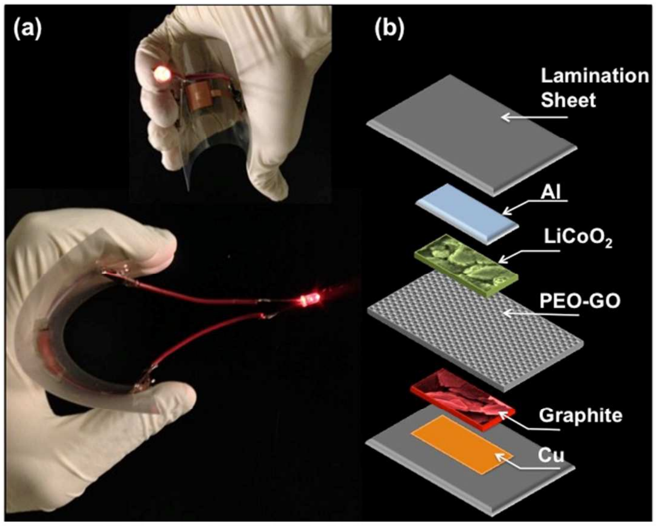

Figure 1 (a) Images of flexible Li ion battery based on solid PEO/1%GO electrolyte powering an LED, (b) schematics of the flexible LIB materials and configuration.

It is important to note that over the past few years, many novel fabrication techniques and innovative structural and material designs including nanoscale materials have been employed to

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Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x realize high performance flexible LIBs [1, 2, 5]. Nano-engineered and nano-sized materials including metal oxide nanowires, [6-8] carbon nanotubes (CNTs), [9-12] carbon nanofibers and graphene [3, 13, 14] have been utilized as electrodes and current collectors [15-18] in flexible LIBs to enhance the interfacial and mechanical properties required in flexible applications subjected to bending, twisting, or folding.

For example, Cui's group [11] constructed a flexible LIB using highly conductive carbon nanotube current collector and a paper dipped in organic liquid as electrolyte/separator, exhibiting high energy density of 108 mWh g^{-1} and 50 bending cycles. Kwon et al. reported a novel design of flexible battery consisting of a spiral Ni–Sn anode with a multi-helix structure, $LiCoO_2$ cathode coated on aluminum wire and organic liquid electrolyte at the center of the structure. [19, 20] The cable battery demonstrated a stable reversible capacity of 1 mAh cm^{-1} with discharging rate of 0.1 C between 2.5 and 4.2 V and excellent mechanical flexibility.

Several flexible LIBs reported have incorporated gel or solid polymer electrolytes. [21-25] Skorobogatiy and co-workers [21] fabricated flexible and stretchable batteries composed of $LiFePO_4$ and $Li_4Ti_5O_{12}$ as cathode and anode, respectively, and investigated various solid polymer electrolytes including PEO, formed directly on the surface of the electrode by polymer solution deposition. Lee et al. [22] reported a novel concept of fabricating printable and bendable polymer electrolytes composed of ultraviolet cured ethoxylated trimethylolpropane triacrylate (ETPTA) polymer matrix, liquid organic electrolyte, and Al_2O_3 nanoparticles for flexible lithium-ion batteries. Wei et al. proposed a flexible all-solid-state battery with monolayer graphene anode, thin polyethylene glycol electrolyte, and Li foil cathode that could bend to a 1 mm radius with capacity of 0.02 mAh cm^{-2} over 100 cycles. [23]

Yang et al. [24] designed a transparent battery created on a flexible polydimethylsiloxane (PDMS) substrate based on a transparent gel electrolyte, exhibiting a capacity of 80 mAh g^{-1} after 15 cycles. Recently, Rogers and co-workers developed a stretchable Li ion battery by using segmented active materials composed of a gel electrolyte, $Li_4Ti_5O_{12}$ and $LiCoO_2$ electrodes and serpentine electrical interconnects with a capacity density of 1.1 mAh cm^{-2} at C/2 and 300% strain capability. [25]

Flexible batteries with ceramic solid electrolytes have also been reported.[26, 27] Koo et al. created an all-solid-state flexible LIB with ceramic lithium phosphorus oxynitride electrolyte, $LiCoO_2$ and Li metal electrodes and PDMS encapsulation exhibiting a capacity of ~0.11 mAh cm^{-2}, an upper voltage of 4.2 V, and an energy density of 2.2 mWh cm^{-3} at a rate of 46.5 µA cm^{-2}.[26] A key imperative in the development of a solid-state flexible LIB is the replacement of the traditional organic liquid electrolyte with high performance solid electrolyte. Ceramic and glass electrolytes such as $Li_{10}GeP_2S_{12}$ can exhibit very high ion conductivity, as high as 12 mS cm^{-1} at 300K, [28] however, the electrode-electrolyte interfacial issues and mechanical brittleness of the electrolyte pose challenges. [29, 30]

Development of high performance flexible solid polymer electrolytes (SPEs) for LIBs has been a major research focus over the past few decades. [31-42] Polymer electrolytes offer many advantages including enhanced safety, flexibility, stretchability, higher thermal and electrochemical stability, and thin film manufacturability.[31] A main challenge associated with SPEs is their poor ionic conductivity especially at room temperature (i.e. 10^{-7} to 10^{-8} S cm^{-1}). [33, 43] Furthermore, non-ideal interfacial contact and high resistance can result in low utilization of the electrode materials. Nanoscale fillers have been shown to enhance the ion conductivity of SPEs without compromising the mechanical properties. [44-48] Among the filler types, graphene oxide nanosheets have recently been reported, including our previous work by Ardebili and co-workers [49], to significantly improve the ion conductivity and thermo-mechanical properties of polymer electrolytes. [49- 51] The electrically insulating properties of GO fillers in PEO have also been previously verified [49].

In this study, a high performance flexible Li ion battery (**Figure 1**) is developed based on a solid polymer nanocomposite electrolyte that exhibits a capacity of 0.13 mAh cm^{-2} and good cycling stability over 100 charge/discharge cycles. The polymer nanocomposite electrolyte is composed of 1 wt% graphene oxide (GO) nanosheets embedded in solid polyethylene oxide (PEO) host. The flexible LIB displays a high maximum operating voltage of 4.9 V and an energy density of 4.8 mWh cm^{-3} at room temperature, which is within the range of values reported for thin film LIBs (1-10 mWh cm^{-3}). [52] The battery is encapsulated using a simple lamination process that is economical and scalable. The flexible LIB exhibits robust mechanical flexibility over 6000 bending cycles and good electrochemical performance in both flat and bent positions.

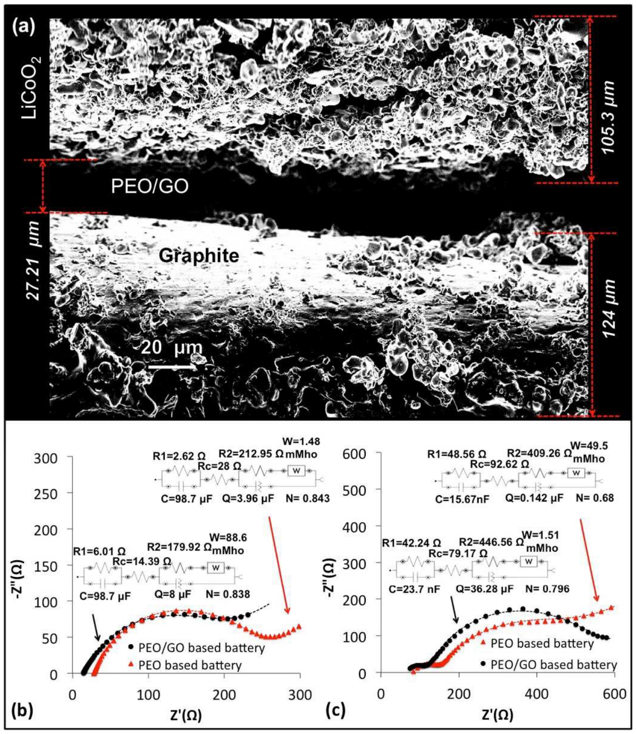

Figure 2 (a) Scanning electron microscopy image of the cross-section of the flexible LIB, (b) impedance spectra of flexible LIB based on pure PEO and PEO/1 wt% GO, compared in the first charge/discharge cycle and (c) after 100 cycles, discharged at 1mA (2C) from 2.0 V - 4.9 V at room condition.

Experimental Section

Polymer Electrolyte Film Preparation: Polyethylene oxide (PEO) with chemical formula C 2n+2 H4n+6 On+1, and 100,000 Mw was purchased from Sigma Aldrich. Lithium perchlorate salt (LiClO4) was purchased from Sigma Aldrich with 99.99% trace metals basis and stored inside the dry glove box under constant Argon flow (H_2O < 0.5 ppm). Solid polymer electrolyte films were prepared by solution casting method. The electrolyte solution was prepared by mixing 2g of PEO and 0.3g of Li salt (ether-oxygen-to Lithium ration (EO/Li) of 16:1) in a 4 oz. jar half filled with the solvent acetonitrile (C_2H_3N). After sonication for 30 min in Branson 3510 Sonicator, the resulting viscous solution was then casted on a Teflon petri dish (area = 76.9 cm^2) and dried at room temperature for ~24 h, to obtain free standing solid electrolyte films with a thickness of ~200 µm. The nanocomposite films were made similar to the fabrication of the pure polymer films except that 1 wt% of GO content was added to the solution. To ensure the removal of the solvent, the membranes were vacuum dried at 50 °C for an additional 24 hours and stored in the glove box prior to testing.

Morphological Characterization of the SPEs: The morphology of the SPE was investigated using a scanning electron microscope SEM Zeiss, Model: Leo 1525 Gemini. Polarized light microscopy (PLM) was used to investigate the crystallinity of the PEO films carried out with Advanced EPI Trinocular Infinity Polarizing Microscope 50x -1600x.

Flexible Battery Fabrication: All components of the flexible LIB were stacked onto two commercial plastic sheets with a simple lamination process inside the glove box. Aluminum foil coated with $LiCoO_2$ (cathode) and copper foil coated with graphite (anode) both with ~0.1 mm total thickness were purchased from MTI Corporation. Lightweight copper tape thin films were used as current collector for both the anode and cathode and were bonded onto the battery electrode materials using the adhesive face of the copper. The area of the solid polymer electrolyte covered all electrode surfaces (area of 4 cm^2). Few drops (5 to 7 wt%) of plasticizer (aqueous 1 M $LiPF_6$ in ethylene carbonate and dimethyl carbonate (EC/DMC 1:1 vol/vol) were added to the polymer electrolyte surface to enhance interfacial contact and ionic conductivity. In the final packaging phase, the battery was laminated and encapsulated with Scotch thermal laminating polyester sheets (thickness of 3 mm) using Fellowes Saturn2 95 Laminator at 120 °C.

Flexible Battery Electrochemical and Mechanical Testing: All batteries were tested at room temperature, using the multichannel Metrohom Autolab and Arbin BT2000 with direct charge-discharge chronopotentiometry procedure and by applying the adequate current rate with defined lower and higher cut-off voltage (2 to 4.9 V). The CV results were obtained from the cyclic voltammetry potentiostatic procedure using the Autolab by varying the voltage from 1.5 V to 5 V with a scan rate of 0.5 mV s^{-1}. The mechanical bending of the flexible LIBs was conducted using a motorized mechanical testing stand Mark-10-ESM301L. Method of shear by tension loading was used to evaluate comparative adhesion between the layers in the batteries using Mark-10-ESM301L at a tensile speed of 5 mm/min with maximum load capability of 10 N.

Results and discussion

The scanning electron microscopy (SEM) image of the cross-section of the thin film flexible Li ion battery is shown in **Figure 2a** revealing a good contact between the battery layers. **Figure 2b-c** (flat battery) and **Figure S2** (bent battery) show the complex impedance spectra of the flexible Li ion batteries. The impedance spectra confirm that adding GO nanosheets reduce the internal and interfacial impedance in the battery. Adding a low content GO can disrupt the ordered packing of semi-crystalline PEO chains and reduce the polymer crystallinity, facilitate salt dissociation, and increase ion conductivity.[49, 50, 53-58] This is further verified using polarization light microscopy (PLM) of the pure PEO and PEO/1 wt% GO depicted in **Figure 3** where the addition of 1 wt% GO is shown to increase the amorphous regions of the PEO.

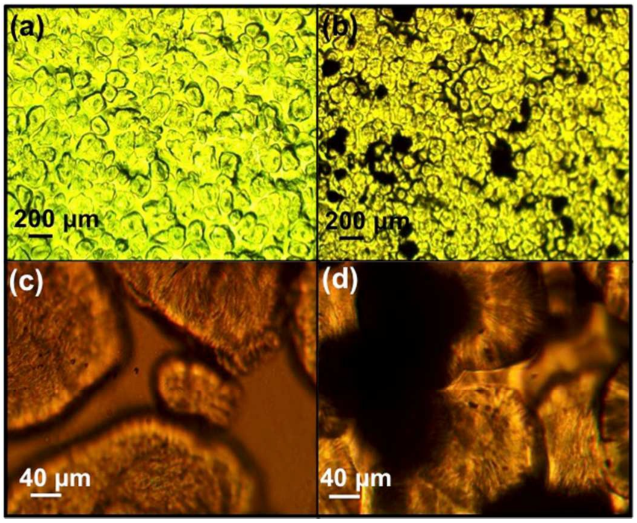

Figure 3 Polarization light microscopy (PLM) of (a),(c) pure PEO and (b),(d) PEO/1 wt% GO.

Figure 4 illustrates the characteristics of flexible Li-ion batteries based on PEO and PEO/1 wt% GO electrolyte, tested in flat and fixed bending positions (**Table S1**). Cyclic voltammetry (CV) measurements (**Figure 4a**) indicate that the batteries have the same CV shape curve, where the redox peaks vary depending on the electrolyte type and the LIB bending position. According to **Figure 4b**, the highest discharge capacity of 0.14 mAh cm^{-2} is recorded for the bent battery (at 18.9 mm radius) based on PEO+1 wt% GO. The electrochemical measurements for 100 charge/discharge cycles of the four batteries are presented in **Figure 4c** where the voltage ranged between 2 V – 4.9 V at a constant current of 1 mA (0.25 mA cm^{-2}). The bent battery with the nanocomposite SPE shows a higher average capacity (0.13 mAh cm^{-2}) compared to that of flat battery. Additional plots of capacities averaged over 3 flexible battery units are also provided in Supporting Information (**Figures S3, S4, S5**). The bending-induced through-thickness compressive forces appear to further enhance the contact between the SPE and the electrodes, and consequently, decrease the contact resistance between the layers of the battery. The flat battery with pure PEO electrolyte exhibits the lowest capacity further confirming the respective higher internal impedance observed in **Figures 2b,c.**

Figure 4(a) Cyclic voltammetry of the flexible batteries, (b) voltage vs. capacity for each battery, (c) discharge capacity during 100 cycles, and (d) coulombic efficiencies of the flexible batteries, discharged at 1mA (2C) from 2.0 V - 4.9 V at room condition.

The coloumbic efficiency of the flexible batteries is depicted in **Figure 4d**. All four flexible batteries demonstrate relatively high charge/discharge efficiencies ranging from 91% to 97%. The flexible batteries were also cycled at different current rates of 1 mA, 1.1mA, 0.5 mA and 0.3 mA over 25 cycles for each rate (**Figure S6**) where all batteries demonstrate good cycling stability, especially at 1 mA (2C) and 0.5 mA (1C), maintaining a high capacity retention of more than 85%. Furthermore, a stable capacity between 0.083 and 0.097 mAh cm^{-2} was delivered by the batteries made with pure and nanocomposite polymer electrolytes at flat position when the rate was reduced back to 0.5mA, suggesting good structural stability of the flexible battery and high reversibility even after high rate of charge-discharge cycles. The characteristics of the charge and discharge cycling at the first and the 100th cycles are also provided in **Figure S7** and **Table S1**. The capacities of four batteries made with composite electrolyte with different percentages of GO filler under fixed bending radius are also presented in **Figure S8**.

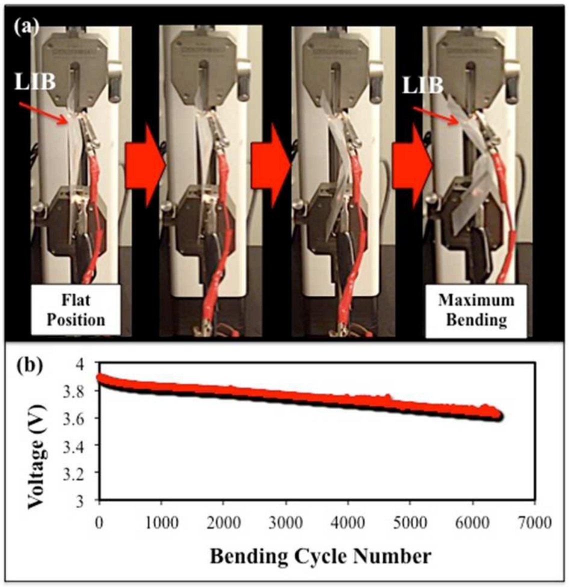

Figure 5 (a) Flexible LIB (based on PEO/1 wt% GO electrolyte) subjected to cyclic bending and *in-situ* voltage measurements, and (b) voltage retention vs. bending cycle number at a speed of 8 mm/s.

Figure 5a shows the images of the flexible battery under cyclic bending test from flat to bent position (17 mm radius) with *in-situ* voltage measurement. The video of the cyclic bending test is provided in the supporting information (**Movie S1**). The results of the flexible LIB bending test in **Figure 5b** show high voltage retention of 93% after 6000 bending cycles. Furthermore, the comparative adhesion of the layers in the flexible battery was evaluated using the method of shear by tension loading (Supporting Information, **Figure S9**). The results indicate that a relatively good adhesion exists between the polymer electrolyte and the adjacent electrodes.

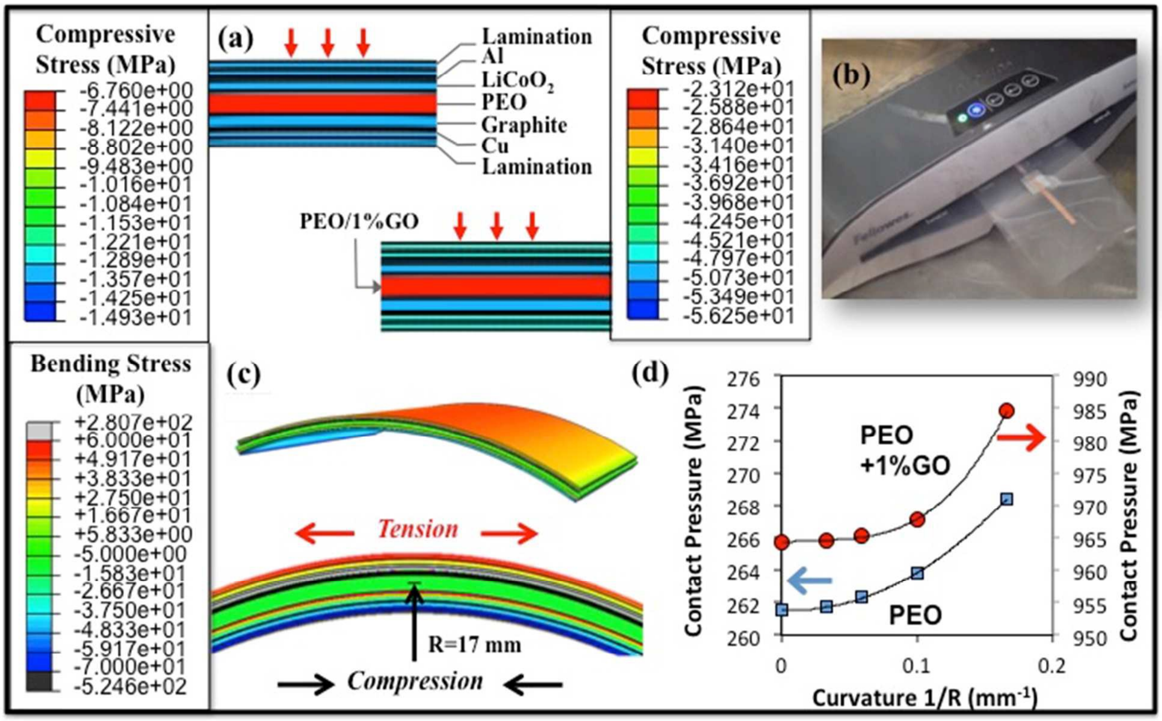

Figure 6 (a) Stresses in flexible LIB after the lamination process predicted by finite element analysis (FEA), (b) a flexible LIB being fed into the lamination machine, (c) bending stresses in laminated flexible battery based on PEO/1 wt% GO electrolyte, and (d) FEA predicted contact pressure vs. bending curvature in flexible LIBs.

Figure 6 illustrates the results of the finite element analysis (FEA) of the flexible LIB subjected to the lamination process followed by bending. The modelling details are discussed in the Supporting Information and the material properties used in the finite element models are listed in **Table S2**. The FEA results indicate that the battery lamination/encapsulation phase produces initial compressive stresses in the battery layers. The compressive stresses shown in **Figure 6a,** are found to be higher in PEO/GO composite-based battery compared to pure PEO based battery. **Figure 6b** shows a photo image of a flexible LIB fed into the lamination machine. **Figure 6c** presents the FEA results of the battery under bending after the lamination process. In the same manner as the experiment, bending was applied as a “column” load that was applied axially to the battery and parallel to the orientation of the layers, to cause the battery to deflect in buckling/bending mode. The column load in the FE model was selected to cause a bending radius of 17 mm at the center of the battery at the neutral axis of the electrolyte. The FEA predicts that the contact pressure increases as bending is applied to both types of flexible batteries (**Figure 6d**) based on PEO and PEO/1 wt% GO electrolytes.

Overall, the capacities obtained in the present study are observed to be higher than that reported for ceramic electrolyte based flexible battery [26] and exhibit lower values compared to gel electrolyte based flexible batteries. [24,25] Furthermore, the polymer nanocomposite electrolyte (PEO/GO) provides enhanced safety and mechanical stability compared to gel electrolytes, [24,25] mainly attributed to the presence of graphene oxide fillers, and a much lower weight percentage of liquid plasticizer used (i.e. 5-7% in this study) relative to other gel-based thin-film batteries (e.g. ~90% [25]). Higher maximum operating voltage (4.9 V) and good voltage retention over 6000 bending cycles are also promising attributes of PEO/GO based flexible battery. A unique advantage of the laminated PEO/GO based flexible battery is the more economical, simple, and scalable fabrication procedure compared to other fabrication techniques reported. [26] Regarding the electrochemical stability during charge and discharge cycling, ceramic electrolyte based battery reported by Koo et al. [26] exhibits the highest capacity retention among the reported thin-film flexible batteries. Future studies should focus on enhancing the encapsulation of the flexible batteries to further reduce the capacity fading. The electrochemical performances of previously reported flexible thin-film batteries are also summarized in **Table S3** in the Supporting Information.

Conclusions

In summary, flexible and relatively safer lithium ion batteries were fabricated based on pure and nanocomposite solid polymer electrolytes (SPEs). Flexible batteries with 1% graphene oxide embedded in polyethylene oxide polymer host showed enhanced performance. Considerably high charge-discharge capacities with relatively low fading rates over 100 cycles were observed while the cyclic mechanical bending test of the flexible batteries reveal high voltage retention. The battery is encapsulated using a simple lamination process that is economical and scalable. The laminated flexible LIB offers good electrochemical performance and can function over large ranges of mechanical deformation. Finite element analysis reveals the formation of initial compressive stresses during the lamination process and increased contact pressure during bending. The nanocomposite SPE based flexible Li ion batteries pave the way for new forms of safer and more cost-effective energy storage devices that would adapt to stringent shape and space requirements of modern flexible applications.

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