Chapter

Micro Nano Manufacturing Methods for Chemical, Gas and Bio Sensors, Water Purification and Energy Technologies

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Abstract

This chapter reports on the various methods of fabricating and manufacturing micro and nano sensor, membrane and energy devices. Firstly, the characteristic often sought after by scientists and engineers for effective and efficient performance of these technologies were thoroughly discussed in details together with the characterization techniques for evaluating them. Several state-of-the-art fabricating techniques for sensor devices, water and medical based-membranes, solar cells and batteries were also discussed.

Keywords: micro-nano device, fabrication, sensors, manufacturing, membrane, battery, solar cell

1. Nanoscience and nanotechnology

Nanoscience can be described as the study of the phenomena and manipulation of materials at atomic, molecular and macromolecular scales, where properties differ specifically from those at a larger scale (macro scale). The macroscopic objects we see around us in our day-to-day activities are the products made from bulk materials. These objects possess physical properties that are in some way different from nano and the intermediate scale called micron-sized material (such as grains of sand or dust produced during volcano eruption). However, bulk and nanomaterial may share the same constituent but the dimension or length scale usually distinguishes between the two groups [1, 2]. Nanometer scale is conventionally defined as 1 to 100 nm which simply means one billionth of a metre (10^{-9} m). The lowest limit of nanometer size range is normally set to 1 nm which is very close to the length of a single atom since the atomic radius is just by a little femtometre less than 1 nm. However, nanoscience is not just the science of small-scale material but also the science in which materials with small dimension (in other words shape) show new physical phenomena. For instance, the principles of classical physics such as energy, force, momentum, space, time, and so on, that govern the behavior of macroscopic and microscopic systems (bulk material) are no longer applicable to nanoscale materials [3–4]. This Nanoscience is not new per se, it is a name that was

given to a number of fields of research that share common principles, and hence it is referred to as an interdisciplinary science. Nanotechnology integrates a wide range of sciences which includes; Physics, Chemistry, Biology, Microbiology, Engineering, Surface Science, and Biotechnology, and apply them to practical devices [5]. There are two major approaches normally employ in fabrication techniques namely; **top-down approach** (Larger to smaller: a materials perspective) and **bottom-up approach** (Simple to complex: a molecular perspective). Top-down approach involves creating Nano-scale materials by physically or chemically breaking down larger materials. These include statistical mechanical effects, as well as quantum mechanical effects. Solid-state techniques can also be used to create devices known as nanoelectromechanical systems or NEMS, which are related to micromechanical systems or MEMS [6] while bottom-up approach simply involves simple to complex: i.e. a molecular perspective technique. These techniques are used today to manufacture a wide variety of useful chemicals such as pharmaceuticals or commercial polymers. Molecular nanotechnology, sometimes called molecular manufacturing, describes engineered nanosystems (nanoscale machines) operating on the molecular scale. Molecular nanotechnology is especially associated with the molecular assembler, a machine that can produce a desired structure or device atom-by-atom using the principles of mechanosynthesis [7].

2. Significance of micro and nano fabrication in novel devices and technologies

Micro and nano fabrication is an essential process in the manufacturing of novel devices and technologies. Many sciences, technology and engineering oriented products are developed using the concept of micro and nano fabrication. From radio transistors, integrated circuits, personal computers, to micromechanical systems (MEMS), transducers, sensors, batteries and super capacitors, solar cells, water treatment membranes and filters and other novel devices, micro and nano techniques have played significant and important role in realizing reliable technology. However, huge credit relating to the success of these technologies must be ascribed to the materials development and analyses techniques such as the analytical, macroscopic, microscopy and spectroscopy ones.

For instance, before one can realize product of gas sensor, chemical sensor and biosensor device, especially in the case of metal oxide semiconductors, carbon materials and polymers, critical studies and analyses of the materials properties is required to qualify the performance of the sensing element. The first set of investigation which must be performed on the materials intended to build these devices are crystal structure and microstructures, morphological and surface roughness studies, defects studies, thermal stability and adsorption property [8–10]. Hence, the material must be thoroughly characterized with X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HR-TEM) methods to study its crystal structure. X-ray diffraction spectroscopy which is commonly used technique for characterization of crystalline materials provides information about elemental analyses such as structures, phases and preferred crystal orientations. Physical measurements like average particle size of material, homogeneous and inhomogeneous strain and crystal defect could also be estimated from the data collected using XRD technique [8–11]. The HRTEM approach has been severally employed in sensors material research to unveil material's crystallographic structures at an atomic scale [8–9]. The scanning electron microscope (SEM), scanning tunneling microscope (STM) and atomic force microscope (AFM) are important for all surface structure studies such as morphology, particles distribution, nanoscale topography and

surface roughness [9–13]. These are also essential properties needed to be analyzed for chemical, gas and bio sensors devices fabrication. Other properties necessary to be investigated for these types of application include quantitative analysis of the material's elemental composition and chemical state. This study is often achieved using X-ray photoelectron spectroscopy (XPS) [9–11]. The adsorption ability and properties of the sensors materials are usually evaluated using the popular Brunauer–Emmett–Teller (BET) technique which relies on the physical adsorption (physisorption) of gas molecules on the surface of solid-state materials. With this technique, information about the specific surface area, microporous, nanoporous and mesoporous of a sensor's material can be acquired [9, 14–16].

In the same way, materials for fabricating water and medical membranes and energy devices such as solar cell, lithium and sodium ion batteries also required critical studies with the above materials characterization techniques before the manufacturing process could be initialized. The properties which Materials Scientist and Engineers are usually sough for when building in solar cell architecture are crystal and microstructures of all the semiconductors and polymers involved. These techniques are necessary to unveiled the effect of the grain's boundaries on the charge transfer of the cell, especially when device is of a p-n junction or multi-junction type [17]. This often help materials engineers in proper understanding of interfacial properties of the cell [18]. Studies of morphology, surface roughness and topology is also of a great importance when evaluating the solar cell materials for prototyping and manufacturing. This is needed to ensure a homogeneous film surface in a bid to enhance the transport of the charges for an improve energy conversion efficiency (ECE) [19]. Thermal stability studies with Thermogravimetric analysis (TGA) are another important method adopted by materials scientist to study the degradation of solar cell device [8, 20]. Lithium and sodium ion batteries are not an exception when it comes to their materials development and analyses. TGA techniques are often used to study the thermal stability, XRD and HRTEM for crystal, micro structures, particles size analysis and how monodisperse the particle are before fabricating the device. The SEM, STM and AFM techniques are being employed for particles morphology, surface roughness and topography [21].

XPS is another important technique for qualifying materials for lithium and sodium ion battery application. XPS is suitable to give important information about the interaction of membrane-based materials with electrolyte materials and further assist to develop a definite insight of interfacial structure and as well performance of the battery. From one of the previous published studies XPS was employed and useful information of the membrane interaction with vanadium electrolytes was revealed which led to understanding of interfacial structure and battery performance [22]. Nanosized fibers have great advantages owing to their high specific surface area to volume ratio, electrospun nanofibers have find their useful applications in the field of clean energy (solar cells, fuel cells and batteries), electronics, health (biomedical scaffolds, artificial organs), and environment (filter membranes) [23].

3. Advance manufacturing methods for chemical, gas and bio sensor applications

Prototyping and manufacturing sensor devices (Gas, Chemical or Bio sensor) required that the sensors materials be deposited or coated on an electrode for easy contact and connection to the device electronic circuitry or source measuring unit of the gas sensing and test station. Interdigitated electrode (IDE) type have been widely used for sensors laboratory research, prototyping and manufacturing of sensors and related products. This is a cost-effective method which often made from an

aluminum oxide (Al_2O_3) substrate whose front-side surface is coated with platinum (Pt) metal in comb-like structures for sensors electrical signal measurement and the rear-side coated with nickel (Ni) metal as Microheater [9, 24]. **Figure 1** showed a schematic layout of KSGA565 KENOSTATIC gas detection station where μ-nano IDE was used as the sensor's electrode. The layout consists of an enclosed chamber called sensing chamber containing IDE with deposited sensor material. The front-side of the IDE is connected to the Keithley pico-meter source meter and the rear-side to the power supply.

During fabrication, sensor materials are usually deposited onto the IDE using micro-nano deposition technologies such as chemical vapor deposition (CVD), pulse laser deposition (PLD), physical vapor deposition (PVD) and magnetron sputtering technique [25–27]. These technologies are physical methods which have been reported to offer thin and homogenous film surface with excellent gas, chemical and bio molecule sensing properties. These technologies have also been used severally to deposit non-IDE pattern like glass, silicon wafer etc. for sensors and related device fabrication [28].

The printed patterned substrate and Lab-on-a-chips are another micro-nano contacting and printing technology commonly used when manufacturing Gas, Chemical or Bio sensor devices. These techniques are expensive and regarded as state of the art method which required specialized equipment like photolithography (PL), plasma enhanced chemical vapor deposition (PECVD) and electron beam lithography (EBL). The methods offer patterned deposition of nanostructures such as nanowires, nano-rods, nano-tubes etc., high precision contacting, highly aligned printing and deposition onto flexible substrates as advantages over others [25–30].

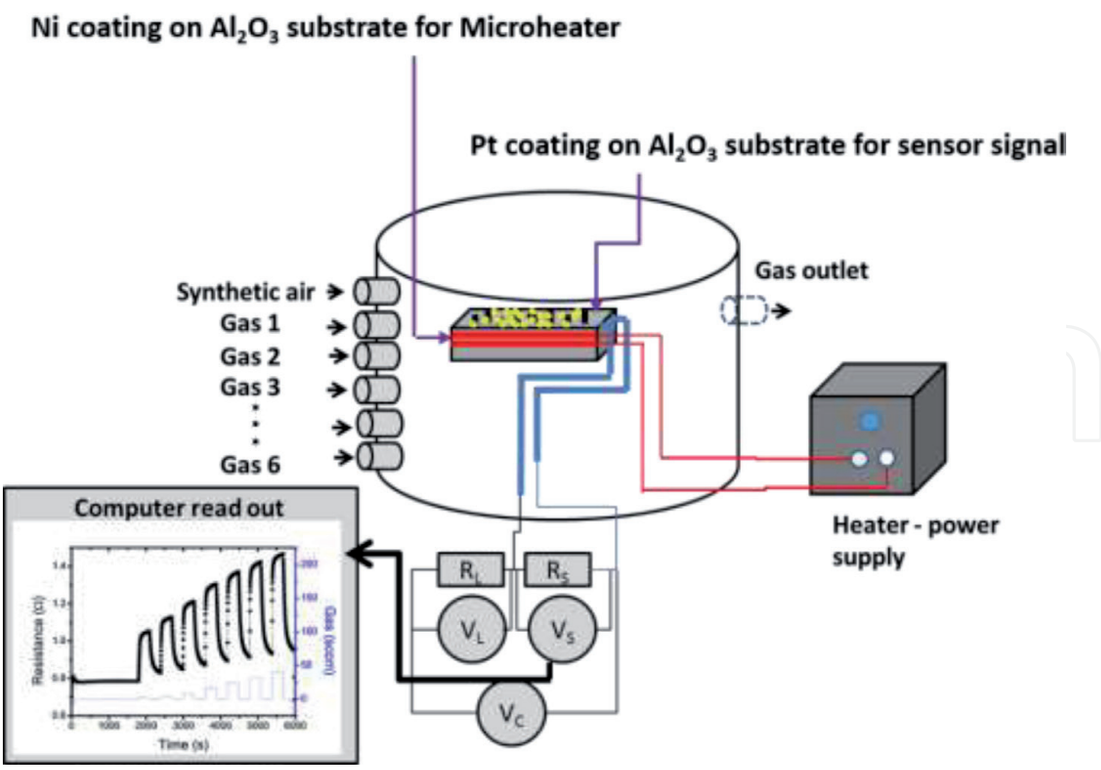

Figure 1.
Schematic diagram of KSGA565 KENOSISTEC gas sensing station illustrating how the μ-nano IDE sensor can be tested. The electronic circuit displays of the gas sensor's element showed R_L, which is the load resistor connected in series with the sensor's element ($R_L = (V\text{-}V_S)/I$). V, is the voltage on the R_L, $V_S = V_C - IR_L$, represent the sensor's signal voltage. V_C is a constant voltage applied on the R_L and sensor's element and finally, R_S is the sensor's resistance ($R_S = V_S/I$). Adapted from Ref. [24].

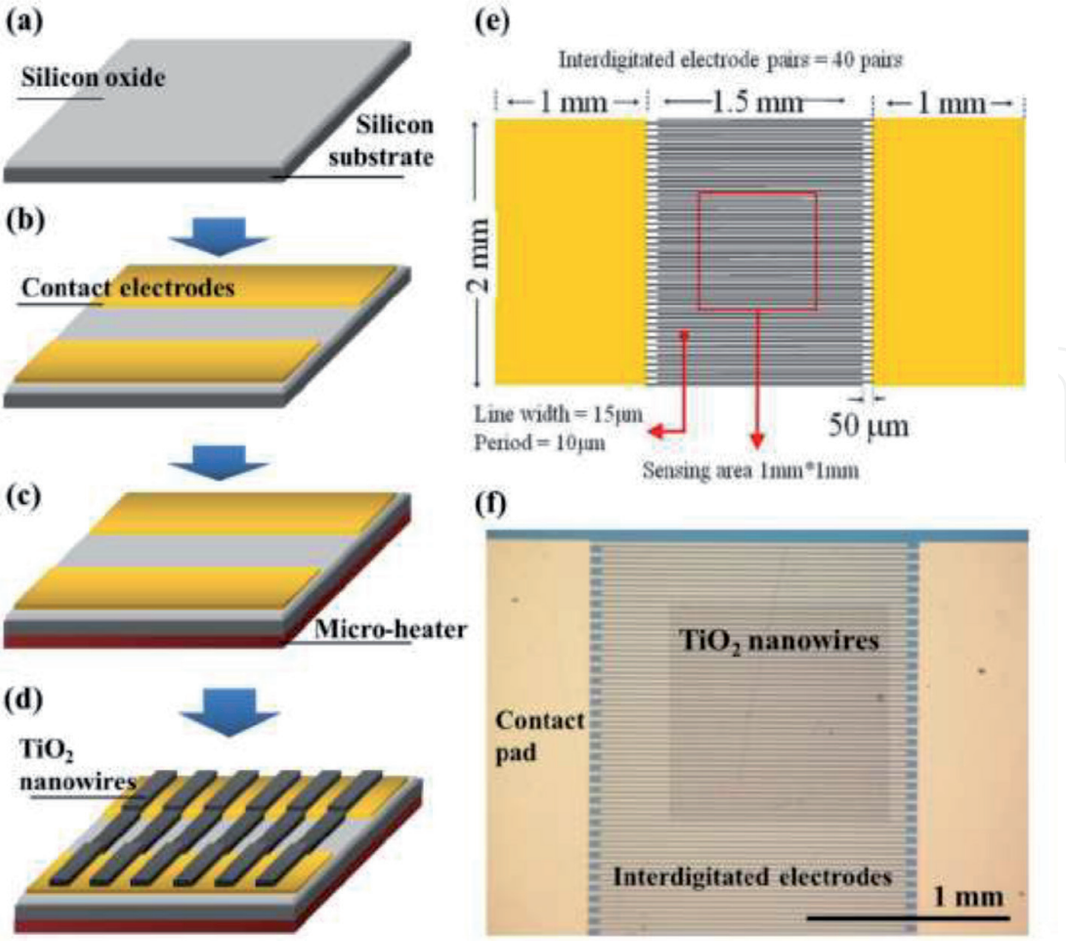

Figure 2.
Fabrication process of electron beam patterned TiO2 based gas sensor; (a) oxidation process of Si wafer, (b) Cr/Au contact fabrication, (c) microheater fabrication, (d) photoresist deposition, lift-off and TiO2 nanowire arry deposition, (e) showed the dimensions of each section of the device and (f) the optical image of the entire device. Adapted from Ref. [30].

A typical process involving the fabrication of TiO2 nanowires-based gas sensor is shown in **Figure 2**. The materials used for the fabrication are; p-type silicon wafer (**Figure 2(a)** and interdigitated Cr/Au electrodes which was initially fabricated using PL process on an oxidized Si substrate (**Figure 2(b)** [30]. The Cr and Au thin films were also blank deposited on the rear-side of the silicon wafer in an interdigitated fashion to make heating element (Microheater) (**Figure 2(c)**). Thereafter, EBL approach was used to pattern the chip surface and to produce photoresist on the film before depositing the p-type TiO2 on the top of the chip with aid of sputter machine. The photoresist was later lift-off to form the TiO2 nanowire array as shown in **Figure 2(d)**. **Figure 2(e)** showed the dimensions of each section of the device and **Figure 2(f)** the optical image of the entire device.

4. Advance manufacturing methods for water purification, lithium ion batteries and medical applications

Electrospinning is one of the techniques suitable for the fabrication of materials through innovative technology. Membrane-based technologies through electrospinning have been employing for the fabrication of both nano- and micro-based materials which finds useful applications in various fields such as in the water purification, lithium ion batteries, medical applications etc.

4.1 Applications of electrospun fibers in water purification

Electrospinning is a fabrication technique that involves application of a high electric field to generate nanofibers from a charged polymer solution or melt. it is a useful method for the fabrication of complex structures consisting on continuous fibers. The morphology of electrospun fibers can be controlled by adjusting experimental parameters, such as precursor solution concentration, type of spinneret, voltage and the spinneret-collector distance. Using this technique affords us numerous benefits such non-complicated and inexpensive equipment, easy to modify, ability to carefully monitor the morphology of materials, and as well almost all polymers with even high molecular are applicable in the synthesis [23]. The chemical properties of electrospun fibers are mainly influenced by two factors: hydrophilicity and chemical composition of the fibers. The characterization of the mechanical features is critical for the electrospun nanofibers. It can be stated that the electrospun nanofiber membranes are appropriate for the pressure driven membrane procedures where the target product is mainly the permeate phase, for example, water/wastewater treatments [31]. Water purification is mostly defined by filtration through size exclusion or adsorption. The water purification process is classified according to the average pore size of the materials and applications include microfiltration (MF) (0.1-10 μm), ultrafiltration (UF) (0.001–0.1 μm), nanofiltration (NF) (0.001–0.01 μm), reverse osmosis (RO) (0.0001–0.001 μm), and forward osmosis (FO) (0.0001–0.001 μm) [32]. In a study conducted by Mahadevappa Y et.al, where electrospinning was used to fabricate nanofibrous membranes for MF applications using polyvinyl alcohol. Owing to its cost-effectiveness, stability (thermally and chemically) and non-degradability, poly (vinyl alcohol) was selected as a precursor in the fabrication process [33]. However, the poly (vinyl alcohol) nanofiber membranes, produced from electrospinning process, must be treated with cross-linking agents for preparing a 3-D waterproof system before being utilized as water filters [31]. Liu's team has introduced a nanofiber MF membrane that required doping with copper oxide (CuO) nanosheets (**Figure 3**). The fabricated membrane has a separation efficiency of >99.89% for polystyrene (PS) microspheres with a diameter > 300 nm in water [34]. The introduction of such functional materials can not only achieve the corresponding modification purpose, but also enhance static electricity to improve the strength of individual nanofibers. Stable high porosity, good interconnectivity, and ultra-thin membrane thickness are key major factors responsible for its strong permeate flux and excellent bacteria rejection efficiency [35].

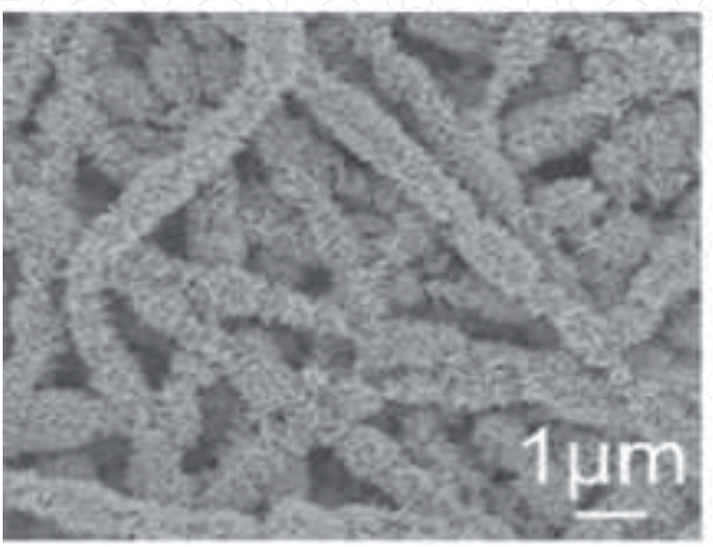

Figure 3.
Morphology of PVDF/CuO nanosheet nanofiber MF membrane. Adapted from Ref. [34].

4.2 Applications of electrospun fibers in lithium ion batteries

Electrospun carbon nanofibers exhibit favorable properties, such as nanometer-sized diameters, high specific surface areas, and web morphologies, making them highly suitable for an anode material. Electrospinning has been identified as the most promising route for designing novel anode materials and structures, owing to its simple process setup. The electrospinning technique is suitable for the implementation of existing anode material research based on the process being able to mass-produce anodes [36]. In a study conducted by Peng et al. and co-workers, the porous carbon nanofibers were synthesized using a PAN/polymethyl methacrylate (PMMA) precursor solution with the aid of electrospinning technique. PMMA is immiscible with PAN, during the course of preparation macro phase separation was observed and was then thermally treated at high reaction temperature-800°C which caused elimination of PMMA while creating pores on the surface of the fiber. In order to investigate the fiber morphology and the electrochemical performance of carbon nanofibers, the author varied the concentration effect of PMMA in the precursor solution. The variation of PMMA showed that its addition significantly improves the surface area and pore volume of the prepared fibers.

The morphologies of the electrospun fibers after carbonization are shown in **Figure 4**. In **Figure 4(a)**, the carbon nanofibers prepared using neat PAN exhibited long and bead-free morphology. By contrast, the PAN/PMMA-derived carbon nanofibers were uneven and interconnected, particularly for 5:5 PAN/PMMA-derived carbon nanofibers (**Figure 4(c, e)**). The interconnected structure was attributed to the presence of PMMA. PMMA is a thermally liable polymer, which melts during pyrolysis. **Figure 4** also provides the inner structure of the nanofibers. As observed in **Figure 4(b)**, neat PAN-derived carbon nanofibers were internally nonporous. The introduction of PMMA in precursor solution facilitated the development of pores and channels inside the carbon nanofibers (**Figure 4(d, f)**). The availability of the fiber morphology consequently resulted to highly efficient discharge capacity compared to counterpart neat PAN-prepared carbon nanofibers. Therefore, the 5:5 PAN/PMMA-derived carbon nanofibers exhibited a discharge capacity of 446 mAh/g at a current density of 150 mA/g. They exhibited a discharge capacity of 354 mAh/g after 100 cycles at a current density of 200 mA/g equivalent to 67% retention, demonstrating the favorable cycle stability. The significance of their study was based on the manipulation of morphology of electrospun carbon nanofibers for the use as anode materials for lithium ion batteries application to secure good performance. Therefore, it can be said that the superior electrochemical performance of the PAN/PMMA-derived carbon nanofibers was mainly attributed to the prevalent mesopore volume and the high-specific surface area which earned them desired contact between the fibers and electrolyte and consequently improved the diffusion of electrolyte ions into the material [37].

4.3 Electrospun fibers in biomedical applications

Electrospun nanofibers are materials of multi-applications, hence have been widely studied in the field of biomedical and tissue engineering owing to their good characteristic properties and suitability to be incorporated into various morphologies to stir the desired influence in them, such as nonwoven form, aligned nanofibers, core–shell structure, and hybrid nanocomposites. The interesting characteristic properties of electrospun nanofibers- loose structure, high porosity, and superb flexibility possess perfect features to mimic the extracellular matrix (ECM) for cells to grow and, therefore, they have been employed in tissue engineering applications [38]. In a study, a composite nanofiber scaffold made of poly (vinyl alcohol)–poly (vinyl acetate) (PVA–PVAc) was manufactured and subsequently

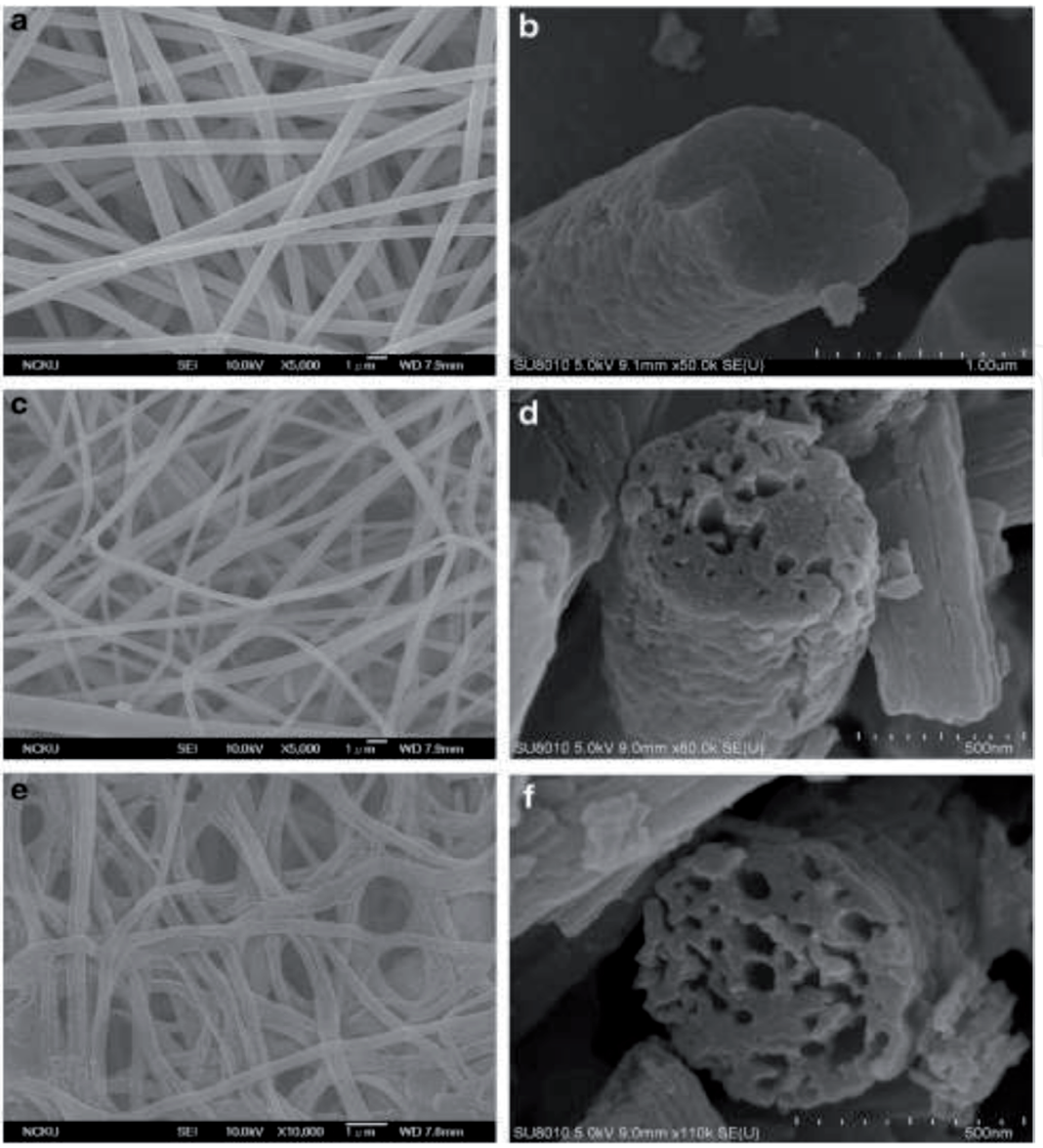

Figure 4.
SEM micrographs of electrospun fibers carbonized at 800°C. a and b PAN/PMMA = 10:0; c and d PAN/PMMA = 7:3; and e and f PAN/PMMA = 5:5. Adapted from Ref. [37].

loaded with simvastatin superficial layer to obtain an efficient osteogenesis process by the continuous release of the drug [39]. The use of PVA was attributed to its environmentally benign, elasticity, flexibility, proper mechanical properties, nontoxicity, swelling ability, and biodegradability. PVA is not stable in aqueous state, this instability however creates limitation in its use in drug delivery processes. In order to overcome instability issue, PVA was then crosslinked with biocompatible and biodegradable PVAc that possess hydrolysable groups. Afterward a simvastatin drug was loaded into the blended solution of PVA–PVAc in order to promote the efficiency of bone regeneration. The obtained results revealed good bioactivity, inducing the precipitation of bone-like apatite minerals on its surface and successfully simulating physiological conditions for cell growth [39]. Electrospun nanofibrous dressings have high surface-to-volume ratio, allow gas permeation, help to regulate wound moisture, enhance tissue regeneration, improve removal of exudates, and have high porosity, which qualifies them to be used in wound healing treatment. Previous studies have shown low inflammatory reaction and fast re-epithelization with the use of nanofiber-based wound dressing [38].

Bredigite polymer electrospun nanofibers have been widely investigated to access their suitability in wound-dressing processes. It has been reported as a

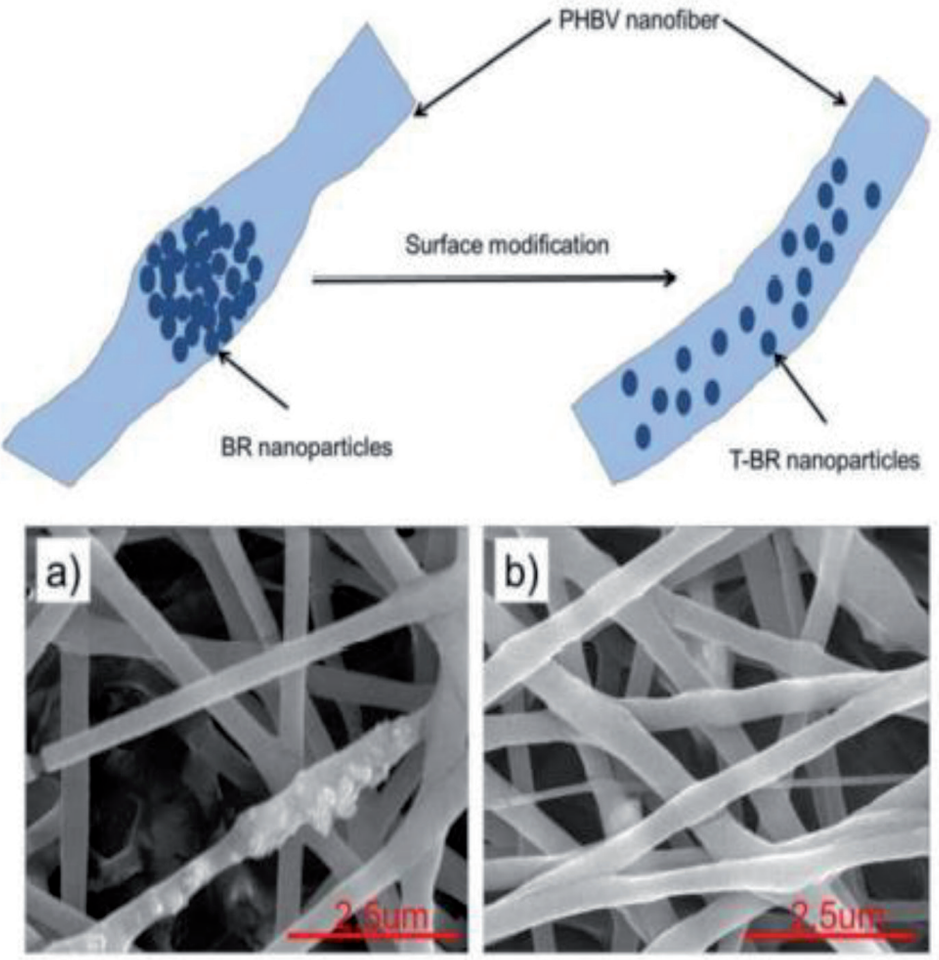

Figure 5.
Schematic illustration and SEM images of PHBV nanofibers containing 15% of (a) bredigite (BR) and (b) T-BR nanoparticles. Adapted from [40].

scaffold however, results showed that while the bioactivity of the composite nanofibers was improved, and the low dispersibility and high agglomeration of nanoparticles decrease the efficiency of prepared electrospun nanofibers [40]. In another attempt, bredigite (BR) nanoparticles were modified by an organosilane coupling agent in order to increase its dispersibility [40]. The SEM results reveal that the modified BR nanoparticles are widely dispersed in the body of the nanofibers without any agglomeration (**Figure 5**). Moreover, the mechanical and biodegradation rate of the scaffolds dramatically improved after BR modification.

5. Advance manufacturing methods for energy applications

The fabrication of energy device material such as thin film photoelectrode for splitting water into H_2 and O_2 during photoelectrochemical process and the development of photovoltaic cells, for solar energy conversion is tasking and difficult, requiring a special operational technique. For efficient solar energy capturing and conversion in photovoltaic cells, effective separation electrons and holes in photoelectrode required [41, 42]. This depend on the deposited semiconducting material ultrathin layer, evenly coated and tightly connected to conductive layer. Atomic layer deposition (ALD) as a vapor phase technique is capable of producing thin films of different materials. ALD is applicable in the fabrication of uniform and ratio structures with thickness control to Angstrom level, and tuneable film composition [43]. Due to all this advantages, ALD has emerged as a powerful tool for many

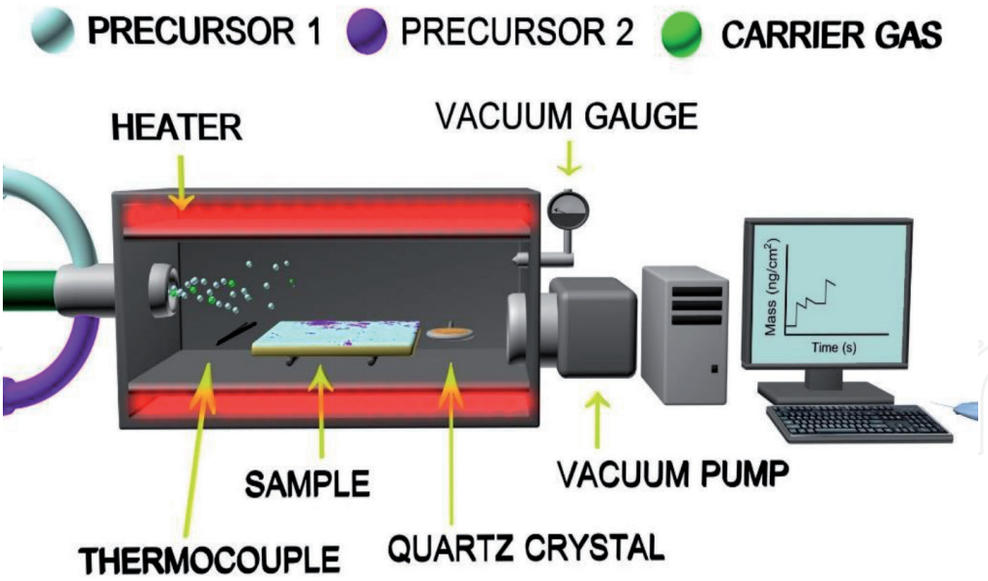

Figure 6.
Atomic layer deposition (ALD) reactor. Adapted from Ref. [43].

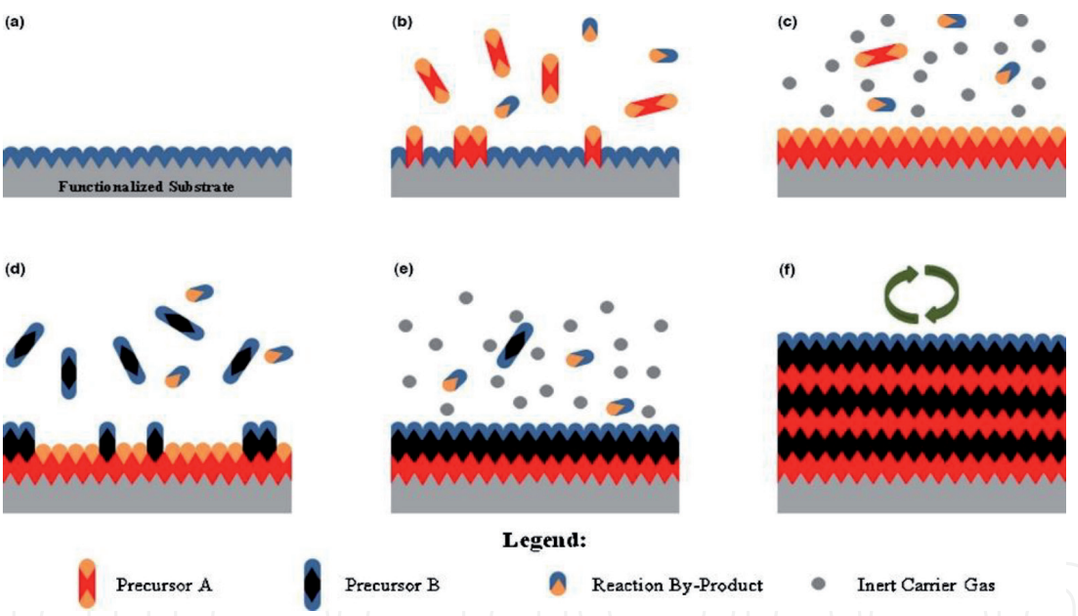

Figure 7.
Schematic illustration of ALD process schematic of ALD process. (a) Substrate surface has natural functionalization or is treated to functionalize the surface. (b) Precursor a is pulsed and reacts with surface. (c) Excess precursor and reaction by-products are purged with inert carrier gas. (d) Precursor B is pulsed and reacts with surface. (e) Excess precursor and reaction by-products are purged with inert carrier gas. (f) Steps 2–5 are repeated until the desired material thickness is achieved. Adapted from Ref. [45].

energy research material fabrications. ALD method has been a useful tool for the deposition of ultrathin-layered semiconductors on conductive substrate.

ALD process generally consists of sequential alternating pulses of gaseous chemical precursors that react with the substrate, these individual gas-surface reactions called 'half-reactions' and appropriately make up only part of the materials synthesis. During each half-reaction, the precursor is pulsed into a compartment under vacuum (< 1 Torr) over a selected extent of time to allow the precursor to fully react with the substrate surface through a self-limiting process that leaves no more than one monolayer at the surface [44, 45]. Then, the chamber is purged with an inert carrier gas (typically N_2 or Ar) to remove any unreacted precursor or reaction by-products.

This is then followed by the counter-reactant precursor pulse and purge, creating up to one layer of the desired material. This process is then cycled until the appropriate film thickness is achieved (**Figures 6** and 7).

6. Conclusion

The interdigitated electrode is reported as cost effective method for prototyping gas, chemical and bio sensor and the method is widely used for laboratory research purpose. State of the art techniques such high tech semiconductor deposition instruments, photolithography and electron beam lithography are used for commercial sensors built with printed electronics and Lab-on-a chip. Electrospinning method is highly important in the fabrication of micro and nano porous fibers for the manufacturing of membranes and battery devices. This method has also been identified for designing anode materials suitable for lithium ion battery fabrication. Atomic layer deposition is useful for producing ultrathin layer-layered semiconductors with inherent properties necessary for efficient energy capturing. This deposition technique is very useful in the manufacturing of photovoltaic cells and related devices for effective separation electrons and holes in photo-electrode.

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