

Recent Patents on Fabrication of Nanowires

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Abstract: Nanowires are the building blocks of future nanodevices and thus methods for fabricating nanowires of various materials in various forms are fundamentally important. Although nanowires have been intensively studied, there are only a few methods that showed promising characteristics for practical applications. Here, we intend to review those patents, which enable nanowire growth to be more controllable and feasible for applications. Various methods for fabricating metal, semiconductor and organic nanowires with promising features are reviewed, where some emphasize the characteristics of individual nanowires, others address the uniformity and alignment of an array of nanowires as a whole. The patents for fabricating nanowires of various materials are introduced in the first part. In the second part, the patents to improve crystalline quality, morphology, uniformity of nanowires are introduced. Finally, the patents for growing aligned nanowire arrays and aligning dispersed nanowires are reviewed.

Keywords: Nanomaterial, nanowire, catalyst, superlattice, lattice mismatch.

INTRODUCTION

Low-dimensional nanomaterials are a new class of advanced materials that have been receiving a lot of research interest in the last decade due to their superior physical and chemical properties. Among them, nanowires are especially attractive for nanoscience studies and nanotechnology applications. Compared to other low-dimensional systems, nanowires have two quantum-controlled directions, while still leaving one unconfined direction for electrical conduction. This enables nanowires to be used as nanoconductors for various nanodevice applications. Nanowires have been demonstrated to possess excellent electrical [1], optical [2,3], mechanical [4], piezoelectrical [5], and field emission [6] properties for applications in various fields. So far, nanowires can be fabricated using many methods, including thermal chemical vapor deposition (CVD) [7], metal-organic CVD [8], hydrothermal method [9], electrochemical deposition [10], sputtering [11], etc. and great attention has been paid to the continued improvement of these fabrication methods. Although there has been significant advancement recently made in fabricating nanowires, nanowires are still far from practical applications. The obstacles to more applications are the lack of efficient methods to control almost all aspects of as-grown nanowires and, therefore, more advanced methods need to be developed. In this review paper, we focus on the methods for fabricating nanowires, improving crystalline quality, morphology, and the alignment of nanowires based on the patents recently issued by US [17-18, 20-21, 26, 29-31, 39-42, 44] and WO [14, 38] Patent Offices. It should be noted that most of the methods could only be applied to grow certain material systems with the corresponding growth mechanisms specifically to the materials used.

FUNDAMENTAL METHODS FOR FABRICATING NANOWIRES

Ever since the demonstration of carbon nanotube growth by arc discharge in 1993 [12], many other methods via different growth mechanisms have been proposed aiming to yield more consistent nanowire performance in mass production in an effort to overcome at least part of the main drawbacks met in arc discharge. The first variant has been known as vapor-liquid-solid (VLS) growth [13], which enables the one-dimensional nanostructures growth to be more controllable with the assistance of metal catalysts prepared from particles or thin films. After injecting precursor gases, one-dimensional nanostructures such as carbon nanotubes and semiconductor wires could be nucleated and grown from the catalytic particles at an elevated temperature [14], as schematically shown in Fig. 1. Consequently, one-dimensional nanostructures growth can be controlled to some degree by the catalysts (for example, the density, diameter and length of the nanowires). The relative ease of handling catalysts on substrates and the chemical reactions has given rise to extensive research employing this method as a basis. Fig. 2 is a scanning electron microscopy (SEM) image of the ZnO nanowires grown by the VLS approach using Au catalysts [15]. Besides, the VLS method is also widely employed to synthesize nanotubes [16].

However, the metal catalysts involved in VLS growth seriously contaminate the grown nanowires and complicate the entire nanowire harvest procedure, which demands an extra purification process for removing the catalysts. Therefore, one of the major challenges in fabricating nanowires is to synthesize single-crystalline and high purity nanowires in large quantities without using metals or other catalysts in the process. Along this line, there have been many attempts to grow nanowires without using catalysts; nevertheless, most have only been published in academic

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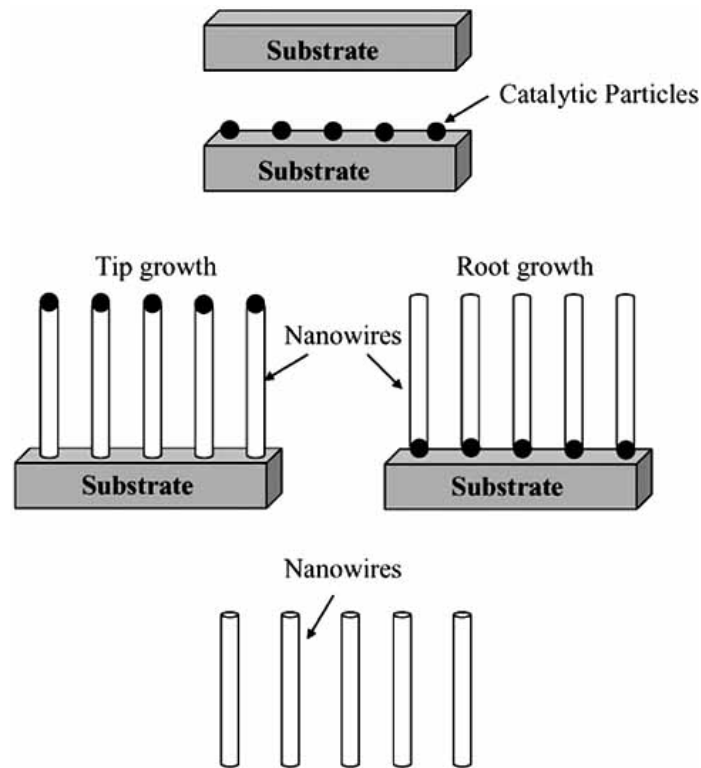


Fig. (1). Schematic illustration of a typical nanowire synthesis process using a catalyst driven VLS process [14].

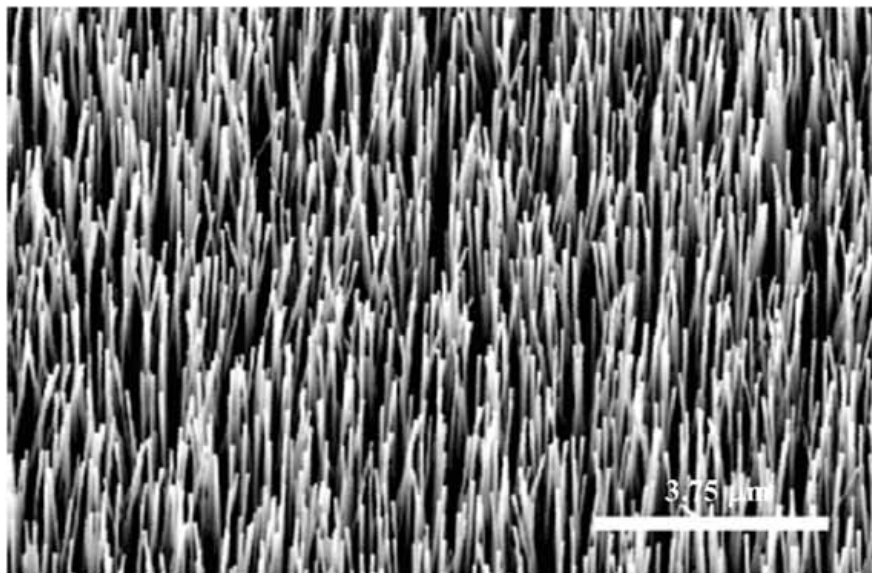


Fig. (2). SEM image of the ZnO nanowires grown by the VLS approach using Au catalysts [15].

journals. Only one patent was dedicated to synthesize a particularly large quantity of silicon nanowires and nanoparticle-chains in a large area from silicon monoxide or suboxide on any kind of substrates without using catalysts [17]. The method consists of vapor phase activation of silicon monoxide or suboxide by plasma or magnetron sputtering, chemical reaction of the precursor vapors, and material deposition to form silicon nanowires and nanoparticle-chains as schematically shown in Fig. 3. Figure 4 shows a transmission electron microscopy (TEM) image of

Si nanowires and nanoparticle-chains synthesized by the method. The diameter and length of the Si nanowires can be controlled by varying synthesis temperature and working pressure.

On the other hand, rod-like semiconductor crystals could be formed by colloidal methods patented by Alivisatos *et al.* [18]. By using mixtures of surfactants that bind to different crystallographic faces, the shape of growing crystals could be controlled. The surface energies of the growing crystal

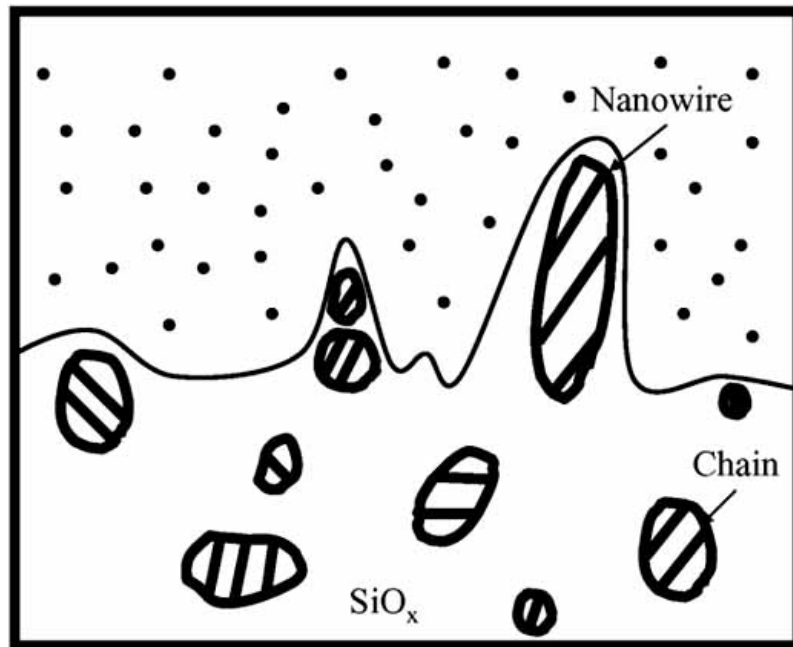


Fig. (3). Schematic diagram of the nucleation and growth of silicon nanowires and nanoparticle-chains from silicon monoxide or suboxide vapor without using catalysts [17].

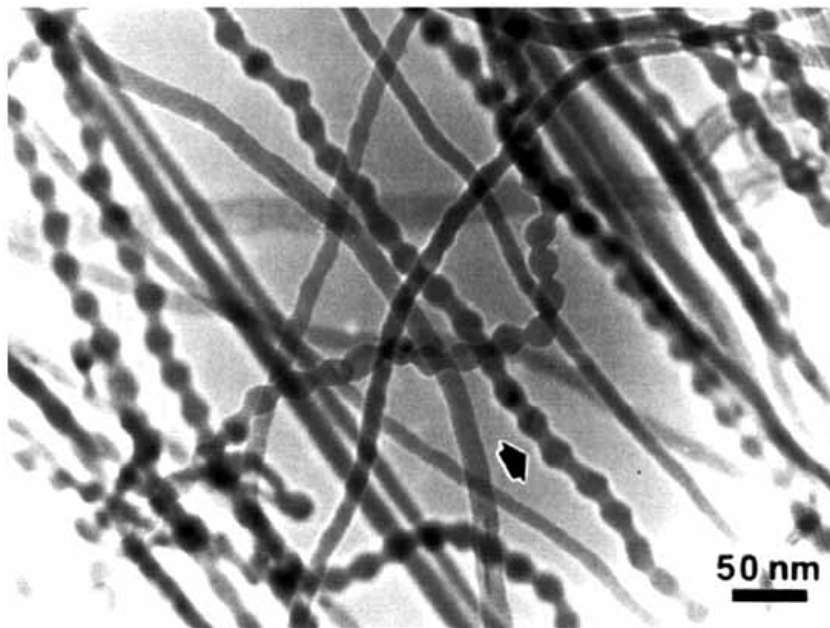


Fig. (4). Bright-field TEM image of silicon nanowires and nanoparticle-chains [17].

faces could be changed by adjusting the ratio of organic surfactants to control the growth directions of nanocrystals. For example, rod-like and spherical semiconductor nanocrystals, such as Group II-VI nanocrystals are formed by dissolving the binary mixture of non-reactive organic surfactants in a solvent at suitable temperature [18]. The particular Group II and Group VI precursors may be dissolved in any organic liquid, such as trialkyl phosphine, compatible with the binary mixture of surfactants. Increasing the percentage of the phosphorus-containing liquid surfactants in the binary mixture can promote the growth of

spherically shaped semiconductor nanocrystals. In contrast, increasing the percentage of the other surfactant which comprises an organic-substituted acid, or acid salt surfactant containing phosphorus in the binary mixture can promote the growth of rod-like shaped semiconductor nanocrystals. Figure 5a-5b show TEM images of CdSe nanoparticles and rod-like nanocrystals synthesized by this method [19].

Nanowires can also be fabricated using electrodeposition [20], where metal nanowires are grown on a template containing nanometer-sized cylindrical pores in a suitable insulating medium, such as polycarbonate, or mica. Chien *et*

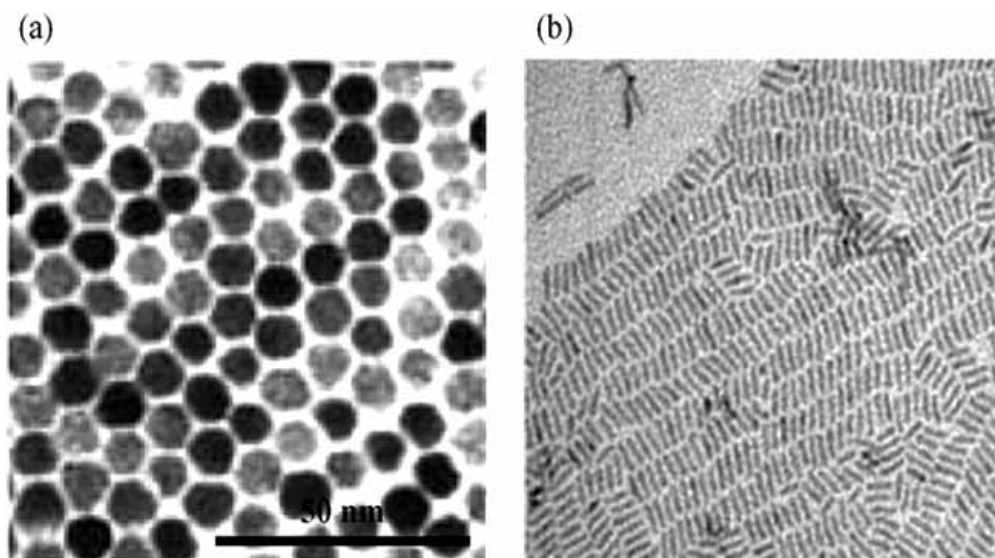


Fig. (5). Bright-field TEM image of CdSe (a) nanocrystals and (b) rod-like structures synthesized by the colloidal method [19].

al. patented an invention [20] on the fabrication of bismuth nanowires by using this method. The method starts with the preparation of an insulating membrane as a template, with one side of the membrane coated with a metal layer as an electrode. After inserting the coated membrane into a suitable deposition solution containing a bismuth-based electrode as the second electrode, bismuth nanowires can be obtained by applying a voltage. The diameter of the wires can be controlled from tens of nanometers to microns depending on the pore size, and the area density can be varied from 10^4 / mm^2 to 10^7 / mm^2 . The method could possibly be extended for the fabrication of other metallic nanowires.

Besides the above-mentioned electrodeposition method, another patent related to the template growth is the employment of asymmetric lattice-mismatch between two crystalline materials [21]. This strategy was proposed for the intentionally epitaxial growth of nanowires by choosing an overlayer material that has a close lattice-match (<4 %) to the substrate along one major crystallographic axis but has a great lattice mismatch (4~ 10%) along the perpendicular axis. This allows the unrestricted growth of the epitaxial crystal in the first direction but limits the width in the other, leading to the growth of self-assembled epitaxial nanowires on a crystalline substrate. Additionally, the nanowires can be aligned on the surface due to preferential growth on the regular steps of a clean surface. Finally, self-assembled nanowires are only formed with an appropriate deposition rate and deposition temperature. The nanowire materials that can be grown by this method on a Si substrate are represented as MSi_2 , where M is a metal chosen from the group consisting of Y, Sc, and rare earths including Dy, Th, Er, Ho, Tb, Gd, and Sm.

The discovery of carbon nanotubes possessing unique structures and properties has led to many attempts to develop various tubular inorganic [22] and organic [23-25] nanotubes. Nevertheless, the direct growth of non-carbon nanotubes still remains difficult. Recently, an invention [26]

patented by Hong *et al.* has provided a method for synthesizing calyx[4]hydroquinone(CHQ) organic nanotubes with excellent electrochemical and photochemical properties. This method starts by dissolving CHQ in an aqueous acetone solution, and allowing acetone to evaporate off the resulting solution at a temperature ranging from 0 to 20°C for CHQ to self-assemble into nanotubes. The resulting nanotubes are characterized by high aspect ratio, which can be varied by changing the growth temperature, and the degree of crystallization depends on the rate of the acetone evaporation. These nanotubes also serve as ideal templates for the growth of single-crystalline nanowires.

METHODS FOR IMPROVING QUALITY AND UNIFORMITY OF NANOWIRES

The methods mentioned in the previous section for fabricating nanowires or nanotubes with/without catalysts employ different growth mechanisms. Nonetheless, no method is perfect and the resulting nanowires still suffer from many artefacts including imperfections in the characteristics of individual nanowires or non-uniformity among them. In this section, the patents which address methods aiming at resolving these problems are reviewed.

Anodic alumina and mesoporous materials are frequently employed as templates for fabricating nanowires, where arrays of pores inside those materials are filled by an appropriate metal in a liquid state [27]. However, the major problem with this approach is that it is relatively difficult to acquire densely continuous nanowires. Although, the problem could be partially overcome by material injection at high pressure and high temperature [28], the crystalline quality of nanowires is not good enough. Therefore, an invention patented by Ying, *et al.* was proposed [29] to improve the control over the crystalline quality, size, spacing and length of mesoporous template-grown metal nanowires by pre-treating the pores. The pore wall property can be modified to reduce the contact angle for complete filling by applying an acid solution, such as H_2SO_4 . The thickness of the pore wall and composition of the pore surface can be

varied and modified by vapor depositing a desired species. Thus, an array of nanowires with a relatively small diameter, close spacing, and long length can be made and the crystalline quality of nanowires could be improved effectively. By using this method, they have obtained ultra-fine Bi wires of good crystallinity confirmed by x-ray diffraction.

Another drawback that was met with template growth is that the maximum nanowire length is limited by the thickness of the porous host film. The method patented by Penner *et al.* [30], called the step edge decoration method, is claimed to have the capability to synthesize metallic nanowires which are long (greater than $10\mu\text{m}$ in length), uniform in diameter, and removable from the surface on which they are synthesized. The method starts with selective electrodeposition of metal oxide from an aqueous solution as a precursor, along step edges present on the stepped surface, such as graphite, to form a bead of metal oxide nuclei and, with continued deposition, rapidly grow into hemicylindrical nanowires. Subsequently, metal nanowires are obtained from the transformation from metal oxide

nanowires by a reduction reaction in H_2 at 500°C . Afterwards, polystyrene is cast into the gaps between nanowires to form a nanowire/polystyrene film. Finally, the cast nanowire/polystyrene film is peeled off the graphite surface with the metal nanowires embedded therein. The entire procedure is sketched in Fig. 6. The metal nanowires can be also prepared by direct electrodeposition of metal from an aqueous solution at a low current and low deposition rate. Fig. 7 shows a SEM image of MoO_2 nanowires which were formed for the deposition of 256 seconds using the method [30].

One of the serious problems often encountered for nanowires, especially those grown by the VLS method, is non-uniform length in the product of the same batch from the same growth conditions. Metz *et al.* have patented an invention [31] to grow germanium and silicon nanowire segments with uniform length using catalytic particles, such as gold particles. Multiple nanowire segments are produced by individual nanoparticles as schematically shown in Fig. 8. Alternating segments of nanowires are made of a sacrificial material, such as Ge, and another material of interest, such as

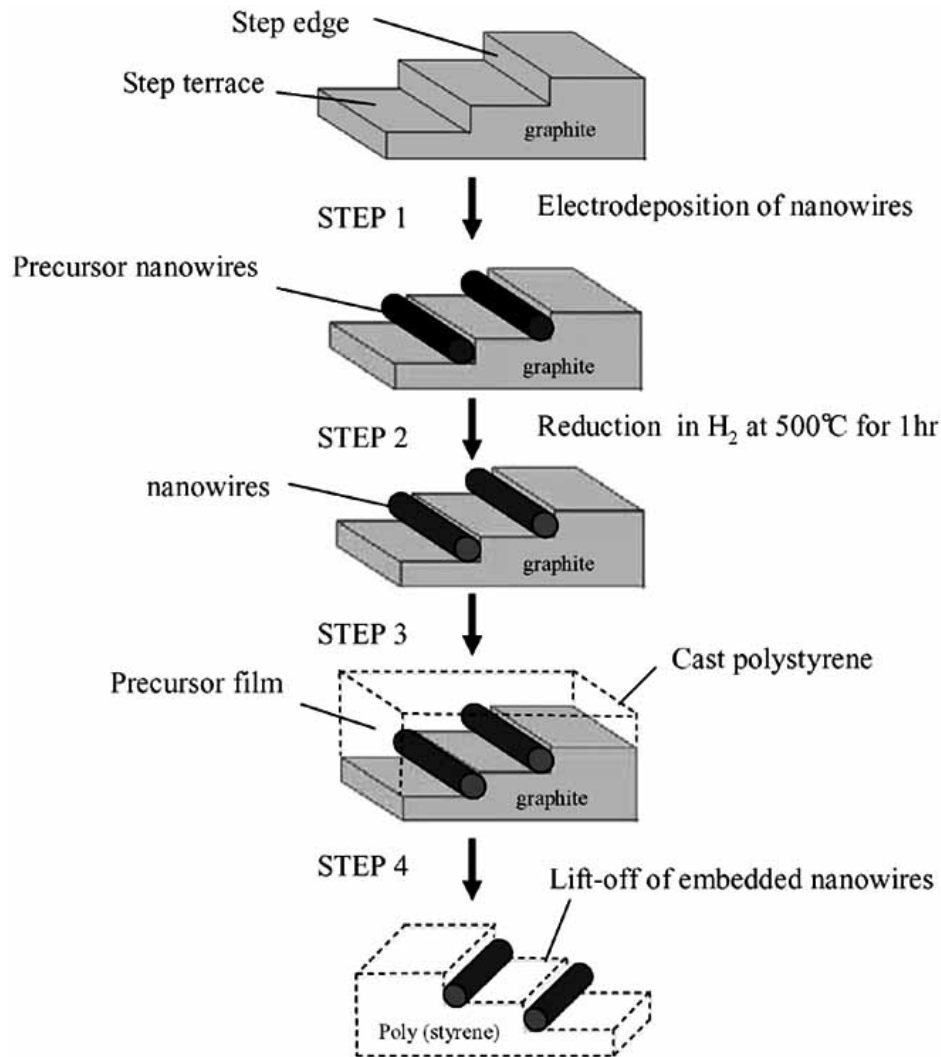


Fig. (6). Schematic diagram of the invention for preparing long metallic nanowires by metal electrodeposition on the step edges of a stepped surface, such as graphite [30].

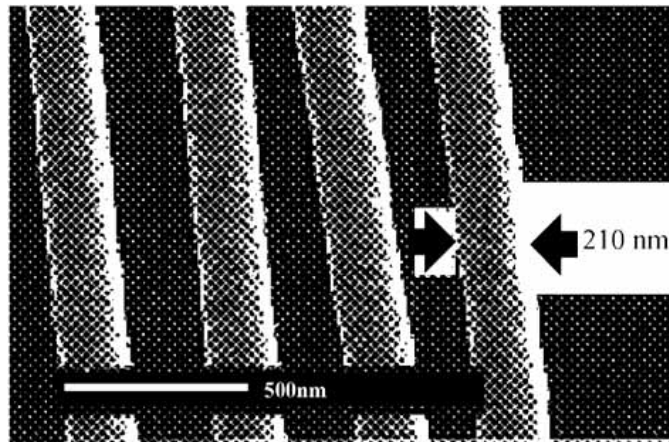
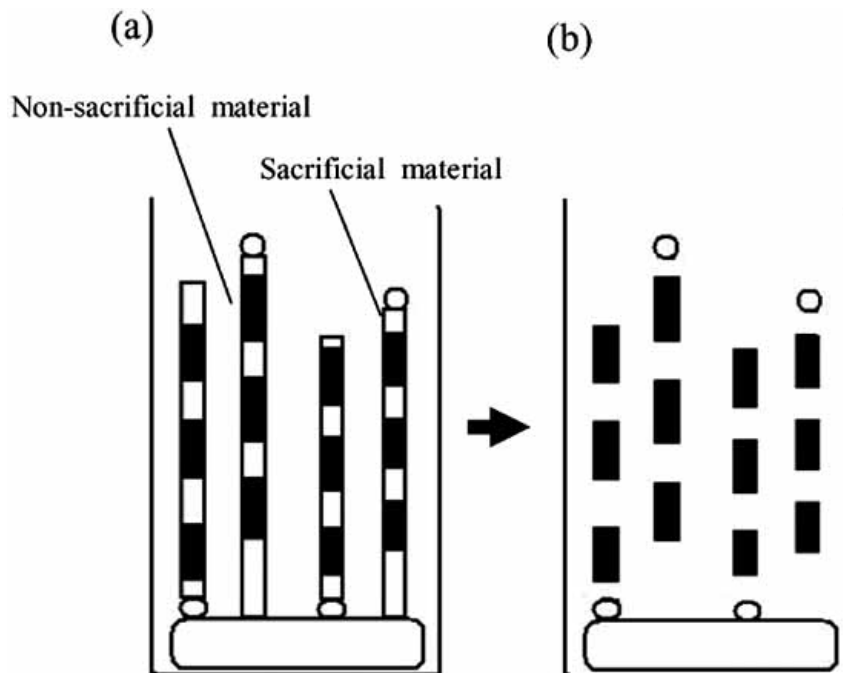


Fig. (7). SEM image of electrodeposited MoO₂ nanowires on the step edges of a stepped surface [30].



Figs. (8a-8b). Schematic diagrams of the method for fabricating nanowires with uniform length by dissolution of sacrificial segments [31].

Si. As shown in Fig. 8, the segment length of the second material can be better controlled than the first material. The multiple nanowire segments can be separated by utilizing the property of different solubility in solution between the first and second material. Subsequently, the nanowire segments are immersed into a water solution or oxidizing agents to selectively dissolve the sacrificial segments. Therefore, the desired number of nanowire segments is obtained.

METHODS FOR FABRICATING ALIGNED NANOWIRES OR ALIGNING DISPERSED NANOWIRES

Nanowires formed by various mechanisms are often randomly oriented [32-34]. Although, vertical alignment of as-grown nanowires has been demonstrated [35-37], only limited material systems can be applied. Practical devices require to have wires parallel to each other and possibly parallel or perpendicular to the substrate surface. The common way of fabricating aligned wires by photoli-

thography fails to create very fine and closely spaced arrays of wires. Therefore, the strategies in some recent patents for alignment of nanowires are reviewed in this section.

The first invention, patented by Parel *et al.* [38], employs an etched superlattice to fabricate aligned nanowires as shown in Fig. 9. The superlattice material comprises of two alternating layers of conductive and non-conductive materials. The conductive layers are then etched away preferentially with a higher etching rate to produce an extruded non-conductive layer with furrows for the subsequent growth of semiconductor, ceramic, organic, polymeric and other types of nanowires. This etching causes the working surface to be rippled. The furrows can be helpful in fabricating nanowires with the size determined by the depth of the furrows. During wire growth by electrodeposition, the rippled working surface is regarded as a low-adhesion layer and the non-etched side works as another conductive electrode. The ions

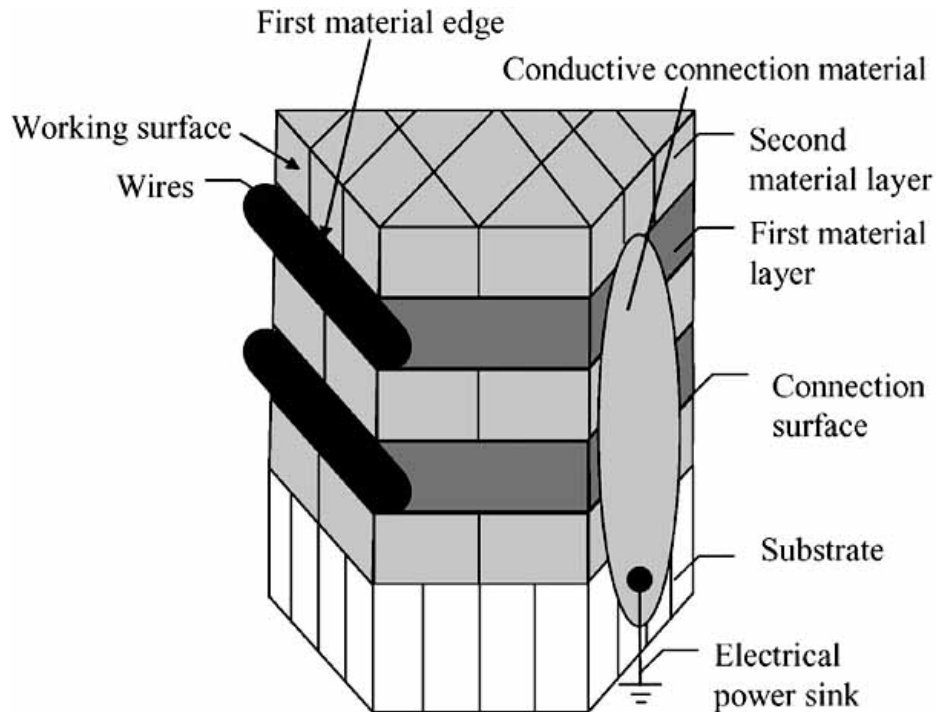


Fig. (9). Schematic diagram of the method for fabricating aligned nanowires by using superlattice material comprised of two alternating layers of conductive and non-conductive layers as a template [38].

are attracted to the conductive edges, which have different electrical potentials, to build an array of nanowires at the working surface as shown in Fig. 9. If the superlattice with the wires was brought into physical contact with a substrate of high adhesion on the surface, the nanowires could be transferred to the substrate easily and the alignment of nanowires on the substrate is achieved, which is schematically illustrated in Fig. 10.

An array of nanowires can also be directly formed and aligned on an alloy substrate as shown in an invention patented by Jin *et al.* [39], where the alloy substrate must be phase separated into catalytic and non-catalytic elements in regular patterns. The regular patterns in the alloy substrate

can be produced by spinodal decomposition or nucleation-and-growth type transformation. Subsequently, CVD is used to grow aligned one-dimensional nanostructures such as carbon nanotubes, semiconductor wires comprising silicon or germanium, ceramic wires comprising at least one of boride, carbide, and nitride, metallic nanowires, and nanowires having a heterojunction from the recessed pore structures. If the substrate was sectioned with any angle to the original surface normal, the nanowires with a reduced diameter can be grown and aligned along any direction.

A less complicated method has been proposed to align randomly-oriented nanowires by energetic ion bombardment, which was patented by Kamins *et al.* [40]. As shown in Fig.

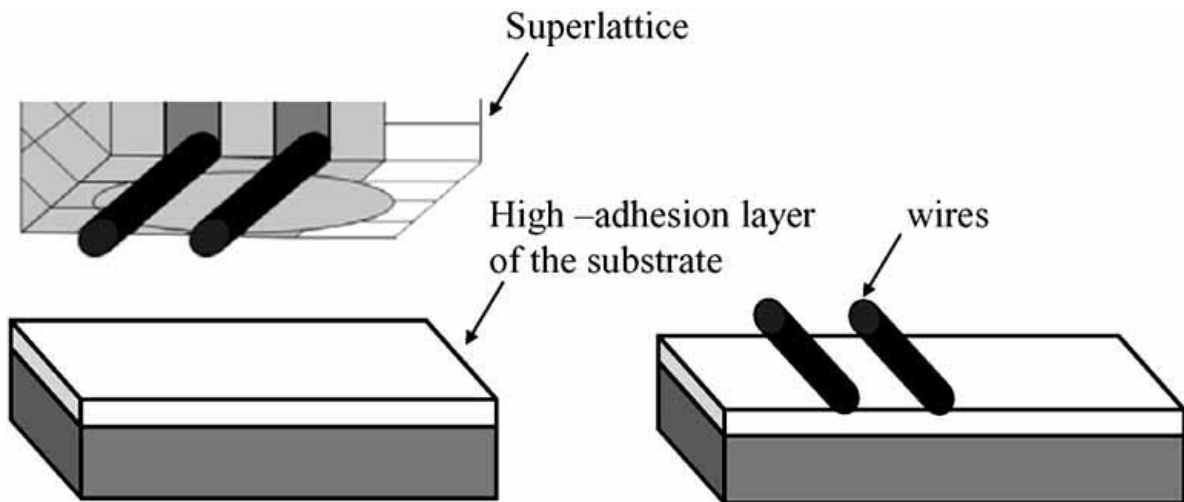
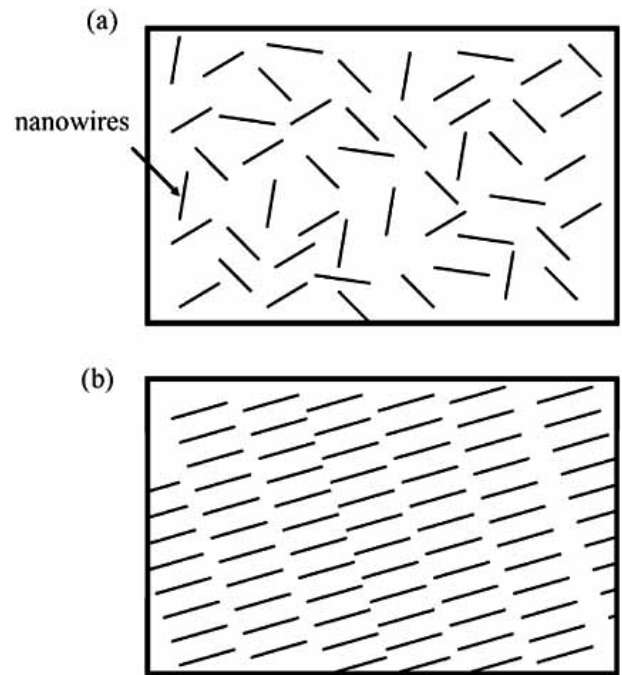


Fig. (10). Schematic diagram of the method for transferring nanowires to a substrate to form aligned nanowires [38].

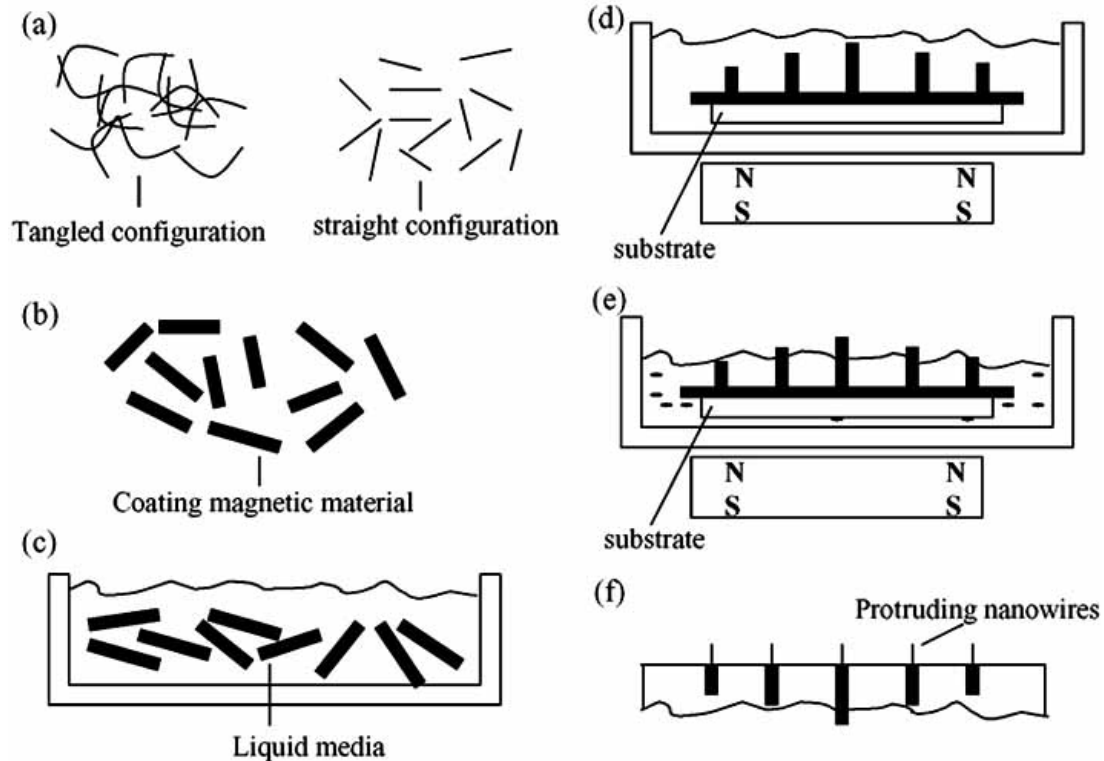
11, most as-grown nanowires; for example, Si nanowires grown by titanium catalysts, are randomly oriented and can be easily dispersed on a substrate randomly. When the Si nanowires are exposed to a flux of energetic ions, such as argon ions with an ion energy of 5 KeV and a typical ion current of 1 μA , the nanowires become parallel to each other and parallel to the surface of the substrate. However, how much damage exerted by the ion bombardment to the nanowires requires more studies.

The last patent, by Filas *et al.* [41], deals with the alignment of nanowires magnetically and particularly for field emitters as schematically shown in Fig. 12. The first step is to produce nanowires such as carbon nanotubes or semiconductor nanowires by any well known growth method as shown in Fig. 12(a). The nanowires are then coated with a magnetic material as shown in Fig. 12(b). Strongly magnetic materials such as ferromagnetic or ferrimagnetic materials are favorable owing to their high saturation magnetization. The next step, shown in Fig. 12(c), is to distribute the magnetic-coated nanowires in a low-adhesive liquid medium by mixing and vigorous stirring. The next step, as shown in Fig. 12(d), is to magnetically align the coated nanowires. In order to reduce magnetostatic energy, the magnetic bodies in a liquid medium are aligned along the direction of applied magnetic field and are likely to have array structures with a specific spacing as a result of repulsion induced by neighboring magnetic dipoles. The liquid medium is then evaporated while maintaining the magnetic field, as shown in Fig. 12 (e). The final step, illustrated in Fig. 12(f), is to etch the matrix from the back side by acid, ion milling, laser beam, evaporation, or other techniques. The etching step



Figs. (11a,11b). Schematic diagram of the method for aligning nanowires by a flux of energetic ions [40].

provides the protruding nanowires with relatively equal heights. It is optional to remove the magnetic coating from the protruding portions of the nanowires to expose the original nanowires, as shown in Fig. 12(f).



Figs. (12a-12f). Schematic diagram of the method for fabricating a field emitter structure with aligned nanowires magnetically [41].

CURRENT & FUTURE DEVELOPMENTS

With high aspect ratio and unique electronic and optical properties, nanowires have a large potential for applications in optical-electronic nanodevices, including flat panel field emission displays, microwave amplifier driving circuits for active matrix liquid crystal displays [42], low energy consumption of nanowires light emitting diode displays [14], and field emission transistors [43]. Besides, nanowires also have an ideal shape to act as the probes of atomic force microscopes or scanning tunneling microscopes [44]. Therefore, to fit the demand of practical applications, more techniques for controlling the growth and qualities of nanowires efficiently thus become very important.

According to the above review, it is obvious that current patents issued concentrate mostly on the fabrication of nanowires and methods to improve the qualities of nanowires, making significant progress. However, it is still far from completion. There is no method efficient enough to be able to control a combination of characteristics of the as-grown nanowires. It is definitely desired to continuously improve the capability with which many nanowire parameters can be well controlled at the same time, especially in a large area.

In addition to improving the crystal quality, uniformity, aspect ratio and alignment of nanowires, fabrication of nanowires with tunable properties by doping is fundamentally important for practical applications. Although the methods for synthesizing doped nanowires are few, primarily reported in some academic journals, they have attracted a lot of attention lately. Doped nanowires [45,46] have shown tunable optoelectronic properties due to varied energy bands and carrier concentrations. Nanowires doped with ferromagnetic elements are also promising for dilute magnetic semiconductor-based nanodevices [47,48]. On the other hand, nanowires with heterostructures in the radial [49-51] or axial [52-54] direction, which may possess confinement effects, are in high demand for nanodevice applications. So far, only a few special examples have been reported [49-54] and more universal concepts and working examples are deficient. How to fabricate and control the heterostructures of nanowires effectively still remains a challenge. There is no doubt that fabrication of nanowires with tunable composition and heterostructures will be the new focus of research now and in the near future.

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