

# Chapter 1

## Emergence of Nanowires

**Abstract** The design, development and understanding of synthetic materials, with at least one dimension below 100 nm, have been driving a broad range of research in the scientific community for a number of years given the potential of such materials to substantially impact many areas of science and technology. In particular, one-dimensional nanowires, with diameters reaching to the molecular or quantum regime, have been a focus of research over the past two decades. The underlying principles for synthesis of one-dimensional materials have been investigated in different contexts for almost half a century ago, although significant challenges existed in developing the critical understanding to control (i) diameters to the deep nanoscale dimensions as well as (ii) structure and composition in the axial and radial coordinates as necessary for the synthesis of materials with designed and tunable functionality. In this chapter, the emergence of the nanowire research platform is introduced, including the concept and importance, synthetic challenges and initial design, and the development of vapor-liquid-solid crystal growth mechanism. In addition, other nanofabrication based approaches explored in the early years of this field will be briefly introduced.

### 1.1 Introduction: Motivation for Nanowires

“Nano” has become a nearly ubiquitous prefix used today in science and technology, and also one widely recognized by the general public. But what is the science that can build nanotechnologies and may make nanotechnology a unique field of endeavor that revolutionizes many other areas? Indeed, nanometer scale structures represent an exciting, intellectually-challenging and rapidly expanding area of research that crosses the borders between many areas of the physical sciences and engineering [1, 2]. Nanostructures can be defined as systems in which at least one dimension is less than 100 nm. That is, reducing 1, 2 or 3 dimensions (D) of a bulk material to the nanometer scale produces nanometer thick 2D layers, 1D nanowires (NWs), or 0D nanoclusters, respectively. Interest in nanometer scale structures has been driven by fascinating questions and the potential to impact basic

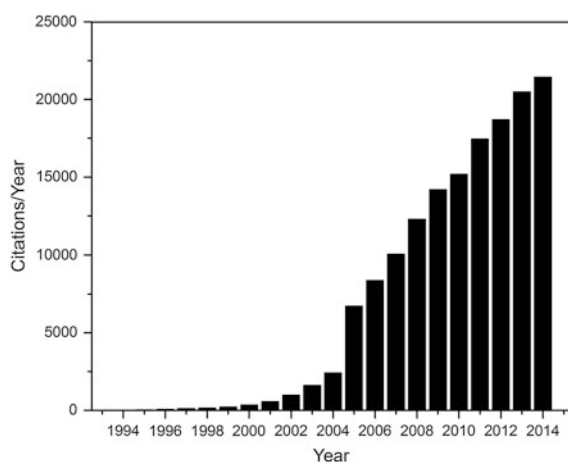
science and technology. Three questions at the heart of basic chemistry and physics research of nanomaterials are: (1) how can nanostructures that have controlled dimensionality be synthesized or fabricated; (2) what are the intrinsic and potentially unique physical properties of these synthesized nanostructures; and (3) how can nanostructures with tailorable properties contribute to and even create breakthroughs in fields, including but not limited to, electronics, photonics, energy conversion and storage, and the life sciences? Before delving in detail to these questions in subsequent chapters of the book, an abbreviated history of the development of the unique 1D nanostructure now known as NWs is reviewed.

### ***1.1.1 Importance of One-Dimensional Materials***

Dimensionality plays a critical role in determining the properties of materials due to, for example, the different ways that electrons interact in 3D, 2D and 1D structures [3–5]. The study of dimensionality has a long history in chemistry and physics, although this has been primarily with the prefix “quasi” added to the description of materials. That is, quasi-1D solids, including square-planar platinum chain and metal trichalcogenide compounds [6, 7], and quasi-2D layered solids, such as metal dichalcogenides and copper oxide superconductors [3, 5, 8, 9]. The anisotropy inherent in quasi-1D and -2D systems is central to the unique properties and phases that these materials exhibit, although the small but finite interactions between 1D chains or 2D layers in bulk materials have made it difficult to address the interesting properties expected for the pure low-dimensional systems [10, 11].

The focus on nanometer scale wires is motivated by basic scientific and technology questions. How can atoms or other building blocks be rationally assembled into structures with nanometer size diameters but much longer lengths? By the end of the 20th century, there had been relatively well developed methods for the synthesis of 0D nanoclusters via arrested precipitation [12] and the growth of 2D layers using molecular beam epitaxy (MBE), although general methods for the growth of 1D structures with diameters less than 10 nm had not been available. In addition, 1D structures with nanometer diameters, such as NWs, have great potential for testing and understanding fundamental concepts about the roles of dimensionality and size on physical properties. For instance, 1D systems should exhibit density of states singularities, can have energetically discrete molecular states extending over large distances, and may show more exotic phenomena, such as spin-charge separation predicted for a Luttinger liquid [13]. There are also many applications where 1D NWs can be exploited, including (i) functional nanostructure materials, (ii) novel probe microscopy tips, (iii) nanoelectronics, (iv) nanophotonics, (v) quantum devices, (vi) energy storage, (vii) energy conversion, (viii) chemical and biological sensing, and (ix) nano-bio interfaces. To realize these and other exciting uses of 1D nanostructures, however, requires an understanding of the fundamental chemistry and physics questions raised above [11, 14].

Semiconductor NWs represent arguable the most important and versatile nanometer-scale wire structures. In contrast to another classes of 1D nanostructures, such as carbon nanotubes (CNTs) [10, 11], semiconductor NWs can be rationally and predictably synthesized in single crystal forms with all key parameters controlled, including chemical composition, diameter and length, and doping and electronic properties. Thus, semiconductor NWs represent one of the best-defined classes of nanoscale building blocks, and this precise control over key variables has correspondingly enabled a wide range of devices and assembly/integration strategies to be pursued [15]. For example, early work showed that semiconductor NWs could be assembled into functional electronic devices, including crossed NW *p-n* diodes, crossed NW-FETs, nanoscale logic gates and computation circuits, as well as optoelectronic devices such as nanoscale light-emitting diodes (LEDs) and lasers. In addition, it is possible to combine distinct NW building blocks in ways not possible in conventional electronics, and to leverage the knowledge base that exists for the chemical modification of inorganic surfaces to produce semiconductor NW devices that achieve new function and correspondingly lead to unexpected device and system concepts [14, 16, 17]. In the past 20 years, the research community has witnessed a substantial increase in development of NW research with a near exponential increase in publications, which reached over 20,000 new publications per year in 2014 (Fig. 1.1). Nonetheless, significant challenges existed at the very early days when people started to explore the methodologies for 1D NW synthesis.



**Fig. 1.1** Histogram of citations by year to “nanowire(s)” (Search performed on 7th July 2015 with Web of Science)

### 1.1.2 Synthetic Challenges and Initial Design

How can atoms or other building blocks be rationally assembled into structures with nanometer size diameters but much longer lengths? Answering this question is central to the preparation of 1D nanostructures, and correspondingly, research had pursued several strategies to synthesize CNTs and NWs. For example, multiple-walled CNTs had been prepared via the condensation of hot carbon plasmas. The growth mechanism producing these 1D nanostructures is, however, specific to the tubular structures of  $sp^2$ -bonded carbon and isostructural hexagonal boron nitride. It is also possible to favor the formation of single-walled CNTs from carbon plasmas by adding certain metals [18]. These metals appeared to function as catalysts, although a sufficiently good understanding of the growth mechanism needed to control the diameter and helicity of SWCNTs had not been available by the end of last century.

In general, the growth of 1D nanostructures requires two dimensions to be restricted to the nanometer regime, while the third dimension extends to a much greater length scale [10, 19]. This overall requirement is considerably more difficult to achieve than the corresponding constraints needed for successful growth of 0D and 2D structures [20]. For example, many important semiconductor materials adopt a cubic zinc blende structure, and thus when growth is stopped at an early stage, the resulting nanoscale structures are nanocrystals with various polyhedron shapes but not 1D NWs. To achieve 1D growth in systems, where atomic bonding is relatively isotropic, requires that the growth symmetry be broken rather than simply arresting growth at an early stage.

By the end of the last century, considerable efforts had been placed on the bulk synthesis of NWs, and various strategies had been developed to break the growth symmetry either “physically” or “chemically”. A common theme in many of these studies had been the use of linear templates, including the edges of surface steps [21], nanofibers [22, 23], and porous membranes [24], to direct chemical reactions and material growth in 1D. Although a promising and conceptually simple technique, template mediated growth generally produces polycrystalline materials with diameters greater than 10 nm. Hence, it was unclear based on these previous studies, whether a general method for the growth of single crystalline NWs with diameters less than 10 nm existed [10, 11].

In order to understand the intrinsic behavior of 1D structures in a size regime where quantum effects produce new phenomena, the first goal should be to develop a general and predictive method for preparing compositionally diverse single-crystal materials with nanometer to tens of nanometer diameters. From the outset it was believed that a catalytic approach, in which the catalyst is used to direct growth in a highly anisotropic or 1D manner, would enable one to meet this overall goal [11]. Depending on the phases involved in the reaction, this approach is typically defined as vapor-liquid-solid (VLS) [25, 26], solution-liquid-solid (SLS) [27] or vapor-solid (VS) [28, 29] growth, which will be introduced in more details later in this chapter and the following chapter.

### ***1.1.3 Top-Down and Bottom-Up Nanotechnology***

Nanotechnology aims to be a revolution, not an evolution, in science and technology. It distinguishes itself from all previous scientific and industrial revolutions in three major aspects [30]. For the first time in human history, one can (i) change the fundamental properties (such as band gaps and luminescence) of matter as well as make materials with desirable attributes; (ii) observe and manipulate nanoscale objects (such as atoms and molecules); and (iii) fabricate and build single-NW/nanotube single-electron/photon nanodevices. The first characteristic is due to the so-called quantum-size effects whereby the properties of a material change with its size in the nanometer regime. The second is made possible by the invention of high-resolution transmission electron microscopy (HRTEM) and scanning probe microscopy (SPM), including scanning tunneling microscopy (STM) and atomic force microscopy (AFM). The third is the result of the developments of various nanofabrication techniques (such as nanoimprint and electron beam lithography) as well as due to a physical phenomenon known as “quantum confinement” in the nanorealm. It is quite possible that the nano-revolution will ultimately impact most aspects of human activities, not just in science and technology.

How can this revolution be realized? There are, in principle, two approaches to nanotechnology: the bottom-up and the top-down strategies [31, 32]. The bottom-up strategy focuses on building or assembling nanodevices from atomic or molecular components. The top-down one seeks to fabricate nanodevices on silicon or other semiconductor chips directly using increasingly high resolution lithography [33]. It is widely held that present top-down semiconductor technology has not exploited the chemistry and physics (e.g., quantum-size effects) of nanomaterials, and also not taken advantage of the development of bottom-up nanotechnology [30, 32]. Many reasons come to mind. First, nanomaterials and their properties in the nanorealm, especially at the interface, are not well understood and/or still under investigation. Second, the fabrication techniques, both at the materials level and at the device stage, are not well-defined or fully developed. Third, connection to the macroscopic world remains a real problem. Finally, even if all these problems can be overcome, they are not at present mass producible and/or cost competitive enough to challenge the existing semiconductor technology. For a stand-alone bottom-up nanodevice to challenge the top-down nanodevice, it must overcome these hurdles.

Thus, while each approach has its advantages and disadvantages, it is generally believed that future development of nanotechnology will encompass both approaches with their relative contributions depending upon specific applications and taking into account pros and cons associated with factors such as performance enhancement and materials improvement. In other words, application-specific nanodevices can be fabricated using a combination of the two approaches. For example, nanodevices can be made via the bottom-up strategy and assembled in situ or “grown in place” on a nanochip fabricated via the top-down lithography

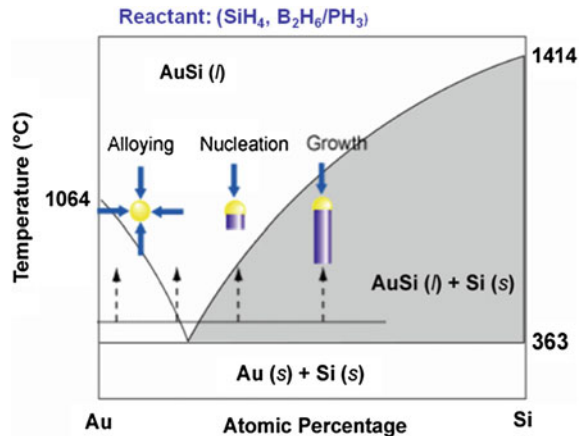
techniques, which enable straightforward interfacing to the outside world. Such hybrid nanotechnologies would allow the exploitation of advantageous attributes of both approaches. In this book, we will focus on the developments of the bottom-up synthesis strategy and NW structures and their characteristics that arise from this bottom-up approach.

## 1.2 Micron-Scale Whiskers: VLS Concept

### 1.2.1 Concept and Key Results

In general, the preparation of wires requires that the addition of material during the growth process be constrained to occur along only one direction. This is conveniently accomplished by taking advantage of ideas from vapor–liquid–solid (VLS) growth [25, 26, 34, 35] (Fig. 1.2). In this mechanism, a metal catalyst, such as a gold (Au) nanoparticle, forms a liquid metal–semiconductor eutectic alloy at an elevated temperature by adsorbing the vapor reactant, such as silicon resulting from silane ( $\text{SiH}_4$ ) decomposition. Continuous incorporation of the semiconductor material in the alloy through the vapor/liquid interface ultimately results in supersaturation of the semiconductor material, and this supersaturated state leads to precipitation of the semiconductor material and formation of a liquid–solid interface to achieve minimum free energy. Accordingly, the 1D crystal growth begins via the transfer of the semiconductor material from the vapor reactant at the vapor/liquid interface into the eutectic, followed by atom (e.g., Si atoms) addition at the liquid/solid interface. In addition, because the gold nanoparticle remains at the tip of the NW during VLS growth, it can define the diameter of the 1D NW as long as all reactant addition is through the liquid/solid interface. An important feature of this concept is that it readily provides the intellectual underpinning needed for the

**Fig. 1.2** VLS growth mechanism of Si wires



prediction of good catalysts and synthesis conditions. First, one uses equilibrium phase diagrams to choose a catalyst that can form a liquid alloy with the NW material of interest. The phase diagram is then used to choose a specific composition (catalyst: NW material) and synthesis temperature so that there is a coexistence of liquid alloy and solid NW material. Second, the liquid catalyst alloy cluster serves as a preferential site for absorption of reactant (i.e., there is a much higher sticking probability on liquid vs. solid surfaces) and, when supersaturated, the nucleation site for crystallization. Preferential 1D growth occurs in the presence of reactant as long as the catalyst remains liquid [11, 36].

The first successful synthesis of Si wires, or filamentary Si crystals with macroscopic dimensions, was reported in 1957 for the synthesis of Si whisker with  $\langle 111 \rangle$  orientation [37]. In 1961, Greiner et al. [38] reported that silicon nanoribbons were made by reacting silicon pellets with iodine and hydrogen in a closed tube in a temperature gradient. Arsenic-doped silicon pellets with resistivity from 0.001 to 0.005  $\Omega\text{-cm}$  produced ribbons, in which the silicon source of 0.002  $\Omega\text{-cm}$  resistivity gave optimum yields. The arsenic content of this silicon source material was estimated to be  $4 \times 10^{19}$  atoms/cm<sup>3</sup>. Several years later, the illuminating work performed by Wagner and Ellis [25, 26] established the mechanism of the Si whisker growth, well recognized as the VLS mechanism, which until today still represents the most important bottom-up approach to synthesize silicon nanowires (SiNWs) [39, 40]. In these and subsequent studies, Si whiskers were grown by the disproportionation of  $\text{SiI}_2$  or by hydrogen reduction of  $\text{SiCl}_4$ . Three important facts emerged: (a) silicon whiskers do not contain an axial screw dislocation; (b) an impurity, such as Au, is essential for whisker growth; and (c) a small globule is present at the tip of the whisker during growth. In this VLS mechanism, the role of the impurity is to form a liquid alloy droplet of relatively low melting temperature. Thus, the liquid droplet is a preferred site for deposition from the vapor, which causes the liquid to become supersaturated with Si.

Typical of this early work, a small particle of Au was placed on a  $\{111\}$  surface of a Si wafer and heated to 950  $^\circ\text{C}$ , forming a small droplet of Au-Si alloy [25, 26]. A mixture of hydrogen and  $\text{SiCl}_4$  was introduced. The liquid alloy acts as a preferred sink for arriving Si atoms, that is, as a catalyst for the chemical process involved. The Si enters the liquid and freezes out, with a very small concentration of Au in solid solution, at the interface between solid Si and the liquid alloy. Continuation of this process results in the displacement of the alloy droplet from the substrate crystal atop the growing whisker. The growth direction is  $\langle 111 \rangle$ , and the side facets of the whisker are usually  $\{211\}$  and sometimes  $\{110\}$ . The whisker grows in length by this mechanism until the Au is consumed or until the growth conditions are changed. Similar results were obtained with Pt, Ag, Pd, Cu or Ni, either by replacing a particle on the Si substrate or co-deposition [25, 26]. VLS growth of twinned Si ribbons having a  $\langle 211 \rangle$  or a  $\langle 110 \rangle$  growth direction and  $\{111\}$  main faces were also observed [25, 26].

## 1.2.2 Limitations

The primary products identified in these early studies were micrometer to millimeter diameter Si whiskers or wires [25, 26]. Within the framework of the VLS mechanism, one can readily imagine synthesizing NWs of many different materials and diameters, if catalyst clusters of nanometer dimensions are available. This raises an important point, because equilibrium thermodynamics can be used to define the minimum radius,  $r_{\min}$ , of a liquid metal cluster as:

$$r_{\min} = 2\sigma_{LV}V_L/RT \ln \sigma$$

where  $\sigma_{LV}$  is the liquid-vapor surface free energy,  $V_L$  is the molar volume,  $R$  is the ideal gas constant,  $T$  is the absolute temperature, and  $\sigma$  is the vapor phase supersaturation [11]. Substituting typical values into the above equation yields a minimum radius on the order of 0.2  $\mu\text{m}$ , and thus under equilibrium conditions, one does not expect to be able to grow NWs with sufficiently small diameters to exhibit interesting new properties. Indeed, previous studies using a growth process similar to that described above, termed VLS growth, had only yielded micrometer diameter whiskers [25]. The large micrometer diameter clusters at the ends of these whiskers suggested strongly that the constraint imposed by the above equation led to the lower diameter limit in these previous studies. This constraint from equilibrium thermodynamics were overcome rationally by employing laser ablation and condensation, which has been studied extensively in the past [41], to generate nanometer diameter clusters. We will go into details of this breakthrough in Chap. 2.

## 1.3 Other Early Works

### 1.3.1 Top-Down Lithography-Based Si Nanopillars

In 1993, Chou and coworkers [42] reported the fabrication and preliminary photoluminescence study of free-standing Si pillars with diameters as small as about 10 nm and aspect ratio greater than 15. The pillars were fabricated using electron-beam lithography (EBL), chlorine-based reactive ion etching (RIE), and subsequent hydrofluoride (HF) wet etching. Photoluminescence with a peak at 720 nm was repeatedly observed from an array of nanoscale pillar with  $\sim 20$  nm diameters, although the origin of such photoluminescence was not clear. They further presented the fabrication of sub-50 nm Si pillars, ridges, and trenches using ultrahigh resolution EBL and chlorine based RIE [43].

On the other hand, Chen and Ahmed [44] reported the fabrication of high aspect ratio, sub-10 nm size structures in silicon without any wet chemical etching. A 50-nm thick double layer of low and high molecular weight polymethylmethacrylate



(PMMA) resist was exposed to an 80 kV electron beam with a beam diameter smaller than 5 nm. After exposure, the resist was developed and a 5-nm-thick AuPd film was deposited and sub-10 nm AuPd dots were obtained by liftoff. These dots served as a RIE etch mask on the Si substrate, yielding silicon nanocolumns with 5–7 nm diameters about 7:1 aspect ratios. Similarly, about 15 nm silicon nanopillars were fabricated using gold colloidal nanoparticles as the etch mask [45].

### 1.3.2 Carbide Nanorods

In 1995, the Lieber group [22] reported an alternative approach to the synthesis of nanoscale structures based on nanotubes, in which the carbon nanotubes were used as templates and converted to carbide rods by reacting with volatile oxide and/or halide species. In this way they were able to prepare solid carbide nanoscale rods of TiC, NbC, Fe<sub>3</sub>C, SiC and BC<sub>x</sub> in high yield with typical diameters of between 2 and 30 nm and lengths of up to 20  $\mu\text{m}$ . Preliminary studies showed that these rods shared the properties of the bulk materials, such as magnetism and superconductivity, suggesting that they might allow the investigation of the effects of confinement and reduced dimensionality on such solid-state properties. This was especially important as it articulated the key importance of controlling composition—not being limited to simply Si or C—to enable unique functional properties.

Systematic temperature-dependent growth studies of TiC nanorods produced from spatially separated Ti metal and carbon nanotubes in the presence of iodine showed that reaction proceeds initially via the formation of a thin, uniform carbide coating and that further reaction proceeds via inward growth of this coating with a concomitant consumption of the carbon nanotube until a solid nanorod is formed [46]. The coatings and nanorods were polycrystalline, cubic TiC. Similar results were also obtained in growth studies of NbC nanorods from Nb metal and carbon nanotubes. These data showed that the growth of TiC and NbC nanorods involves a template mechanism in which the carbon nanotubes define the overall morphology and furthermore demonstrated that new TiC and NbC nanotubes could be prepared by controlling the growth conditions.

### 1.3.3 Nanowiskers by Vapor Phase Epitaxy

In 1992, Yazawa et al. [47] showed that selective heteroepitaxy of nanometer-scale InAs whiskers on SiO<sub>2</sub>-patterned GaAs substrates was induced by surface contamination with Au resulting from the fluorocarbon plasma etching process used to etch the SiO<sub>2</sub> mask. It was demonstrated that high-density ( $\sim 10^{10}/\text{cm}^2$ ) InAs nanowiskers with 20–30 nm in diameter could be epitaxially grown on InAs (111)B substrates onto which 1 monolayer of Au atoms had been deposited. This pillar-like

growth yielded primarily tapered or cone-like structure and appeared to be induced by ultrafine alloy droplets generated by the reaction between Au-nanoclusters and InAs substrates. Later, these researchers [48, 49] demonstrated the growth of GaAs cone-like structures with diameters of 15–100 nm and lengths up to 2  $\mu\text{m}$  on a GaAs substrate by metal-organic vapor-phase epitaxy. It was found that the growth direction was parallel to the [111] arsenic dangling-bond direction and were controlled by the crystallographic orientation of the GaAs substrate surface. It was further found that the whisker/cone-like structures were zinc-blende type for the growth temperature range of 460–500  $^{\circ}\text{C}$ , but changed to the wurtzite type at 420  $^{\circ}\text{C}$  and temperatures higher than 500  $^{\circ}\text{C}$ . These studies represents important steps forward in extending conventional substrate-based epitaxial growth to out-of-plane structures, but faced many of the same limitations of conventional 2D processing that general synthesis of NWs with arbitrary structure and composition that was needed to realize the potential promise as building blocks for nanotechnology.

## 1.4 Beginning of Rapid Growth: Vapor-Phase Nanocluster Catalyzed Growth

It has long been recognized that metal vaporization can be easily accomplished by irradiation with pulsed high-power lasers [50], much earlier than its use in the growth of 1D nanostructures. For instance, Smalley and coworkers [41] combined the laser vaporization technique with the pulsed supersonic nozzle technology for generating nanometer-sized metal nanoclusters. In 1997 and 1998, the Lieber group [34, 51] introduced a laser ablation method for VLS growth of Si and germanium (Ge) NWs in truly nanoscopic dimensions. Specifically, laser ablation was used to prepare nanometer-diameter catalyst clusters that define the size of wires produced by VLS growth. This approach was used to prepare bulk quantities of uniform single-crystal SiNWs with diameters of 6–20 nm, and germanium NWs with diameters of 3–9 nm, respectively. The lengths of these NWs ranged from 1 to 30  $\mu\text{m}$ . Studies carried out with different conditions and catalyst materials confirmed the central details of the growth mechanism and suggested that well-established phase diagrams can be used to rationally predict catalyst materials and growth conditions for the preparation of NWs.

In 1998, Lee and coworkers [52] also reported a high-temperature laser-ablation method for the preparation of SiNWs, although a distinct oxide catalyzed NW growth mechanism was proposed. The growth rate was measured as 10–80  $\mu\text{m}/\text{h}$ . TEM investigation showed that the NWs were crystalline Si, and had diameters ranging from 3 to 43 nm and lengths up to a few hundreds of microns. Twins and stacking faults were observed in the Si core of the NWs. The lattice structure and constant of the NWs were nearly identical to those of bulk Si, although the relative XRD peak intensities were different from those of randomly oriented Si crystallites.

Raman scattering from the NWs showed an asymmetric peak at the same position as that of bulk crystalline silicon.

These two independent efforts immediately opened up a new era with substantial opportunities in this research field [36]. Significant progress has been achieved subsequently in controlling the morphology, size, composition and doping, on length scales ranging from the atomic and up [17, 53]. In the next chapter, we will focus on the metal nanocluster catalyzed VLS-growth as a representative method for the bottom up approach.

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