



## Vapor Phase Growth of Semiconductor Nanowires: Key Developments and Open Questions

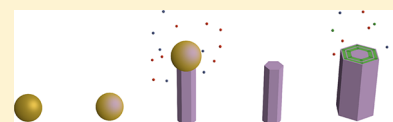
Lucas Güniat,<sup>†</sup> Philippe Caroff,<sup>‡</sup> and Anna Fontcuberta i Morral<sup>\*,†,§</sup>

<sup>†</sup>Laboratory of Semiconductor Materials, Institute of Materials, École Polytechnique Fédérale de Lausanne, 1015 Lausanne, Switzerland

<sup>‡</sup>Microsoft Quantum Lab Delft, Delft University of Technology, 2600 GA Delft, The Netherlands

<sup>§</sup>Institute of Physics, École Polytechnique Fédérale de Lausanne, 1015 Lausanne, Switzerland

**ABSTRACT:** Nanowires are filamentary crystals with a tailored diameter that can be obtained using a plethora of different synthesis techniques. In this review, we focus on the vapor phase, highlighting the most influential achievements along with a historical perspective. Starting with the discovery of VLS, we feature the variety of structures and materials that can be synthesized in the nanowire form. We then move on to establish distinct features such as the three-dimensional heterostructure/doping design and polytypism. We summarize the status quo of the growth mechanisms, recently confirmed by in situ electron microscopy experiments and defining common ground between the different synthesis techniques. We then propose a selection of remaining defects, starting from what we know and going toward what is still to be learned. We believe this review will serve as a reference for neophytes but also as an insight for experts in an effort to bring open questions under a new light.



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### 1. INTRODUCTION

Nanowires are filamentary crystals with a tailored aspect ratio ranging between few and hundred nanometers. Their particular shape and dimensions render them particularly attractive for a large variety of applications and open new perspectives in many others. Reviews and manuals on the synthesis, applications and properties of nanowires are abundant.<sup>1–28</sup> An up-to-date overview on the properties and applications can be found in this issue.

Among several interesting properties, nanowires offer two key advantages in the form of their excellent material quality and the stunning design freedom associated with their morphology. These characteristics open new possibilities both in the advance of engineering and fundamental science. Historically, the bottom-up growth vapor phase is celebrated for providing the highest degree of materials perfection. Opposed to this, the top down approach can potentially lead to defected surfaces and poor control on the resulting facets, with some exceptions.<sup>29,30</sup> Several breakthroughs have conferred nanowires the reputation for outstanding functional properties and design/engineering of materials at the nanoscale level.

This review provides a historical review of what we believe are the main quantum leaps. This gives us the standpoint to conclude with a detailed perspective on what may be the future outcomes, milestones, and challenges to face.

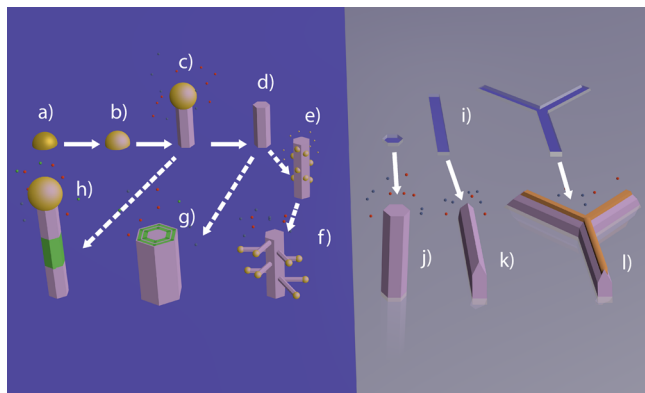
### 2. VAPOR PHASE GROWTH: A HISTORICAL PERSPECTIVE

Nanowires were not born as such. Initial works on the field referred to these structures as whiskers. Whiskers were more of a curiosity-driven experiment of crystal growth than an achievement or breakthrough. They were considered as single crystalline filaments, exhibiting improved mechanical properties with respect to the bulk counterparts.<sup>31</sup> Whiskers were obtained by the precipitation of vapor in slight under-supersaturation.<sup>32</sup> Historically, the first significant milestone for vapor phase growth of nanowires is the work from Wagner and Ellis on the vapor–liquid–solid mechanism, VLS.<sup>33</sup> For the first time, it was realized that a liquid metal droplet (impurity) can mediate the crystallization of silicon at a lower temperature. Crystallization of Si at low temperatures was achieved by alloying gold with silicon for which the eutectic temperature is as low as 360 °C.<sup>12,34</sup> Silicon tetrachloride, SiCl<sub>4</sub>, was provided as the vapor phase as a silicon growth precursor. SiCl<sub>4</sub> would decompose at the gold surface at temperature below its pyrolysis. Alternative to SiCl<sub>4</sub>, silane (SiH<sub>4</sub>) or pure Si can also be used for VLS growth of silicon nanowires.<sup>35–37</sup> Silicon would then form an alloy with the gold until it would melt. Upon supersaturation, silicon would

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precipitate underneath the droplet.<sup>38</sup> The liquid droplet would define both the location of the growth and the lateral size of the nanowire. This process is schematically drawn in Figure 1a–c.



**Figure 1.** An overview of possible nanostructures/heterostructures using VLS and/or VS growth. (a) The primary catalyst droplet, followed by the formation of a eutectic in (b). (c) The formation of the stem wire that can be used to produce (h) axial heterostructure or (d) by removing the droplet catalyst to produce (e), (f) branched “tree like” structure, or (g) radial heterostructure. (i) From openings in an oxide mask, one can also produce (j) nanowires, (k) nanomembranes, or (l) networks by selective area epitaxy (SAE) growth.

VLS has had a major impact in the area of crystal growth, as the main principle can be applied to a broad variety of material systems.<sup>39–41</sup> Still today, it is probably the most commonly used method for the synthesis of nanowires. The liquid component of VLS plays a central role in the process: it not only provides a nucleation point for the solid nanowire, but it fosters the gathering and in some cases the decomposition of precursors. It is because of the latter that it is often called “catalyst” or “physical catalyst” when it does not lead to a reduction of the activation energy for precursor decomposition.

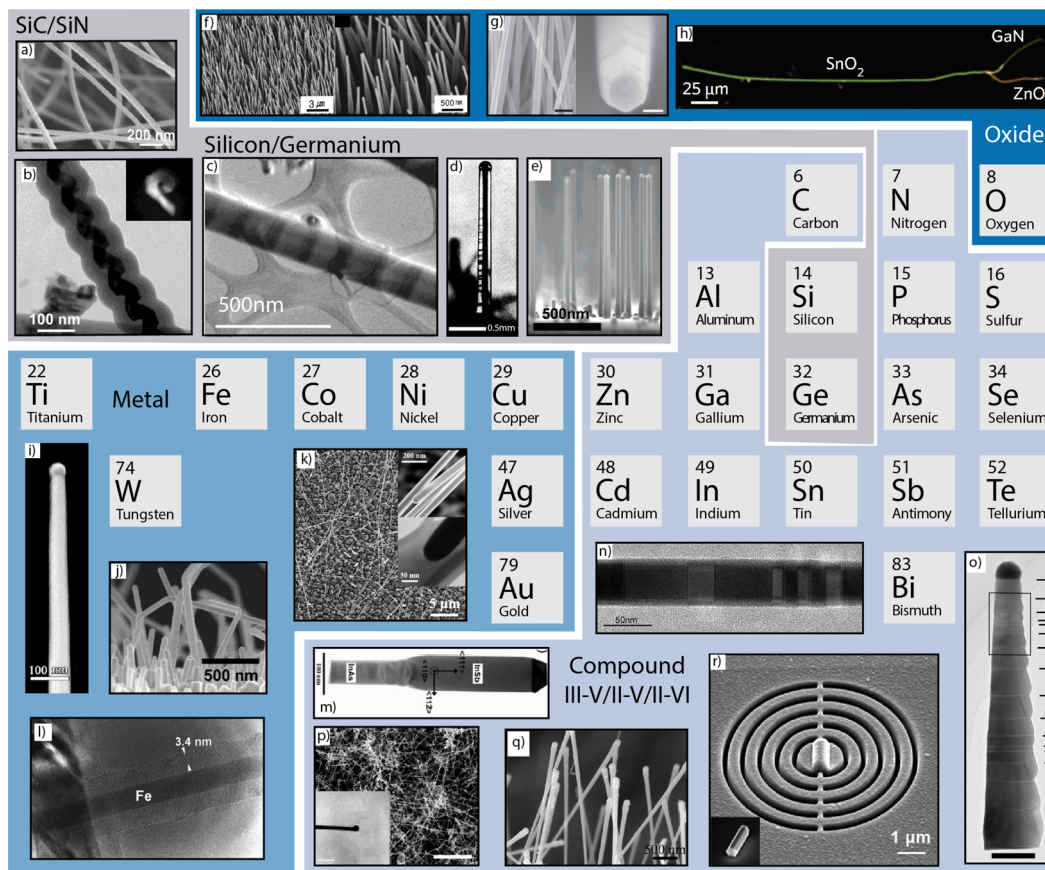
As it was conceived in the early stages, VLS could have been of capital technological importance as it would have allowed the production of Si ingots at a lower thermal budget. Contamination of the Si by the metal used slowed down the activities in this area. Wagner and Ellis did not show particular interest in applying VLS at the nanoscale. Their discovery did not reveal to be a real breakthrough until few decades later at the avenue of nanotechnology. The discovery of fullerenes<sup>42</sup> sparked the interest in nanoscale carbon allotropes, including carbon nanotubes (CNTs).<sup>43</sup> CNTs were obtained from the vapor phase and most commonly by using a ferromagnetic (Fe, Ni, Co) nanoparticle as seed.<sup>44</sup> At least in the early stages, CNTs were obtained also in the vapor phase.<sup>43</sup> CNTs could be obtained in relatively high volumes in the form of black powder.<sup>45</sup> For the fabrication of devices, they were dispersed onto a substrate by previously diluting them in a solution. The growth mechanisms of CNTs and the role of the metal nanoparticles in deciding the chirality was a very vivid field of research for at least two decades.<sup>46</sup> Transmission electron microscopy and theory played a very important role in this progress.<sup>47–51</sup> CNTs renewed the interest in filamentary structures, allowing VLS to be recalled a second time.<sup>52</sup> Pioneers like Lieber, Yang, and Samuelson demonstrated that

high quality nanowire structures could be obtained by VLS.<sup>53–56</sup> Their ideas gave birth to a whole new area of research whose momentum is still increasing today.

Nanowires have been extremely successful as building blocks in nanotechnology research and in proposing novel concepts in domains traditionally dominated by non-nanotech approaches. This success relies in the high versatility of these structures both in growth possibilities and in their functionality, which opens new grounds for existing and novel applications. A few pioneering groups sparked enthusiasm in the synthesis of materials in the nanowire form, initially by demonstrating superior properties with respect to the bulk.<sup>57,58</sup> This recognition inspired research on the synthesis of nanowires in many different materials systems such as ZnO, Ge, Si, and GaN.<sup>41,53,55,59</sup> Lieber and his group recognized very early on that the nanowire geometry and the VLS provide the setting for combining materials in a very different manner than classical thin film or bulk crystal growth.<sup>54</sup> He and his group demonstrated the nanowire composition can be varied abruptly or in a graded manner along and across the nanowire axis. Mastery of the composition on the nanoscale is especially relevant in the field of semiconductors. Aligning materials of different bandgap forming heterostructures results in the formation of internal fields that can localize, store, separate, and/or displace carriers.<sup>60,61</sup> What is particularly novel and attractive with the nanowire geometry is that heterostructure formation can occur in three dimensions.<sup>62–66</sup> Examples of this are depicted in Figure 1h–f. In addition, epitaxy is no longer limited by the condition of lattice match.<sup>67</sup> In this particular morphology, the strain can relax in a more effective manner, providing the setting for novel heterostructures. Strain relaxation is much more effective for axial than for radial structures.<sup>67,68</sup> One should also note that in the eventual case that dislocations will form in nanowire heterostructures, they are often of the misfit kind.<sup>69</sup> In addition, they do not significantly affect the functionality of the materials/devices.<sup>70,71</sup> Finally, one should highlight other design opportunities provided by selective area growth on a patterned substrate, as shown in Figure 1j–l. Here, restricting epitaxy in nanoscale regions of a substrate allows for additional nanowire geometries, junctions, and heterostructure formation.

Figure 2 depicts the extensive materials deployment of vapor phase growth of nanowires. To emphasize this, we have adopted the scheme of the periodic table of the elements, highlighting the elemental components found in this crystalline form. Starting with single elemental structures, we find nanowires made of transition metals: Ti, Fe, Co, Ni, etc. The applications envisioned for these structures are composites, spintronics, and interconnects. We find then single elemental semiconductors, mainly group IV, such as C (diamond), Si, Ge, and Sn. Finally, there is a large range of compounds from III–Vs, II–Vs, and oxides.<sup>72–89</sup> Overall, this figure shows the universality of the one-dimensional structures along many materials systems as well as their use in a vast number of applications.

The mastery of the composition in nanowires also apply to doping schemes. The ability to engineer the electrical conductivity through impurity doping is an essential property of semiconductors and thus a necessary step for the implementation of semiconductor nanowire devices. This was recognized early on. Already in the late 1990s Hiruma et al. provided evidence of doping of GaAs nanowires.<sup>52</sup> Interestingly, the nanowire configuration allows for three-



**Figure 2.** This scheme shows the variety of materials that can be used to build nanowires using vapor-phase growth. Examples of works are shown here such as (a) SiN,<sup>72</sup> (b) SiC,<sup>73</sup> (c) SiGe heterostructures,<sup>74</sup> (d) Si,<sup>75</sup> (e) Ge,<sup>76</sup> (f) and (g) ZnO,<sup>77,78</sup> (h) SnO<sub>2</sub>,<sup>79</sup> (i) W,<sup>80</sup> (j) Cu,<sup>81</sup> (k) Ag,<sup>82</sup> (l) Fe,<sup>83</sup> (m) InSb and InAs heterostructures,<sup>84</sup> (n) GaAs,<sup>85</sup> (o) InAs,<sup>86</sup> (p) CdSe,<sup>87</sup> (q) Zn<sub>3</sub>P<sub>2</sub>,<sup>88</sup> and (r) InP.<sup>89</sup> Adapted with permission from ref 72. Copyright 2003 Elsevier. (d) Adapted with permission from ref 75. Copyright 1964 American Institute of Physics. (f) and (g) Adapted with permission from refs 77 and 78, respectively. Copyright 2002 and 2005 American Institute of Physics and Nature, respectively. (h) Adapted with permission from ref 79. Copyright 2009 Nature. (i) Adapted with permission from ref 80. Copyright 2007 Elsevier. (l) Adapted with permission from ref 83. Copyright 1999 American Institute of Physics. (m) Adapted with permission from ref 84. Copyright 2008 Wiley-VCH. Adapted with permission from ref 87. Copyright 2000 Wiley-VCH. (q) Adapted with permission from ref 88. Copyright 2016 The Royal Society of Chemistry.

dimensional doping profiles such as radial p–n junctions.<sup>52,90,91</sup> In addition, structures of lower dimensionality such as quantum wires and quantum dots can be added on the nanowire facets to increase the functionality.<sup>28,64,92–94</sup> Finally, the detached nature of nanowires allows for a further extension of the free-standing structures in three dimensions. Nucleation of a second generation of nanowires on the facets results in the formation of branched and/or hierarchical structures, which can be used in applications where a large surface-to-volume ratio is required,<sup>95</sup> and electronic devices.<sup>96</sup> This design freedom provided by the nanowire morphology and growth mechanism inspired many materials systems and applications. The easy positioning of the structures via the catalyst on any substrate also opened up new possibilities and the idea of integration with other technologies.<sup>97,98</sup> Among the achievements allowed by this new design possibility: single electron transistors, Majorana physics, biosensors, quantum dot single photon emitters, light emitting diodes, solar cells, etc.,<sup>91,96,99–108</sup>

An unexpected avenue of nanowire technology has been crystal phase engineering.<sup>109</sup> While in bulk materials there is most often a unique crystal structure stable in normal conditions (except for highly ionic crystals such as SiC, GaN, or when using heavy doping<sup>110–112</sup>), nanowires have

shown stable phases otherwise unstable in the bulk. As an example, III–Vs are found both in zinc-blende (bulk phase) and wurtzite structure,<sup>113</sup> silicon and germanium have been synthesized in hexagonal form.<sup>114,115</sup> The hexagonal or wurtzite structure is the result of the modification of the stacking sequence along the (111) direction. The stacking modification can be engineered for the formation of intermediate polytypes and twinning superlattices,<sup>20,22,116–120</sup> opening possibilities for the formation of controlled crystal phase quantum dots.<sup>121,122</sup>

While the large surface-to-volume ratio plays a role in the stabilization of metastable phases, it is only recently that the microscopic reason was elucidated. Even though the importance of the contact angle of the liquid phase in the VLS process was clear from the early ages,<sup>39,113,123</sup> recent in situ studies went a step further. Nanowire growth was followed in situ in a transmission electron microscope. A contact angle close to 90° was associated with nucleation of each bilayer at the triple-phase-line, TPL.<sup>124,125</sup> In this case, energy balance favors the formation of a wurtzite segment as its surface energy at the {11–20} planes is lower.<sup>113</sup> An increase in the contact angle is associated with the formation of a wedge or truncated facet at the liquid–solid interface, favoring nucleation of the new bilayer at a distance from the TPL.<sup>126–128</sup> In this

configuration, zinc-blende is favored over wurtzite. When the nucleus is not exposed to the vapor phase, the most stable phase is regained for zinc-blende.<sup>129</sup> Such an insight could be demonstrated experimentally thanks to one of the most recent breakthroughs in this area: the possibility of observing growth in situ with the precision of a transmission electron microscope. This technique has brought many additional insights that would not have been possible otherwise, to name a few: confirmation that VLS can occur below the eutectic,<sup>130,131</sup> formation of an edge at the TPL,<sup>7,132</sup> variation of instantaneous nanowire growth rate as a function of the crystal phase or presence of defects,<sup>133</sup> and most recently the faceted nature of the bilayer during its formation.<sup>128</sup>

Coming back to the growth mechanism, both the synthesis methods and the microscopic understanding have increased in sophistication with the years. In addition to in situ studies, much progress has been obtained from the growth studies in regular arrays, including the effect of internanowire distance in the diffusion of precursors and shadowing as well as the geometry of the nucleation sites.<sup>134–136</sup> A slight variation of the VLS method consists of the VSS method, standing for vapor–solid–solid.<sup>130,137</sup> The mechanism mostly identical to VLS, the only difference being that the metal droplet stays solid during the whole process. This results into a bit slower nanowire growth. The main advantage of VSS is the possibility of obtaining sharper heterostructures, potentially down to the monolayer accuracy. Growth precursors tend to be much less soluble in the solid than in the liquid metal. Consequently, the switch of precursors does not result in a significant reservoir effect.<sup>130,137</sup>

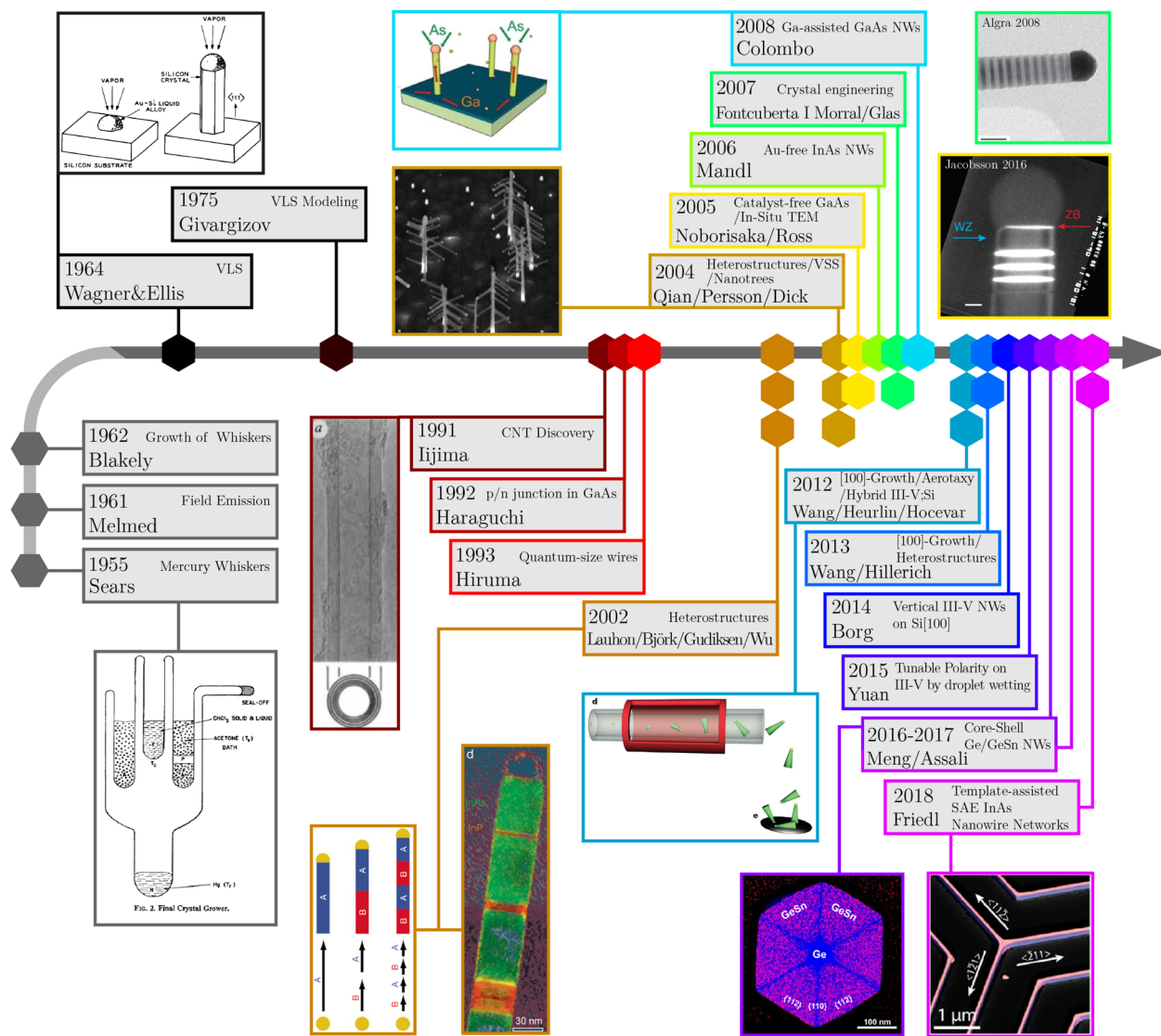
This last point brings us to the choice of catalyst for nanowire growth. Au is the most employed method in VLS as it does not oxidize and supports well the decomposition and gathering of many growth precursors. Still, the use of gold is incompatible with CMOS technology. Gold introduced deep traps in silicon, and it thus damages the electronic properties at very low concentrations.<sup>138,139</sup>

The unsuitability of Au in Si technology has led to the question of what makes a metal a good catalyst for VLS or VSS growth.<sup>22,140</sup> The following criteria should be taken into account for the choice of the catalyst: (i) solubility of the precursor in the metal, (ii) solubility of the catalyst in the material to be grown, and (iii) surface tension of the catalyst. First, indeed, the precursor should be to some extent soluble in the metal so that the catalyst can gather it. The thermodynamic phase diagram of the two will determine the temperature range at which VLS and/or VSS can take place. The thermodynamic phase diagram also provides information on the solubility of the growth precursor. CALPHAD has been used to determine the phase diagrams for new systems in which such diagram did not exist.<sup>141</sup> The solubility of the metal in the grown material is also key. A nonzero solubility value leads to incorporation of the metal during growth. Consequently, the size of the catalyst is progressively reduced until it disappears and the grown material is contaminated. Incorporation of the catalyst in the nanowire can lead to damage on the functional properties or doping, depending on the type of levels introduced. As an example, In or Ga have been used for the growth of Si and Ge nanowires. The catalyst was consumed during growth, leading to p-type structures.<sup>142–144</sup> Subsequent radial growth led to the formation of a p–n junction and a nanowire-based solar cell device.<sup>145</sup>

Finally, it has been argued that surface tension of the liquid metal is also a fundamental property to consider for successful nanowire growth. It has been suggested that low values should not allow for VLS growth due to the lack of stability.<sup>146</sup> This criterion results from the adaptation of the Young equation describing wetting on planar surfaces. Liquids with low surface tension tend to form flatter droplets, which are not favorable to VLS. One should note here Young's equation should not be applied for the wetting of liquids on nonplanar structures such as nanowire tips. Stability of a droplet on a tip cannot be modeled by this equation. In this configuration, a wide range of wetting angles are possible by just modifying the droplet volume,<sup>127,147</sup> in principle in contradiction with the wetting on planar surfaces (Ghisalberti et al., 2019, *Nanotechnology*, <https://doi.org/10.1088/1361-6528/ab139c>). Experimentally, low surface tension metals were indeed extensively used for nanowire growth.<sup>90,142,148</sup> Surface tension also plays a role in the Gibbs–Thomson effect as it determines the diameter below which precursors are not able to incorporate in the liquid metal, thereby stopping nanowire growth.<sup>149,150</sup>

Alternatively, one can just use an element composing the grown material for VLS. This method is also called self-assisted growth. This kind of growth was already predicted in the seminal work by Wagner and Ellis: “an excess of one of the component materials can act as a liquid-forming impurity,”<sup>33</sup> the impurity here taking the role of “catalyst”.<sup>151</sup> As an example, Ga or In can be used to assist the growth of GaP, GaAs, and InAs, respectively.<sup>90,152–154</sup> A difference here with traditional VLS and VSS is that the catalyst needs to be continuously refilled, otherwise the axial elongation of the nanowire stops. Also here, the thermodynamic phase diagram acts as a guide to determine the growth parameter space. To illustrate this, one can look at the thermodynamic phase diagram of GaAs.<sup>155</sup> The growth of GaAs and related compounds relies on the thermodynamic equilibrium between the stoichiometric GaAs and the vapor phases Ga and As<sub>2/4</sub>. Thanks to this, the impinging Ga and As<sub>2/4</sub> fluxes must not contain the exact stoichiometry of the GaAs compound. In thin film growth, an excess As<sub>2/4</sub> is preferred as this is desorbed; while in self-catalyzed growth, the excess Ga stays liquid at the tip of the nanowire.<sup>155</sup> Liquid-phase epitaxy predates the self-catalyzed growth, and one has now recognized that mechanism as part of a macroscopic parent. Alternatively, self-catalyzed growth is the nanoscale version of liquid-phase epitaxy.

Among the advantages of self-assisted growth are the high purity, the possibility of varying the catalyst size/volume. In itself, the free variation of the catalyst volume allows a relatively straightforward path to modify the contact angle and the nanowire diameter. The value of the contact angle has a direct impact on the crystal phase<sup>156</sup> and the regulation of the diameter.<sup>90,157,158</sup> The straightforward modification of the catalyst volume also provides obvious means to switch from axial to radial growth and vice versa. Axial elongation can be ceased by complete consumption of the droplet. Further epitaxy results into radial structures.<sup>159</sup> On the other hand, axial growth can be resumed by simply redepositing the catalyst at the nanowire tip.<sup>160</sup> Nonetheless, self-catalyzed method comes with challenges such as the need of a well-balanced precursor's flux which affects the steady-state growth but also the crystal phase control. An alternative path to avoid gold as a catalyst is to simply avoid any metal or intermediate phase. This is often referred as vapor–solid, VS. VS relies on the imbalance in crystal growth velocities, depending on their

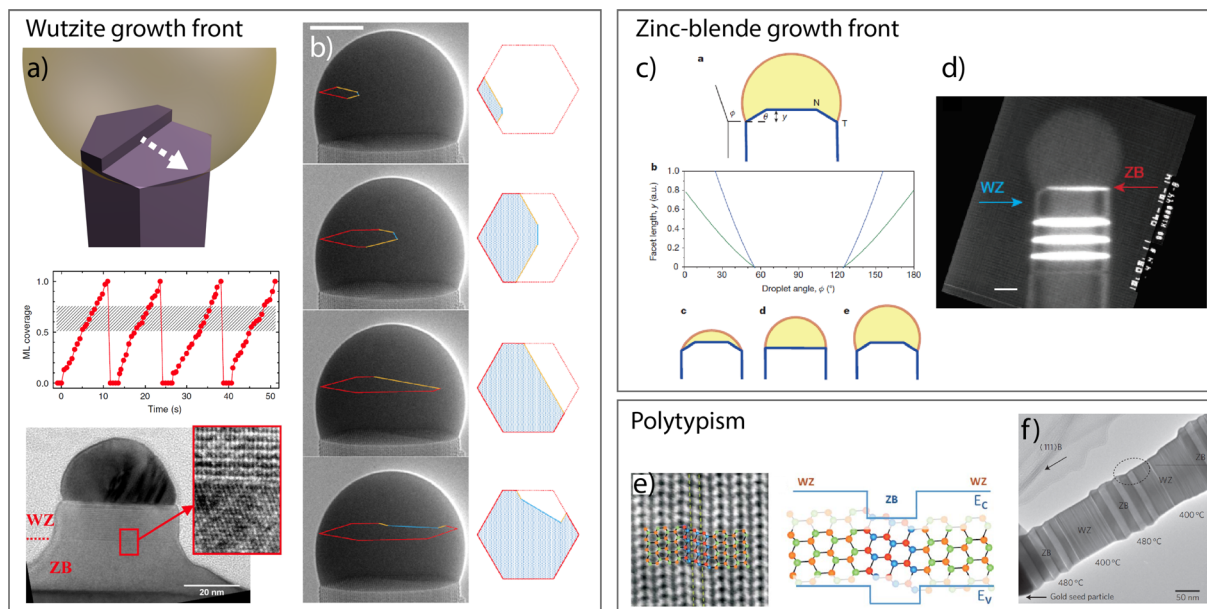


**Figure 3.** Milestones/breakthroughs from historical perspective. Some representative images are included for illustration.<sup>32,43,75,90,95,118,127,180,183–186</sup> Adapted with permission from ref 32. Copyright 1955 Elsevier. Adapted with permission from ref 75. Copyright 1964 American Institute of Physics. Adapted with permission from ref 43. Copyright 1991 Nature. Adapted with permission from ref 183. Copyright 2002 Nature. Adapted with permission from ref 90. Copyright 2008 The American Physical Society. Adapted with permission from ref 184. Copyright 2012 Nature. Adapted with permission from ref 127. Copyright 2016 Nature Adapted with permission from ref 118. Copyright 2006 Nature.

crystallographic orientation.<sup>161,162</sup> There are two main approaches to obtain nanowires by VS: in a self-assembled manner and by selective-area growth, or selective-area epitaxy (SAG or SAE) on localized areas of a patterned substrate. Direct self-assembly usually relies on a lattice-mismatched substrate where growth does not proceed on a layer-by-layer fashion. It rather starts by islands that then evolve into nanowires.<sup>163–165</sup> In some studies, it has been claimed that growth starts with a liquid droplet.<sup>166–168</sup> Typical examples of self-assembled VS-grown nanowires are GaN on sapphire or silicon and InAs on silicon.<sup>166,169–172</sup> Both in MBE and MOCVD, VS growth tends to result in a higher number of stacking faults compared to VLS.

VS can be initiated with a higher yield by the use of a mask. As an example, where a dielectric mask is, we keep an unfavorable nucleation site and remain free of III–V growth under appropriate conditions. Adatoms are able to diffuse

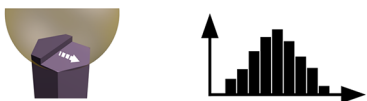
through this mask but cannot easily nucleate a droplet or crystallize, rather they desorb. Small openings or pinholes in the mask act as nucleation points for growth. These can be random due to the partial discontinuity of the oxide layer (aka pinholes) or provoked in a deterministic manner by nanolithography approaches.<sup>165,173–177</sup> SAE can be used to achieve freestanding structures but also structures and networks that are solidary with the substrate. Defect-free nanoscale membranes have been achieved by SAE both by metal–organic chemical vapor deposition and molecular beam epitaxy.<sup>178,179</sup> These membranes can be used as 1D buffer structures for more complex and scalable networks; see image on the bottom right in Figure 3.<sup>180</sup> They can host 1D structures on top, which at the same time can be branched and contacted electronically.<sup>180,181</sup> Among the advantages of the SAE approach are the deterministic localization of the structures, scalability of the process, and a larger freedom in



**Figure 4.** Scheme showing several breakthrough in in situ measurements of the TPL for GaAs nanowires. (a,b) Studies by Harmand, Glas et al. on WZ growth front,<sup>113,207</sup> (c,d) Details of the discoveries by Jacobsson et al. on ZB growth front.<sup>127</sup> (e) The effect of polytypisms on the energy bands<sup>208</sup> and (f) on the morphology of GaAs nanowires.<sup>209</sup> Adapted with permission from ref 113 and ref 207. Copyright 2007 and 2018 American Physical Society. (c,d) Adapted with permission from ref 127. Copyright 2016 Nature. (e) Adapted with permission from ref 208. Copyright 2009 The American Physical Society. (f) Adapted with permission from ref 119. Copyright 2009 Nature.

### What we know:

- o Nanowires grow on a layer-by-layer fashion, starting on a single nucleus. [128]
- o Nucleation statistics of each layer follows a sub-poissonian distribution.[202]



- o The triple phase line can be the nucleation point of a new layer.[128]
- o The presence of truncated facets at the liquid-solid interface is associated with the growth of zinc-blende phase. [127]
- o Engineering the contact angle of the catalyst supports crystal phase and growth direction engineering.[147][190][191]



- o Composition limited by the solubility of precursors in the metal droplet and by the chemical potentials. Limited compositions in multidinary alloys and in heterojunctions. [141][149][150][214]
- o Hierarchical scalable structures: a second life for selective area epitaxy allowing the formation of scalable networks and hybrid structures (e.g. semiconductor-superconductor junctions).[180][181]
- o It is possible to form heterostructures formed by materials of different families (Si/GaAs).[20]
- o The sharpest nanowire-based heterostructure is obtained by a crystal-phase homojunction.[22]
- o Nanowire growth seems to support the formation of otherwise metastable phases and alloys (wurtzite, GeSn).[113][223][224]



- o Surface and interface defects can degrade considerably the functional properties of nanowires.[225-228][229]

### Open questions:

- o Can we go beyond the sub-poissonian distribution? Is it possible to achieve perfect superlattices as in traditional epitaxy? To control supersaturation at the atomic limit ?



- o Where is the nucleation point in a truncated liquid-solid interface ?
- o The microscopic picture relating the contact angle, truncation and crystal growth direction. What is really happening at the LS interface and TPL?
- o The microscopic understanding of the liquid-solid interface at the crossing of two nanowires and its effect on the crystal structure.



- o The role of the surface in ordering at the nanoscale in multidinary alloys. Can it be engineered ?
- o What is the limit in the formation of multidinary alloys in terms of composition control ?
- o Can we avoid VLS for certain alloy compositions and junctions (use SAE/SAG) ?
- o Mechanisms of SAE and TASE, phase tuning/control of wires and networks.
- o The limits of SAE/SAG in terms of : scalability, integration of mismatched materials and the formation of hybrid structures.
- o How can we exploit the ability to form metastable phases for a bandstructure engineering in the broadest sense?



- o Surface and interface defect formation: How much is this given by thermodynamics and how much can be engineered?

**Figure 5.** Table summarizing the advances in the nanowire vapor-phase growth field and the questions that are still to be answered.

shape engineering to suit a large pool of potential applications while retaining lots of the nanowire features. It opens real perspectives toward mass production of connected nanostructures.<sup>182</sup>

A further sophistication of SAE consists in defining the nucleation position of the nanowire but also its growth path. This is the so-called template-assisted selective area epitaxy, TASE.<sup>187,188</sup> This kind of growth is particular of MOCVD, the

difficulties from MBE originate from the directionality of the growth precursors in contrast to gas molecules. A slight variant of this method is also called CELO: confined epitaxial lateral overgrowth.<sup>189,190</sup> Here, nanoscale galleries are fabricated in a dielectric (usually SiO<sub>2</sub>). The end of the gallery is in contact with a crystalline seed (usually Si), which selectively starts nanowire growth. Following this method, defect-free GaAs nanowires have been obtained monolithically on silicon.<sup>136,191</sup> As for SAE, TASE is fully scalable on any wafer size. It is performed on Si (100), allowing for a mostly certain integration of compound semiconductors on the silicon platform.

This brings us to the question of how much can the growth direction in nanowires be engineered. The easy growth axis is (111). Most polar materials even precognize a particular polarity over a large range of growth parameters. In the vast majority of cases the  $\langle 111 \rangle$  B polarity is preferred for III–V and II–IV semiconductor nanowire family. For instance GaAs nanowires tend to form along the (111) B direction independently of the growth mechanism, while InP nanowires form with B polarity for metal-assisted VLS growth and in the  $\langle 111 \rangle$  A for particle-free selective area growth. Still, it has been shown that these natural growth directions can be engineered (De La Mata 2019, *Nano Lett.*, submitted). Initial studies pointed out that the growth direction may depend on the nanowire diameter, suggesting the prominent role of the surface-to-volume ratio in stabilizing the facets.<sup>192,193</sup> It has however been shown that growth direction and polarity can be tuned by engineering the reactant concentration and catalyst contact angle.<sup>147,194,195</sup> In nonpolar group IV semiconductor nanowires, excellent control over the growth direction between  $\langle 112 \rangle$  directions was achieved via a modulation of the precursor concentration in the droplet.<sup>196</sup> Similarly, InP nanowires were shown to be tunable between the  $\langle 001 \rangle$  and  $\langle 111 \rangle$  direction with remarkable control by varying the group III concentration in the droplet.<sup>194</sup> Growth direction engineering opens many perspectives in heterogeneous integration of materials as well as for the formation of 3D nanowire structures and networks.<sup>196–199</sup>

Finally, the substrate is not needed to start nanowire growth. Using the natural tendency of nanowires to solidify at the interface with the catalyst, it was shown that they could be obtained in a single crystalline manner in the gas phase. This new kind of growth is called aerotaxy.<sup>184</sup> Aerotaxy or growth in the gas phase was further sophisticated for the growth of nanowires and related junctions on the gas flow and on demand.<sup>200–202</sup> To provide an overview of the time line of the field, we have summarized the main milestones in a time arrow in Figure 3.

### 3. OUTLOOK: QUO VADIS NANOWIRE GROWTH?

We now switch gears to discuss the outlook and open questions in the area. Figure 3 provides a timeline of the milestones achieved in the last 63 years. It is clear that the level of sophistication and progress has been accelerated, especially in the past few years. Most of it has been driven by the higher level of understanding achieved, enabled by techniques, such as nanoSIMS, synchrotron, and Cs-corrected TEM, and stimulated by the broad range of avenues that nanowires promise. Extrapolating this trend, future evolution will probably be driven by the still open questions. Here, we propose still unanswered matters whose answers should spark new avenues. Figures 4 and 5 summarize a list of accepted paradigms in the

field of vapor phase nanowire growth along with the open questions they leave. Answering the open questions will certainly derive into new paradigms, some of them may contradict what is widely accepted today.

The direct observation of crystal growth in an electron microscope with spatial resolution down to the atomic size is breathtaking. This kind of experiment has brought considerable advancement in understanding the growth process by providing images of the process in an instantaneous manner.<sup>127,128</sup> The recent technical progress in in situ transmission electron microscopes allow monitoring growth with atomic resolution. This kind of experiment also provides the necessary knowledge to generalize the basic growth mechanisms independently of the growth technique. Some of the recent findings in the topic of polytypism are represented in Figure 4. While previous works argued that the growth occurs on a layer-by-layer basis and the point of nucleation plays a major role in defining the phase, only recently these two assumptions could be verified.<sup>127,128</sup> We expect that the next breakthroughs will be achieved when ultrahigh speed cameras will be used, capturing growth instants well below the ms.

So far, it is well accepted that nanowires progress on a layer-by-layer fashion. The time spacing each bilayer nucleation follows a subpoissonian statistics, a consequence of emptying the liquid reservoir each time a bilayer has been obtained.<sup>203</sup> The existence of subpoissonian nucleation statistics implies that the elongation of nanowires/heterostructures cannot be determined down to the monolayer precision.<sup>204–206</sup> This is contrary to traditional thin film epitaxy, in which the thickness of layers is precise down to the monolayer. Consequently, new strategies should be developed in the future for the creation of perfect superlattices and/or perfectly reproducible quantum heterostructures.<sup>206</sup> One possibility could be the control of the liquid supersaturation down to the atomic liquid. This would require not only growth at a very low rate but also achieving liquid catalysts of exactly the same size in an array.

The role of the triple-phase line, TPL, in the nucleation of each new bilayer is also well established. For a flat liquid–solid interface, in situ TEM studies demonstrated that growth starts at a point of the TPL and extends from there. The nucleation point of a new layer for a truncated liquid–solid interface is less clear. Partly, this is due to the fact that the new layer appears so fast that current microscope systems cannot yet capture the initial stages. Some works suggest that nucleation of new bilayers occurs far from the TPL in the truncated facet. This hypothesis has yet to be demonstrated. The formation of each new bilayer is accompanied by the instantaneous disappearance of the truncation. It could be the formation of a new bilayer as well. Nucleation could thus still ensue at the TPL. In addition, one could point out to several other questions that are still unanswered: what is the role of liquid ordering at the liquid–solid interface in the formation of every new bilayer?<sup>210,211</sup> What is the nature of layer-by-layer growth of a nanowire growing in the (100) direction?

This brings us to another open issue. Several groups have exposed that contact-angle engineering of the liquid phase in VLS supports the control on the crystalline growth direction and even polarity.<sup>147,194</sup> Even though they have established a direct correlation between the two, the microscopic picture explaining the mechanism is still lacking. In particular, it is not clear how the contact angle favors certain crystalline orientations in the initial stages and/or along the growth.

There is still a causality issue in between contact angle and modification of the balance of surface and interface energies: which is causing which? Understanding these points will permit growth of compound semiconductor nanowires along the (001) direction on silicon. This is the final milestone needed for a realistic integration of optoelectronic applications on the silicon CMOS platform. It may also provide new solutions in the era “beyond Moore”.

We would also like to point out the open questions with respect to SAE/SAG. Most publications assume the growth mechanism is well-known. However, we believe this is not the case. As an example, it is not clear why polytypism exists in this kind of growth and whether a liquid phase can be present at the initial stages and/or during growth.<sup>212</sup>

One of the most discussed advantage of nanowires is the potential for integrating materials with a high lattice mismatch or even of a different family (e.g., InP/InAs or GaAs/Si/GaAs). The integration of materials of different families such as semiconductor/ferromagnetic or semiconductor/superconductor in so-called hybrid structures has been demonstrated.<sup>213–215</sup> We believe the achievements are still limited, and they have not yet been explored to its full potential, as illustrated by the new promises of superconducting metal/semiconductor radial heterostructures.<sup>216</sup>

The above-mentioned points today still leave many open questions even in the traditional heterostructure area. First, the sharpness in the heterostructure formation is mostly dictated by the thermodynamics at the liquid phase.<sup>20,67,68</sup> As a result, changes in the nanowire composition are not possible with monolayer precision. The only atomically sharp heterostructures possible are of the crystal phase kind.<sup>22</sup> The ruling of the nanowire composition by the liquid phase can also impose some limitations in the nanowire composition. Dubrovskii, Glas, and Johansson have demonstrated the existence of solubility gaps in ternary alloys such as InGaAs<sup>141,149,150,217</sup> that may also exist in other element combinations. The synthesis of nanowires with any desired composition at the ternary and quaternary levels and beyond is still to be resolved. Similarly, it is not yet clear whether multinary alloys with a random distribution of the elements can be obtained. Several studies point out the ordering effects at the nanoscale both in the nanowire core and shell.<sup>62,218–222</sup> Mastering of the order of multinary alloys at the atomic and nanoscale is still an open issue to be explored and debated.<sup>223–225</sup>

Finally, one should point out the ability of VLS to produce materials in a metastable state. We have discussed the formation of polytypes in nanowires. In addition to this, it has been shown that nanowires support the growth of metastable composition alloys such as GeSn beyond the Sn solubility limit.<sup>186,226</sup> The technological impact of this is very high, as it opens up even more possibilities in the area of bandgap engineering. We expect to see many advances in this front in the coming years.

Last but not least, we would like to address the impact of surface and interface defects on the functional properties of nanowires. Among the effects of surface and interface defects are the increase of linewidth in optical emission of nanowires and nanowire-based quantum dots<sup>227–230</sup> and hysteresis in the transfer characteristics of transistors.<sup>231</sup> Although the functionality of nanowires is not addressed here, the origin of defects is of a pure materials processing origin. We need to understand how surfaces and interfaces can be produced in a pristine and reproducible manner if nanowires are ever to be integrated in

the optoelectronics or electronics industry. In this area, we may expect the synergistic collaboration between surface scientists, theorists, and epitaxy experts to bring important and necessary outcomes.

As the top-down method often appears to be the practical choice, it is important to keep in mind that a highly aesthetic bottom-up growth plants its roots in the most fundamental techniques used by nature. Overall, we believe vapor phase growth of nanowires will continue delivering discoveries in the area of crystal growth. Providing new paradigms of crystal formation will certainly impact this century's technology in the most diverse aspects.

## AUTHOR INFORMATION

### Corresponding Author

\*E-mail: [anna.fontcuberta-morral@epfl.ch](mailto:anna.fontcuberta-morral@epfl.ch).

### ORCID

Lucas Güniat: 0000-0001-7883-4433

Anna Fontcuberta i Morral: 0000-0002-5070-2196

### Notes

The authors declare no competing financial interest.

### Biographies

Lucas Güniat is a Ph.D. student in the laboratory of semiconductor materials (LMSC) in the material science department of the École Polytechnique Fédérale de Lausanne (EPFL) since January 2017. His interest for epitaxy and nanoscience lead him to study III–V implementation on Si using exotic nanostructures. He also studies MOVPE reactors for nanowire growth and makes extensive use of microfabrication techniques.

Philippe Caroff obtained his Ph.D. degree in physics in 2005 from the Institut National des Sciences Appliquées (INSA Engineering School) in Rennes (France) and was a postdoctoral scholar in Lund University from 2006 to 2008. He became a tenured CNRS Research Scientist in 2008 and worked in Lille (IEMN), France, for four years before joining the Australian National University (ANU), Department of Electronic Materials Engineering, in 2013 as an independently funded Australian Research Council Future Fellow. He joined Cardiff University and the newly created Institute for Compound Semiconductors (ICS), UK, in December 2016 to serve as Sêr Cymru Senior Research Fellow and MBE lab facility manager. Since December 2017, he is senior researcher at Microsoft Quantum Lab Delft, The Netherlands. As a materials scientist, his research interests span the III–As, III–P, III–Sb, and III–N nanostructures and semiconductor/superconductor heterostructures for a wide range of applications ranging from optoelectronics, telecommunication, renewable energy (solar, thermoelectrics), nanoelectronics, to topological quantum computing.

Anna Fontcuberta i Morral obtained her degree in physics in 1997 at the University of Barcelona. In 2001, she obtained a Ph.D. in materials science at École Polytechnique (France) with Prof. Roca i Cabarrocas. She then moved to the group of Harry Atwater at CalTech, where she worked in the area of multijunction solar cells and costarted the company Aonex Technologies. In 2005, she obtained a group leader position at the Walter Schottky Institut of TU Munich, cofinanced with the Marie Curie Excellence grant. Among her recognitions are the ERC starting grant, the SNF consolidator grant, and the EPS Emy Noether prize in physics. Since 2008, she leads the Laboratory of Semiconductor Materials at EPFL. Her research focuses on the exploration of novel semiconductor structures for next generation energy harvesting and computing technologies.



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