# Integration of one-dimensional nanostructures with microsystems: an overview

# **B. Erdem Alaca\***

The current state of solutions provided for the issue of integration between micro- and nanoscales is reviewed for the specific case of nanowires and nanotubes. Such structures serve as field emitters, transistors or laser sources, digits for manipulation and handling, as sensing elements or as agents for the modification of surface properties such as the adhesive strength. However, it is noteworthy that the majority of reported device work remains confined to component level prototype development without the prospect of full scale system integration due to the lack of batch compatible fabrication and processing techniques. On the one hand, nanostructures made by self-assembly do not possess a high level of control on their orientation and numbers, and hence, their interfacing and integration with a microsystem pose difficulties. On the other hand, top-down approaches such as manipulation, serial deposition and high resolution lithographic techniques do not satisfy the needs of large scale fabrication due to their expensive and/or non-parallel working principles. These techniques along with hybrid approaches taking advantage of the structural control of self-assembly and geometric control of high resolution lithography will be discussed and major applications will be highlighted to shed light on the capabilities and limitations associated with each process.

Keywords: Nanowires, Carbon nanotubes, Self-assembly, Top-down fabrication, Lithography, Batch compatibility, Microsystems, Review

# Introduction

A variety of one-dimensional nanostructures, i.e. carbon nanotubes (CNTs) and nanowires (NWs), can be fabricated with a tight control on their structure and dimensions. Most of the early challenges regarding structural control were overcome or a considerable progress has been made. For example, one of the major concerns regarding device applications of CNTs has been the difficulty of differentiating between metallic and semiconducting tubes. Careful synthesis of catalytic particles was reported to increase the ratio of semiconducting CNTs to 70% via chemical vapour deposition (CVD) growth.1 Introducing plasma enhanced chemical vapour deposition (PECVD) as a growth tool further increased this ratio to 90%.<sup>2</sup> Post-fabrication sorting techniques are also under intense study.3,4 Similarly, the structure of NWs can be closely dictated by various epitaxial growth techniques.<sup>5,6</sup> Doping of both CNTs and NWs<sup>7,8</sup> and building NW heterostructures<sup>9–11</sup> were also reported with important implications in electronic and optoelectronic applications.<sup>12-14</sup> Depending on the specific growth technique, application specific dimensional requirements imposed on onedimensional nanostructures can also be met. For example, changing the catalyst size in a vapour phase growth method changes the diameter of the resulting structure.<sup>14</sup> High resolution lithography is a powerful tool in this respect.<sup>15</sup>

Naturally, such progress in dictating the properties on CNTs and NWs had direct implications in device applications, a variety of which were proposed starting from early nineties ranging from photonic and electronic to mechanical elements.<sup>14,16–18</sup> However, the research in this field remains mainly confined to component development, whereas the leap from nanoscale components to a full scale system such as a circuit with NW transistors necessitates further studies on the integration aspect. In the near future the suitability of a fabrication technique for integration with higher level structures will be considered as important as its capability of producing well controlled nanostructures. For example, in spite of their ability of producing high yields of single walled CNTs with structural uniformity, early techniques such as electric arc19 and laser ablation20 were gradually replaced by techniques based on CVD<sup>21</sup> due to the elimination of the need for alignment and hence, better suitability for system integration. Among the successful examples of system level approach to nanofabrication, one can mention IBM's data storage system, the <sup>c</sup>Millipede',<sup>22,23</sup> and Samsung's and others' CNT field emission displays.<sup>24,25</sup> Considering the relatively short history of CNTs,<sup>26</sup> the existence of such systems is surely an indication of a strong potential.

Further progress in this field requires the development of batch compatible integration techniques, where

Department of Mechanical Engineering, Koc University, Rumeli Feneri Yolu, 34450 Sariyer, Istanbul, Turkey

<sup>\*</sup>Corresponding author, email ealaca@ku.edu.tr

resulting nanostructures are fully registered and aligned with respect to microscale components in a parallel fashion. Traditionally, photolithography has been the workhorse of miniaturisation in this respect. Nowadays, around 600 integrated circuits with more than 40 alignment and exposure steps can be fabricated in parallel on a 300 mm diameter wafer.<sup>27</sup> Currently, with 193 nm deep ultraviolet (DUV) lithography with immersion technology, a resolution of 40 nm is achievable, and extreme ultraviolet (EUV) technology is predicted to lead to 32 nm technology node and beyond.<sup>28</sup> Although other top-down techniques such as direct write e-beam provide excellent control on both the location and orientation of nanostructures, they do not provide the ultimate answer, mainly due to their serial working principle. At this point it should be emphasised that an increased resolution that is inherent to top-down techniques may still necessitate the company of further processes for structural and chemical control. Hybrid approaches utilising both top-down techniques and selfassembly in dictating both the coordinates and the structure/chemistry of a nanostructure are good examples of alternative integration techniques that are at their infant stage. Initial demonstrations are usually carried out on flat surfaces of chips with simple microstructures such as electrodes. However, the level of sophistication of such integration studies is steadily increasing.

The merit of any fabrication method is, of course, to be judged according to its targeted application field. Hence, for those systems where a statistical placement of nanoscale extensions suffices, pure self-assembly is an ideal solution. Similarly, when there is a strong need for deterministic placement, even an extremely serial topdown solution such as electron beam induced deposition proves to be a viable approach for low throughput systems, such as a pair of microtweezers. In this paper, a comprehensive review of the current state of the nano-/ micro-integration will be presented. The first section is dedicated to a discussion of the available alignment and integration methods for self-assembly products. This discussion is divided into three parts. The first approach is based on fabricating nanostructures and microscale parts separately and combining them subsequently. In the second approach, integration takes place at the original fabrication sites of nanostructures, where the nanofabrication process itself provides an integration platform with a certain degree of directionality. In the third technique, nanostructures are directionally grown on top of microstructures. In the limit, growing a single nanostructure at a desired spot is possible with this method, turning it into one of the most promising integration techniques. Therefore it is not surprising to see a variety of device demonstrations with directional growth. The remainder of the review provides a summary of top-down methods, where manipulation, lithographic tools, high resolution deposition techniques, and finally, batch compatibility issues are covered. The review is concluded with a brief discussion of adhesion issues between nano- and microstructures.

# Integration with self-assembly products

Self-assembly provides a fast solution to fabricating NWs and CNTs with a tight control on their structure. Available synthesis techniques have been covered by many topical reviews.<sup>5,13,29</sup> One of the most widely used

growth techniques for NWs is the template assisted synthesis, where porous media such as anodic alumina membranes and ion track etched polymers<sup>30</sup> or selectively etched diblock copolymers<sup>31</sup> are filled with a material of choice through techniques such as electrochemical deposition. Vapour–liquid–solid (VLS) method is another synthesis technique based on using a catalyst material, such as Au, in the form of a nanodroplet.<sup>32</sup> The NW material supplied in gas phase is absorbed into the catalyst forming a supersaturated alloy. The growth of a single crystalline NW then takes place through precipitation at the substrate/particle interface. Coating of biological molecules such as peptides<sup>33</sup> or DNA<sup>34</sup> with metal or conductive polymers, and crystal surface decoration<sup>35</sup> constitute other techniques of NW synthesis.

Carbon nanotube synthesis was originally carried out either by laser ablation of carbon targets<sup>20</sup> or by the electric arc technique,<sup>19</sup> where an arc is generated between two graphite electrodes. Both techniques require the presence of transition metals as catalyst material. Chemical vapour deposition and PECVD are now more frequently used to grow CNTs.<sup>2,21</sup> Similar to VLS technique for NWs, CVD provides a very attractive means of location and size control for CNTs by controlling the placement and size of catalyst particles.

Depending on the nature of the synthesis technique, there are three main venues for integrating self-assembly products with microstructures as shown in Fig. 1. It is to be noted that at this stage the microstructure of interest is a simple two-dimensional feature such as a surface electrode rather than a sophisticated microscale device. In the first approach, the microstructure and nanostructures are fabricated separately. Nanostructures are then removed from their fabrication sites and dispersed in an appropriate solvent, where they align under an externally applied field. In the second approach, the synthesis technique allows a certain degree of alignment. This might be, for example, crystal surface decoration or filling of an anodic alumina template. In both cases, the alignment of the resulting NWs is known, in the plane of the substrate for the first technique, and out-of-plane for the latter. Hence, the application of an external field renders unnecessary. In the last technique, a microstructure is fabricated first, upon which NWs or CNTs are grown on selected sites through catalytic reactions using VLS and CVD. Hence, the integration technique of choice determines which self-assembly based synthesis method can be utilised. The relatively high number of devices reported using directed growth - the third technique - indicates its advantage and dominance over other approaches for system integration. In the remainder of this section, these three integration philosophies will be discussed in detail.

#### Remove-disperse-align

In the remove-disperse-align technique, the integration of nano- and microstructures requires further postprocessing and alignment. Nanowires and CNTs are removed from their fabrication sites such as anodic alumina templates and dispersed in an appropriate medium with complete lack of alignment. Alignment is subsequently carried out by using secondary forces. These forces are created either by external fields such as electric, magnetic or flow fields or the forces are generated from within the microstructure achieved



1 Integration methodologies for self-assembly

through selective functionalisation such that nanostructures are attracted toward and attached on these selected sites. If alignment occurs by external fields, aligned objects are to be attached on to the microstructures through more labour intensive techniques. In both methods, the adhesion between NWs/CNTs and microstructures may be improved by depositing thin films or using direct current (DC) welding, which will be discussed at the end of this review under adhesion issues.

#### Alignment under external fields

#### (Di)electrophoresis

The alignment of anisotropic particles in electric fields has a long history and a wide application field.<sup>36</sup> Electric field assisted alignment of NWs and CNTs in a suspension is based on the polarisation of these structures in an inhomogeneous electric field. After the suspension is delivered on to a chip with prepatterned electrodes, NWs and CNTs are polarised in an alternating current (AC) field with or without a DC bias. A dielectrophoretic force arises due to the interaction of the induced dipole moment in the structure with the applied electric field. Using AC fields eliminates electrochemical and electro-osmotic effects associated with the DC field. The geometry and orientation of the electrodes are easily dictated by various lithographic techniques (Fig. 2). Motion of the nanostructure is induced due to the resulting dielectrophoretic force.41

The magnitude of the dielectrophoretic force F, exerted by an electric field E, on a dipole with a dipole moment p, can be approximated by

$$F = (p \cdot \nabla)E \tag{1}$$

Eliminating higher order terms, this approach is based on the assumption that the dimensions of the nanostructure are small compared to the electrode dimensions and is widely utilised.<sup>38,42,43</sup> A generalised approach utilising a Maxwell stress tensor is reported by Ref. 44.

If the nanostructure is more polarisable than the surrounding medium, it will be attracted toward regions

of high electric field strength independent of the field direction, a phenomenon called positive dielectrophoresis. In fact, the difference between the dielectric constants of metallic and semiconducting CNTs makes their separation possible. While a positive dielectrophoretic force is exerted on metallic CNTs and they are collected at the electrodes, semiconducting CNTs under negative dielectrophoresis remain in the suspension.<sup>3</sup> With decreasing distance between nanostructures and electrodes, the field strength increases and nanostructure tips are attached to the electrodes followed by the removal of the solvent. Gaps anywhere between 20 nm and 20  $\mu$ m can be bridged by this technique.<sup>45</sup>

This is in contrast to electrophoresis where the motion of the nanostructure is governed by its net electrical charge. In this case the applied electric field can be uniform as well as divergent. The field emission study on single walled CNTs<sup>46</sup> can be considered as one such example, where surface charge and hence the electrophoretic effect were enhanced by the addition of Mg(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O to CNT powder. It is well known that the sign of the charge is heavily influenced by the nature of the solvent due to the preferential absorption of ions in the solution.<sup>47,48</sup> While a single walled CNT in a NaOH solution is attracted towards the positive electrode, it will be attracted by the negative electrode if it is placed in a MgCl<sub>2</sub> solution.<sup>47</sup> Similarly, in a neutral suspension no electrophoretic effect is observed.<sup>47</sup>  $\hat{A}$  guideline for suspension preparation is supplied in Ref. 49. Electrophoresis was originally proposed as a technique for the purification of CNTs obtained through DC arc discharge method.<sup>50</sup>

In addition to gold nanoparticles<sup>51</sup> Smith *et al.*<sup>52</sup> reported the assembly of gold NWs between two electrodes. Electrodes are buried under a 500 nm thick Si nitride layer, whose thickness is reduced to 100 nm along the centreline of the electrodes to increase the electric field strength. A 1 kHz AC electric field is generated which polarises the metallic NW by creating charge separation at the NW surface. The resulting



2 Examples of dielectrophoretic assembly. (a) A single CNT spanning a gap of 5 μm between a pair of electrodes. The gap is 100 μm deep. (reprinted with permission from Ref. 37, copyright 2005, Institute of Physics) (b) Assembly of Au NWs. (reprinted with permission from Ref. 38, copyright 2005, Institute of Physics) (c) A 9 nm diameter CNT bundle on four Au terminals. (reprinted with permission from Ref. 39, copyright 2003, Springer) (d) A junction of InP NWs resulting from hierarchical assembly (reprinted with permission from Ref. 40, copyright 2001, Nature Publishing Group)

dielectrophoretic force displaces NWs toward the edges of electrodes over the thinned region where NWs are attached to the electrodes at their tips. When a single NW is aligned and attached, the local field strength is reduced in a neighbourhood of 2  $\mu$ m preventing further deposition of NWs in this region. A contradictory conclusion is reached in a similar study on Au NWs, where no reduction of local field intensity was observed.<sup>38</sup> A more detailed explanation for this interaction is provided by Chung *et al.*<sup>53</sup> where the creation of vortex flows around a deposited nanotube due to the distortion of the electric field is claimed to be the primary reason for the lack of deposition around this site.

Initial parametric observations on CNTs under  $AC^{39,54,55}$  and  $DC^{46,50,56}$  fields were followed by more detailed studies. For example, Chung et al. 53 varied the ratio of the strength of AC and DC components. In parallel to pervious studies<sup>45</sup> it is observed that unwanted particle deposition and non-directional multiwalled CNT trapping take place between the electrodes when the DC component is dominant. This is due to the electrophoretic force attracting any charged objects. Trapped particles then disturb the local electric field and increase randomness. This effect combined with the loss of directionality due to sharp electrodes was previously observed to result in bent configurations of trapped NWs.40 However, with increasing AC component at 5 MHz, particle deposition is eliminated and CNT trapping becomes more orderly and dominant.<sup>53</sup> In fact, for a particular multiwalled CNT suspension in isopropyl alcohol, the ratio of CNTs aligned at the

electrode's edge to the total number of nanotubes and particles is reported to increase monotonically and reach 30% at 10 MHz.<sup>54</sup> It is argued that negative ions surrounding CNTs and particles cannot keep up with changing electric fields beyond 10 kHz. This creates a dipole, where the dipole moment is much stronger in CNTs than nanoparticles.<sup>54</sup> Hence, CNTs are easily attracted while nanoparticles cannot be moved. Additionally, the average spacing between trapped CNTs increases with increasing AC component.<sup>53</sup> Similar observations regarding CNTs are reported by Kumar *et al.*<sup>57</sup> If electrophoresis is used, a prior purification step is necessary to remove such particles.<sup>46</sup>

Following an earlier work on biased AC fields,58 another finding by Chung et al.<sup>53</sup> is concerned about the necessity of the DC component for creating this order. A self-limiting effect, where deposition of a nanostructure prevents further deposition as reported by Ref. 52 on Au NWs, is not possible with CNTs due to high contact resistance between CNTs and the electrode. Direct current component is speculated to generate an electroosmotic flow between the electrodes due to the motion of ions present in the suspension. When a onedimensional nanostructure, a CNT in this case, enters the space between the electrodes, it distorts the electric field, which in turn generates vortex flows around the nanotube. These vortex flows are claimed to be responsible for preventing CNTs approaching each other and forming clusters. This phenomenon was used to span two electrodes with a single nanotube with a vield rate of 90%.

One final explanation regarding this screening effect is provided by Diehl *et al.* using ropes of single walled CNTs.<sup>48</sup> Utilising energy equilibrium between repulsive interactions between a rope landing on to the substrate and the already existing ropes on the substrate and Coulombic interaction between the rope and counterions in the solvent, a screening length is obtained. Experiments reveal that the pitch between the parallel CNT ropes is a strong function of the rope length. Furthermore, successive deposition steps are applied successfully to fabricate crossed structures.

Device applications of electric field assisted assembly are rather limited due to yield and compatibility issues, and the majority of them are realised on CNTs rather than NWs. Usually no subsequent lithography step is carried out leaving assembly between simple electrodes as the only option. For example, temperature dependent I–V characteristics of CNTs are measured by bridging two Au electrodes (300 nm gap) with nanotubes using poly(methyl methacrylate) (PMMA) as a sacrificial material underneath the nanotubes. Poly(methyl methacrylate) is subsequently removed after the integration step.<sup>59</sup> A further evaporation of thin Au is carried out to increase adhesion between CNTs and electrodes. Additionally, a microthermal sensor is reported, where an AZ5214 photoresist layer is used as a sacrificial layer and the suspended CNT bridge (gap distance of 3 to 10 µm) is sandwiched between two parylene sheets.<sup>60</sup> Parylene sheets serve the purpose of providing a firm attachment to the electrodes and protection against contamination. Gas sensors,<sup>61</sup> a field emission cathode for X-rays<sup>62</sup> and a strain gage<sup>63</sup> are among other applications. In the example of the strain gage,<sup>63</sup> CNTs are suspended between two electrodes on a microcantilever. When the cantilever is subjected to external loading, the resistance change exhibited by the CNT bridge is used to monitor the strain that the cantilever undergoes.

Recently, out-of-plane assembly of CNTs is also demonstrated, where two gold pads placed on top of each other and separated by a  $0.7 \,\mu\text{m}$  thick parylene layer are bridged by CNTs using AC dielectrophoresis.<sup>64</sup>

Metal oxide structures are also among the materials used in electric field alignment studies. For example, a gas sensor where free standing single crystalline tin dioxide nanobelts are placed between two Si nitride membranes is reported by Yu *et al.*<sup>65</sup> The electrical conductance of tin dioxide nanobelt increases upon exposure to dimethyl methylphosphonate as a result of a surface redox effect that becomes pronounced at elevated temperatures. To span the membranes with nanobelts, AC dielectrophoresis was employed using Pt electrodes on the membranes. Characterisation of the sensor is carried out using samples with only one nanobelt bridge. No details regarding the yield of the process are supplied.

Bridging can also be achieved without the use of a sacrificial layer between the electrodes, which is especially important in the case of deep and narrow trenches frequently encountered in microelectromechanical systems (MEMS). One example is reported by Lu *et al.*<sup>37</sup> where a 100  $\mu$ m deep and 5  $\mu$ m wide gap between two gold electrodes is spanned by electric field assisted assembly of CNTs. To achieve this, the assembly step carried out in ethanol is followed by critical point drying to prevent capillary forces from drawing attached nanotubes into the trenches.

Based on a previous work,<sup>66</sup> Nakayama<sup>67</sup> and Williams *et al.*<sup>68</sup> used dielectrophoretic assembly to fabricate CNT cantilevers along a knife edge that is to serve as a CNT storage for further integration with probes and tweezers. An example for the usage of such a cartridge is the extraction of inner shells of a multiwalled CNT reported by Akita and Nakayama.<sup>69</sup> The study is geared toward fabricating a capped and sharp tip for scanning probe microscopy. The required sliding force for extraction is also measured during the process. A CNT cartridge in the form of a knife edge similar to that described in the dielectrophoretic assembly of Ref. 67 is utilised as a storage for CNTs, where CNTs are assembled in the form of protruding cantilevers. A Si probe tip is brought into contact with the free end of a nanotube and a voltage is applied leading to the electrical breakdown of the suspended nanotube. The destruction of individual shells is visible in the form of distinct decreasing steps in the electrical current measured at constant potential. After the current stops decreasing, the knife edge is removed from the Si probe leading to the extraction of the inner shell. The sliding force is also calculated from the cantilever deflection. A detailed in situ TEM study of the contact problem between nanotubes and AFM tips coated with gold and amorphous carbon can be found in Ref. 70.

As an enhancement to reduce randomness and achieve attachment of Au NWs at predetermined locations, the electric field in Ref. 52 is further increased around  $4 \times 4 \mu m$  spots on top of the thinned Si nitride region where a second set of electrodes (active electrodes) is deposited leading to capacitive coupling with the lower electrodes. The same approach is also adopted by Evoy *et al.*<sup>44</sup> for the assembly of Rh NWs and CNTs. Lower electrodes of gold and upper electrodes of Ni/Cr alloy are separated by a 150 nm thick Si dioxide layer. After the assembly is carried out in an ac field, SiO<sub>2</sub> is released and I–V responses of the suspended nanostructures are measured.

Such capacitive coupling with alternating assembly signal is also utilised by Narayanan *et al.* for the post-IC integration of Rh NWs with CMOS VLSI circuitry,<sup>71</sup> where the two topmost metal layers serve as buried and active electrodes with the final dielectric layer removed over the active electrodes. It is clear that issues specific to IC processes, such as isolation of the readout circuits from assembly sites, still need to be addressed.

An alternative capacitive coupling technique for spanning two electrodes by a single NW or a single bundle of NWs is proposed by Krupke et al.<sup>72</sup> In this technique, an AC potential is applied between one Ag electrode and the p-type Si substrate, which are separated by a 600 nm thick thermal oxide, and the opposing electrode is floating. The capacitive coupling occurs between the floating electrode and ground. A potential difference between the driven and floating electrodes can be maintained up to a certain frequency, when the capacitive coupling resistance becomes comparable to the impedance of the AC power supply. When the deposition is performed around this frequency, the potential of the floating electrode is kept coupled to the ground until a nanotube bundle forms contact with a resistance comparable to the capacitive impedance. Since the field is now too weak to attract additional nanotube bundles, the particular



3 Examples of magnetic assembly. (a) Alignment of a segmented Ni/Bi/Ni NW on Ni electrodes. (reprinted with permission from Ref. 73, copyright 2005, American Chemical Society) (b) A segmented Pt/Ni/Pt NW trapped by Ni electrodes. (reprinted with permission from Ref. 74, copyright 2002, American Institute of Physics) (c) A multiwalled CNT with a Ni cap immobilised on Ni electrodes. (reprinted with permission from Ref. 75, copyright 2004, American Chemical Society) (d) Several Ni NWs with Au caps aligned across Ni electrodes (reprinted with permission from Ref. 76, copyright 2006, IEEE)

pair of electrodes is spanned by a single bundle. This process can take place independently on a series of electrodes.

#### Magnetic alignment

In addition to the electric field assisted assembly, magnetic fields are also utilised for alignment purposes thanks to the large shape anisotropy associated with NWs (Fig. 3). Magnetic assembly has advantages due to the fact that no specific surface chemistry is required for interaction and magnetic forces act irrespective of the medium.<sup>77</sup> In its most basic form, the alignment requires ferromagnetic NWs and an external magnetic field. For example, Ni NWs used by Tanase *et al.*<sup>74</sup> exhibited an increased level of alignment with increasing magnetic field. In the limit, the configuration consists of head-to-tail chains of NWs that run parallel to each other. Other studies report continuous change of the orientation of NWs following a rotating external magnetic field.<sup>78</sup>

Moving one step up in the hierarchy toward system integration, one arrives at the alignment of nanostructures with respect to simple magnetic electrodes. This is demonstrated by Tanase *et al.*<sup>74</sup> on Ni NWs trapped between elliptic Ni electrodes magnetised along their long axis in an external magnetic field. Electrode spacing is kept similar to NW length. To reduce contact resistance between the electrodes and NWs, Ni NWs are replaced with segmented NWs where only the tips consist of Pt.

Use of segmented NWs does not only reduce contact resistance but also brings about more freedom for tailoring magnetic interactions between nanostructures and ferromagnetic patterns on the substrate.73,76,78 Ni NWs with Au caps are utilised in an alignment study,<sup>76</sup> where NWs are again aligned along the field lines with Au segments ending up on Ni pads. Hangarter and Myung<sup>73</sup> employed NWs with Ni caps with a gold or bismuth midsection. Nanowires are aligned with respect to ferromagnetic contacts on the substrate under an externally applied magnetic field. Ni caps are observed to assemble at the edges of contact pads such that an NW with a length equal to the gap between two contact pads bridges the gap perfectly, whereas in the case of NWs longer than the gap, larger Ni cap is observed to align at the edge with the smaller Ni cap ending up on the top surface of the neighbouring contact. Furthermore, multiple alignment sessions with intermediate drying steps allow control of separation distance and adjustment of NW directionality by rotating the magnetic field at each session.

Imparting selective magnetisation along the length of the nanostructure irrespective of the main material is also used by Niyogi *et al.*<sup>75</sup> with CNTs. After CNTs are grown in a CVD chamber on a Si substrate using an Fe catalyst layer with an ammonia treatment, a Ni cap is deposited on their tips by thermal evaporation. Ni capped CNTs are dispersed in N,N-dimethylformamide and placed on Ni patterns on Si in an external magnetic



4 Examples of fluidic assembly. (*a*) Single-walled CNTs aligned across the width of PDMS channels. (reprinted with permission from Ref. 82, copyright 2002, American Chemical Society) (*b*) Bundles of [Mo<sub>3</sub>Se<sub>3</sub>]<sub>∞</sub> NWs aligned along the edges of PDMS channels on glass. (reprinted with permission from Ref. 83, copyright 2000, American Chemical Society) (*c*) and (*d*) Hierarchical assembly of InP NWs in (*c*) and GaP NWs in (*d*) with arrows showing subsequent flow directions. (reprinted with permission from Ref. 84, copyright 2001, The American Association for the Advancement of Science) (*e*) The bending of a trapped lipid nanotubule at the entrance of an array of microchannels (reprinted with permission from Ref. 85, copyright 2005, American Chemical Society)

field. After the dispersion dries up, CNTs are observed to assemble at the edges of Ni patterns along the field exhibiting a higher density around the corners. Alignment along the field lines is observed to occur as long as CNT concentrations do not exceed a critical value beyond which van der Waals interactions among CNTs overcome the magnetic effect leading to agglomerations. At low concentrations it is possible to bridge neighbouring Ni patterns with individual CNTs. Residual catalyst particles attached to CNTs can also be utilised for alignment purposes without additional deposition of ferromagnetic caps.<sup>79</sup>

In addition to NWs, thick films of oriented CNTs, also called buckypaper,<sup>80</sup> and alignment of biological molecules under strong magnetic fields<sup>81</sup> were reported in the literature.

#### Microfluidic assembly

Microfluidic assembly is yet another technique for aligning NWs and CNTs utilising flow fields in microchannels (Fig. 4). In principle, it is similar to other moulding techniques in microchannels such as MIMIC (micromoulding in capillaries)<sup>86</sup> with the exception that the geometric anisotropy of the one-dimensional nanostructures is the dominant factor in the alignment. Suspensions of NWs are either fed through microchannels where NWs are aligned along the flow direction or evaporation driven assembly, also called combing, is carried out where NWs trapped in evaporating fluid pockets within microchannels align themselves in a similar way. For example, alignment of gallium phosphite, indium phosphite and Si NWs in an ethanol suspension are demonstrated in microchannels in



5 Examples of Langmuir–Blodgett assembly. (a) The result of a two layer assembly followed by lithography and etching. (reprinted with permission from Ref. 100, copyright 2003, American Chemical Society) (b) The detailed view of an integration study with three electrodes and NWs. The inset shows the region where the NWs bridge two structures (reprinted with permission from Ref. 101, copyright 2004, American Chemical Society)

poly(dimethylsiloxane) (PDMS) moulds, where successive alignment sessions with different flow directions can lead to crossed arrays of NWs.<sup>84</sup> Nanowire density is observed to increase with flow duration, while angular deviations from flow direction are observed to decrease systematically with increasing flow rate. By chemically patterning the substrate, selective placement of NWs can also be achieved. Electrical contacts are subsequently added on top of the aligned structures.<sup>87</sup> Assembly in microchannels is similarly applied to biological structures that are one-dimensional, for example, microtubules,<sup>85,88</sup> with far reaching implications in molecular transport.<sup>89</sup>

In addition to the transport and deposition of nanoparticles,<sup>90</sup> evaporation driven self-assembly is also possible in microchannels where NWs confined to receding fluid pockets align themselves along the channel. Molecular wires of Mo<sub>3</sub>Se<sub>3</sub> are observed to form bundles in the form of NWs in microchannels when the solvent is allowed to evaporate.83,91 Resulting NWs are aligned along the edges of microchannels, where the remaining liquid recedes during evaporation. Nanowire width, i.e. the number of molecular chains forming the bundles, decreases with decreasing molecular concentration and microchannel volume and reaches the ultimate limit of a single molecular chain in the limit. Hierarchical assembly is again possible through subsequent deposition of NWs by rotating the microchannel. Driving a droplet containing a dilute suspension of Au NWs through a microchannel was reported by Salalha and Zussman with interesting observations on temporal evolution of NW alignment while crossing the triple phase line between air, suspension and substrate surface.<sup>9</sup>

Similar experiments on CNTs yielded alignment across the channel width instead of alignment along the channel edges.<sup>82</sup> This leads to suspended CNTs spanning the width of the microchannel.

In the absence of microchannels, liquid environment still serves as a very convenient medium for alignment, where less sophisticated alignment studies are conducted such as the attachment of bismuth telluride NWs on MEMS.<sup>93</sup> Utilising a simple rocking motion proves to be sufficient for bridging of electrodes with NWs. Similarly, one can mention the MEMS gas sensors,<sup>65,94</sup> where CNTs or tin dioxide nanobelts are attached to electrodes by spin coating of a suspension, or the intramolecular junctions<sup>95</sup> with CNTs spin coated in a similar fashion.

The efficiency of fluidic techniques can be increased by engineering the surface properties at the assembly site. When one defines the quality of alignment as the reduced deviation of nanostructures from perpendicularity along the triple phase line during combing, it was observed that combing of CNTs on a silica surface with a coating of CF<sub>3</sub> terminated silanes resulted in an enhanced alignment compared to coatings with functional groups of CH<sub>3</sub> or NH<sub>2</sub>.96 Similar treatments of electrode surfaces enhance the bridging rate of CNTs. Periodic surface functionalisation is also claimed to perturb the uniformity of the triple phase line and lead to microdroplet formation. Each microdroplet then serves as a medium for bending CNTs. This technique might then be manipulated to yield CNT curves with well defined radii.97 In the case of DNA combing, surface properties play an equally important role for the optimisation of binding. Fluidic alignment of DNA with its implications on NW fabrication will be discussed later in the section on 'Intergration through selective functionalisation'.

#### Langmuir-Blodgett technique

Derivatives of the Lagmuir–Blodgett technique are also applied to NWs which work similar to evaporation driven assembly where increasing level of confinement leads to alignment. Although high density, NW-like structures can be obtained when Langmuir films of nanocrystals are formed,<sup>99</sup> application of the technique directly to NWs is more promising (Fig. 5). In this technique, NWs trapped in a monolayer of surfactants on the surface of an aqueous solution are compressed by moving barriers while monitoring the surface pressure with a Wilhelmy plate. The resulting NW network at the water/air interface resembles a nematic structure with a long range directional order and positional disorder. The network is then transferred to a solid substrate using well established techniques such as Langmuir–Schäffer horizontal lift-off procedure.<sup>102,103</sup> In essence, this procedure resembles the transfer of CNTs from an oversaturated suspension on to an immersed hydrophilic substrate.<sup>104</sup> Subsequent transfers on to solid substrates can be carried out to obtain hierarchical structures such as layers of crossing NWs.<sup>100</sup> In addition to serving as an integration tool, the technique is also suitable for providing substrates for surface enhanced Raman spectroscopy.<sup>105</sup>

The dependence of the nature of the ordering on process parameters is the subject of intense study. For example, although a nematic structure is observed at low surface pressures, with further increasing pressure, a transition to smectic phase is obtained with BaCrO<sub>4</sub> NWs in isooctane on an aqueous subphase.<sup>102</sup> Increasing the surface pressure even further leads to a transition from the two-dimensional smectic configuration to a three-dimensional nematic structure with defects such as disclinations.

Ultrahigh density NW arrays can also be obtained by compressing NWs with a specific surface coating to form aggregates. After NWs are transferred to a substrate, surface coating can be selectively removed, hence, the spacing between deposited NWs becomes equal to the thickness of the sacrificial surface coating. This idea was demonstrated using Si NWs with a SiO<sub>2</sub> coating that is removed by HF etching or dry etching.<sup>106</sup> The well aligned Si NWs are then used as a lift-off mask leading to the formation of parallel lines of a second material of choice.

As a continuation of this work, parallel layers of high density p-type Si NWs are transferred on to a substrate with a predetermined average spacing.<sup>101</sup> Then this layer is patterned by photolithography into discrete groups. Patterning of NWs is followed by depositing complementary electrode arrays with a spacing comparable to the average spacing of the NWs. This eliminates the need for registry between the NWs and the electrodes and statistically leads to a high yield integration. Field effect transistors built for demonstration purposes exhibited reproducibility across the wafer and the observed behaviour was found to be consistent if the NW density is changed.

#### Integration through selective functionalisation

An alternative to aligning dispersed NWs and CNTs is to attract them toward lithographically defined, functionalised surfaces on microstructures (Fig. 6). Although the dimensions of the functionalised region are limited by the resolution of the lithographic technique used for patterning, functionalisation offers a promising parallel technique for multiple nanostructure attachment.

One of the earlier examples of this approach conducted on CNTs suspended in a N,N-dimethylformamide (DMF) solution<sup>107</sup> provides a good example to start with. In this work a hydrophobic trimethylsilyl (TMS) monolayer on SiO<sub>2</sub> surface is selectively removed with a linewidth of 10–50 nm using a high resolution lithographic technique. The exposed SiO<sub>2</sub> surface is then coated with 3-aminopropyltriethoxysilane creating amino-functionalised

lines within a TMS covered surface. Functionalisation step is followed by soaking the samples into the CNT/ DMF solution. Attracted toward aminofunctionalised surfaces, CNTs are observed to follow printed patterns closely with limited control on the number of attached CNTs on to the patterned lines. The technique was successfully applied to the mechanical testing of a rope of single walled CNTs suspended between two anchors.<sup>112</sup>

Another early example is by Burghard et al.<sup>113</sup> Multiwalled CNTs treated with sodium dodecylsulfate (SDS) are attached to gold electrodes coated with a selfassembled layer of long chain alkanethiol molecules. The surrounding SiO<sub>2</sub> surface is observed to remain intact. On the other hand, if the SiO<sub>2</sub> surface is treated with positively charged ammonium groups and Au electrodes are coated with negatively charged acidic groups, the opposite attachment tendency is obtained, i.e. CNTs selectively deposit on the SiO<sub>2</sub> surface. A similar study conducted on  $SiO_2$  with SDS as the surfactant is reported by Choi *et al.*, <sup>108</sup> where a 100 nm thick PMMA mask (e-beam lithography) is used to selectively deposit 1,2-aminopropyltriethoxysilane by CVD. With subsequent exposure to HCl vapour, NH<sub>2</sub> groups are converted to  $NH_3^+$ . Single walled CNTs suspended in a 1 wt-% solution of SDS are then observed to align themselves along the patterned lines similar to the previous case. Bridging of gold electrodes is also demonstrated in this technique. In another study with SDS, Rao et al.<sup>110</sup> conducted patterning with polar (amino and carboxyl) or non-polar (methyl) groups via dip pen lithography or microcontact stamping. It is reported that when the surface is exposed to a suspension of single walled CNTs in 1,2-dichlorobenzene, CNTs are attracted towards polar regions within 10 s. A yield of 70% over an area of 1  $\text{cm}^2$  is reported at single nanotube level, i.e. a single CNT is deposited to bridge densely packed patterns passivating its immediate neighbourhood. In a similar study, Lewenstein et al.<sup>114</sup> bridged prepatterned electrodes with single walled CNTs by defining funtionalisation sites via e-beam lithography and using a 0.5 vol.-% solution of aminopropyltriethoxysilane (APS) as the functionalisation medium. Covalent attachment of single walled CNTs to Si is also reported, where oligo (phenylene ethynylene) aryldiazonium salts are used as the functionalisation medium.<sup>115</sup>

Another study is conducted on the adsorption of negatively charged V<sub>2</sub>O<sub>5</sub> NWs on an electrostatically patterned substrate.<sup>109</sup> Patterning is achieved by tailoring end groups of self-assembled monolayers (SAM) to have positive or no charges such as amine and methyl groups, respectively. Placing the substrate in an NW solution leads to the attachment of NWs on to positively charged regions. If the pattern on the substrate becomes comparable to NW length, NWs are observed to exhibit a preference to span the negatively charged space between two positively charged patterns instead of spanning neutral regions. Hence, neutral patterns are used as passivation layers to prevent NW adhesion. Spatial control on NWs is again a function of the resolution with which the patterns are deposited, hence, a truly nanoscale control, such as placing a single NW at a desired location, is only possible with a sophisticated lithographic step.

Another electrostatically driven assembly process is reported by Chen *et al.*<sup>116</sup> for CNTs, which is at the



6 Examples of assembly via surface functionalisation. (a) A comparison of CH<sub>3</sub> (left) and NH<sub>2</sub> (right) functionalised SiO<sub>2</sub> surface in term of single walled CNT attachment. The observation has direct implications in surface patterning via self assembled monolayers. (reprinted with permission from Ref. 107, copyright 1999, Elsevier) (b) A similar patterning study where sodium dodecylsulphate treated CNTs are assembled on 1,2-aminopropyltriethoxysilane coated regions appearing bright on the micrograph. (reprinted with permission from Ref. 108, copyright 2000, Elsevier) (c) Single V<sub>2</sub>O<sub>5</sub> NWs assembled on cysteamine islands on a 1-octadecanethiol-passivated surface. (reprinted with permission from Ref. 109, copyright 2005, Wiley-VCH Verlag GmbH & Co. KGaA) (d) 30 × 30 μm area with single CNTs attached on 2-mercaptoimidazole islands on a 1-octadecanethiol passivated surface. (reprinted with permission from Ref. 110, copyright 2003, Nature Publishing Group) (e) Hierarchical contact printing of DNA on Si surface. (reprinted with single stranded DNA on a surface patterned with the complementary DNA (reprinted with permission from Ref. 11, copyright 2002, Wiley-VCH Verlag GmbH & Co. KGaA)

same time utilising dielectrophoresis. Carbon nanotubes are terminated with negatively charged carboxyl groups due to the oxidative shortening by acid. On the other hand, gold electrodes on Si are prepared and coated with a SAM terminating with a positively charged amino group. The interaction between the two oppositely charged structures leads to a vertical assembly of CNTs on the gold surface. However, when the same process is repeated in a DC electric field (1000 V cm<sup>-1</sup>), an increase in the coverage and assembling rate of CNTs on the anode is observed. This increase is also accompanied by a reduction in the number of longer CNTs landing on the substrate horizontally.

The use of fluidic platforms in combination with functionalisation is not uncommon. Carbon nanotubes with a hydrophilic coating are assembled on a substrate with hydrophobic and hydrophilic patterns.<sup>117</sup> During drying of the suspension, the alignment takes place on hydrophilic regions along the receding water-substrate-air triple line with respect to which CNTs have parallel orientations. Any change in the curvature of this line is strictly followed by nanotubes similar to a previous study by Gerdes *et al.*<sup>96</sup> as discussed previously in the section on 'Microfluidic assembly'. Similar techniques are utilised for three-dimensional assembly, too.<sup>118</sup>

Biological templates are also utilised for either growing NWs on microstructures or assembling NWs with a predetermined order. Especially the high level of selectivity and reversibility of interactions among complementary sequences of DNA provide a very attractive means of functionalisation. Although most of the following techniques utilise external fields such as fluidics and electric fields for the alignment of DNA molecules, it is more appropriate to list them in this section due to the dominance of functionalisation when it comes to anchoring, i.e. attachment of the molecule to the microstructure. In most cases, anchoring is carried out using sticky ends, single stranded oligonucleotides that are placed on the site of integration with their complementary strands protruding from the ends of double stranded DNA in a solution. The two combine through hydrogen bonding. Implementation of this approach dates back to early 1970s and provides a very diverse selection for functionalisation.<sup>119</sup>

Before proceeding further with specific examples, let us also emphasise that in length scales on the order of 1– 10  $\mu$ m, which are of interest for integration studies, DNA is a highly flexible medium to work with. In fact, having a persistence length of 50 nm or more,<sup>120</sup> the molecule exhibits considerable bending stiffness only along its two or three turns. Hence, alignment under external fields deviates from the nature of the alignment processes discussed before. The task becomes even more difficult for single stranded DNA.<sup>121</sup> Major manipulation and alignment techniques include viscous drag, electrophoresis, magnetic and optical tweezers and molecular combing.<sup>122–124</sup>

As the first example, one can consider the integration process carried out by bridging two gold electrodes that are 12–16 µm apart with DNA molecules and subsequently using the molecules as templates for NW growth.<sup>34</sup> The first step involves the functionalisation of the surfaces of both gold electrodes with different 12 base oligonucleotides facilitated by sulphur–gold interactions at the 3' end containing a disulphide group.

Functionalisation is followed by the hybridisation of the oligonucleotides with a 16  $\mu$ m long  $\lambda$ -DNA containing complementary 12 base sticky ends. Free DNA molecules are stretched using a flow field that leads both sticky ends to contact electrodes and form bonds. After bridging of the electrodes is accomplished, silver ions are placed along the DNA by Na<sup>+</sup>/Ag<sup>+</sup> ion exchange followed by the reduction of the silver ion - DNA complex in a basic hydroquinone solution forming silver aggregates. The resulting silver NW is further developed in an acidic solution of hydroquinone and silver ions leading to selective deposition of silver on the existing NW. Recent examples of the silver deposition technique exhibit NW diameters around 15 nm.125 DNA alignment via molecular combing and microcontact printing is also shown to enhance the control of the placement of DNA NW templates on Si,<sup>111</sup> which is crucial for subsequent integration with microstructures.

In addition to silver, other metals including gold,<sup>126,127</sup> platinum,<sup>128–130</sup> palladium<sup>131,132</sup> and copper<sup>133</sup> can be deposited on DNA. DNA templates are also utilised to fabricate clusters of semiconductor nanoparticles<sup>134</sup> and conducting polymeric NWs such as polyaniline.<sup>135</sup> Furthermore, deposition along the DNA molecule can be carried out leaving DNA segments exposed at specific sites for further localisation of molecular objects.<sup>136</sup> A brief comparison of these studies is supplied in Table 1.

It should be emphasised that the success of the integration techniques relies on the success of the initial DNA alignment step, which is independent of the chosen NW material. As a side note and in addition to the aforementioned techniques, the polarisation and alignment of double stranded DNA in an electric field should also be mentioned,<sup>137</sup> where a transition from coiled to a rod-like conformation takes place. During the alignment study, a very strong binding between DNA ends and aluminium electrodes are observed. The strength of this contact is reported to be high enough to withstand strong flows in the medium where the molecule breaks along its backbone rather than at its ends supplying an alternative means of anchoring to the functionalisation studies.<sup>138</sup> The technique is later extended to align single stranded DNA.139 Formation of single strands is based on the polymerase chain reaction, and the basic difficulty of preventing backpairing, i.e. the formation of a double stranded DNA after the strands are separated, is achieved by using a biotinylated primer during polymerase chain reaction, which then attaches to avidin labelled microparticles. After separating both strands in a NaOH solution, the unattached strand is easily removed from the system. Keeping the distance between Al electrodes equal to the stretched length of the DNA, formation of single stranded DNA bridges anchored at both of their ends is observed.

Alternatively, DNA can be used as a smart glue instead of a template. For example, Au NWs can be functionalised with single stranded DNA and attached to Au surfaces or other NWs coated with the complementary DNA strand. Mbindyo *et al.*<sup>140</sup> demonstrated a fourfold increase in surface coverage when complementary strands interact as opposed to nonspecific adhesion in the case of non-complementary strands. The difference in surface coverage is observed

Nanowire material/ nanowire diameter	DNA type	Alignment method	Anchoring method	Electrode/ substrate	Electrode spacing	Process steps
Ag 100 nm (Ref. 34)	λ	Fluidic (micropipette suction)	Sticky ends	Au Glass	12–16 μm	<ul> <li>(i) localisation of Ag<sup>+</sup></li> <li>(ion exchange)</li> <li>(ii) formation of Ag aggregates</li> <li>(reduction)</li> <li>(iii) formation of nanowire</li> <li>(development in an acidic solution of hydroquinone silver ions)</li> </ul>
Au 100 nm (Ref. 136)	λ	N/A	N/A	Au Passivated Si	2 μm	<ul> <li>(i) reduction of Ag<sup>+</sup> in AgNO<sub>3</sub></li> <li>by DNA bound aldehyde</li> <li>(ii) electroless Au deposition</li> <li>(iii) Au deposition sites are patterned along the molecule by using homologous recombination with RecA as a masking technique</li> </ul>
Au 20–40 nm (Ref. 127)	double str. calf thymus	Spinning of DNA solution (no preferential alignment)	N/A	Au Thermal oxide	0·2–1·0 μm	<ul><li>(i) treatment in 4-(dimethylamino) pyridine modified Au nanoparticle disper.</li><li>ii) electroless gold deposition</li></ul>
Au (with Pt seed layer) sub-20 nm (Ref. 128)	double str. calf thymus	N/A	N/A	N/A Si	N/A	<ul> <li>(i) platination of DNA with dichloro(2,2':6',2"-terpyridine) platinum(II) or <i>cis</i>-diamminedichloroplatinum(II)</li> <li>(ii) reduction of Pt<sup>2+</sup> in sodium borohydride</li> <li>(iii) electroless Au plating</li> </ul>
Pt 5 nm (Ref. 129)	λ	N/A Samples are deposited on TEM grids	N/A	N/A	N/A	<ul> <li>(i) platination (binding of [PtCl<sub>4</sub>]<sup>2-</sup>) of DNA in K<sub>2</sub>PtCl<sub>4</sub></li> <li>(ii) Reduction in dimethylammine borane</li> </ul>
Pd 20 nm (Ref. 131)	λ	Molecular combing	Amorphous carbon deposition	Au N/A	5–10 μm	(i) activation of DNA in Pd(CH <sub>3</sub> COO) <sub>2</sub> solution (ii) reduction of Pd(II) in a solution of sodium citrate, lactic acid and dimethylamine borane
Cu sub-20 nm (Ref. 133)	λ	No alignment w.r.t. electrodes Molecules extended over an electrostatically charged surface.	N/A	N/A Si	N/A	(i) treatment in aqueous $Cu(NO_3)$ to transfer of $Cu(II)$ ions (ii) reduction of $Cu(II)$
CuS 10 nm (Ref. 134)	λ	Spinning of DNA solution or molecular combing (No preferential alignment)	N/A	N/A Mica	N/A	<ul> <li>(i) treatment in concentrated CuCl<sub>2</sub> solution (to exchange sodium counterions in DNA with copper ions)</li> <li>(ii) thorough rinsing with water</li> <li>(iii) treatment in dilute CuCl<sub>2</sub> solution while bubbling H<sub>2</sub>S</li> </ul>
Polyaniline N/A (Ref. 135)	λ	Fluidic (Alignment normal to the receding water/air/substrate line)	N/A	Au Thermal oxide	1·5 μm	<ul> <li>(i) treatment in protonated aniline solution to organise aniline monomers along the molecule</li> <li>(ii) enzymatic polymerisation with horseradish peroxidase and hydrogen peroxide.</li> </ul>

#### Table 1 Comparison of nanowire fabrication techniques using DNA as template



7 Assembly attempts on original fabrication sites. (a) MoO<sub>x</sub> NWs obtained by step edge decoration on graphite surface (electroplating). (reprinted with permission from Ref. 35, copyright 2000, The American Association for the Advancement of Science) (b) Electroplating of Pd NWs on graphite. Their use in a hydrogen sensor was demonstrated in Ref. 143. (reprinted with permission from Ref. 143, copyright 2002, American Chemical Society) (c) Single walled CNTs grown through CVD on single crystalline quartz substrate with a density of more than 10 CNTs per μm<sup>2</sup>. Their use in a thin film transistor was demonstrated in Ref. 144. (reprinted with permission from Ref. 144, copyright 2005, Wiley-VCH Verlag GmbH & Co. KGaA) (d) Vertical CNTs grown in an anodic alumina template. Their use in a surround gated vertical field effect transistor was demonstrated in Ref. 145. (reprinted with permission from Ref. 145, copyright 2004, Institute of Physics) (e) Single walled CNTs grown on an r-plane sapphire. (reprinted with permission from Ref. 146, copyright 2005, American Chemical Society) (f) An example of a registration free assembly, where patterning of anchors is carried according to the distribution of CNTs on the substrate surface. (reprinted with permission from Ref. 147, copyright 2003, Nature Publishing Group) Device elements patterned on top of CNTs are shown in the inset (reprinted with permission from Ref. 148, copyright 2003, American Institute of Physics)

to increase with increasing length of the DNA strands. Furthermore, selective functionalisation of Au NWs along their length offers possibilities for assembly in three dimensions. The functionalisation can either be carried out only at the tip of the NWs utilising the fact that NW ends are easily exposed in template assisted growth or segmented NWs, e.g. Au/Pt/Au, can be fabricated within the template leading to NWs that are coated with DNA only on Au regions.<sup>140</sup> Similar studies are reported for CNTs, where the critical issue becomes the creation of end groups to serve as functionalisation sites.<sup>141</sup> These interactions do not have to involve DNA hybridisation only. Antibody/antigen, ligand/receptor reactions can also be utilised as well, as demonstrated for the assembly of zero-dimensional structures, such as polystyrene beads and gold nanoparticles.<sup>142</sup>

#### Integration without subsequent alignment

Depending on the nature of NW and CNT fabrication method or the nature of the application, some integration work can also be carried out without removing selfassembly products from their fabrication sites (Fig. 7). Either the fabrication method delivers a reasonably good alignment of the nanostructures or a network rather than an aligned set of nanostructures suffices for a specific application, where registry with the microstructure is the central issue.

Step edge decoration is one of the techniques delivering a set of nanostructures aligned among themselves. The technique is based on the arrest of adatom diffusion on a surface by irregularities. The resulting NWs already have a reasonable amount of alignment at their original fabrication sites, for example, step edges on a cleaved graphite surface. Although the amount of alignment, i.e. the distribution of nucleation sites, is better than that offered by the aforementioned class of self-assembly techniques and hence is suitable for fabricating devices for demonstrative/prototyping purposes, the technique is far away from supplying a batch compatible solution. The method facilitates the deposition of electrical contacts on top of the NWs without the application of any external field for alignment. Although NWs on a variety of substrates have been fabricated with this technique,<sup>35,149</sup> one early example of device integration is a gas sensor made of Pd NWs.<sup>143,150</sup> The NWs were electrodeposited along the step edges on freshly cleaved graphite surfaces. They were then transferred on to a glass substrate coated with cyanoacrylate and silver paint was used to form simple electrodes to monitor changes in the current through the NWs upon exposure to hydrogen.

Combined with lithography, crystalline surfaces provide a better control for the creation of nucleation sites as demonstrated on  $\{111\}$  Si surfaces.<sup>151</sup>  $\{111\}$  Si surface is known to be atomically flat, and it exhibits a well defined step/terrace structure. For example, regular arrangements of atomic steps can be achieved by using etched holes on the surface. When the surface is heated at high temperatures in vacuum, the step motion due to adatom evaporation is pinned at the periphery of the hole. Further heating leads to the formation of a step bunch and finally, the hole, becoming shallower with step retreat, disappaears completely, and regularly ordered step bunches form. Combined with heteroepitaxy, this technique can result in Ge nanostructures on Si.<sup>151</sup>

Another mechanism is observed to be active on the aplane of sapphire while growing ZnO NWs.152 Rather than taking advantage of step edges, this growth technique resembles directional growth, which will be explained in the next section, with one major difference: the growth is confined to the plane of the substrate and the growth direction is dictated by the crystalline anisotropy of the saphhire. If a nanoscale catalyst particle, usually Au, is supersaturated with Zn and O atoms, a ZnO NW grows. If the size of the catalyst Au particle is kept below 20 nm, ZnO NWs grow along [1100] direction in a typical CVD setting. In this direction there is a close match between the crystal structures of both the sapphire substrate and ZnO NWs. The growth is similar to the tip growth mechanism with the Au particle carried along the sapphire surface at the tip of the growing NW. If Au particles coalesce, this inplane growth is observed to be replaced by the out-ofplane growth as explained later. Resulting NWs are reported to have diameters as low as 2 nm. The periodicity of the NWs is determined by the patterning technique of Au nanoparticles, and hence, similar to step edge decoration, this technique also falls short of meeting the demands of rational assembly. Use of crystallographic orientations also provides another challenge for alignment and integration.

CNT growth on single crystalline sapphire substrates is also reported,  $^{146,153}$  where a- and r-planes yield the best alignment and provide the possibility of hierarchical assembly.<sup>146</sup> Rather than step edge decoration, the reason for alignment is argued to be the specific interactions between surface atoms and CNTs.<sup>146</sup> A similar conclusion was reached previously in a study on CNTs grown on <100> and <111> Si surfaces,154 where distinct growth directions were observed on both planes that could be verified by molecular simulations. The spatial density of CNTs is reported to be a function of catalyst concentration.<sup>155</sup> Further control on CNT orientation and position is recently demonstrated via nanosphere lithography.<sup>156</sup> On the other hand the alignment work on quartz crystals suggests step edge decoration where CNT alignment follows alignment of surface steps.<sup>144</sup> Some of the reported device applications include field effect transistors<sup>155</sup> and thin film transistors.144

Similarly, anodic alumina templates lead to a high spatial density and perfect alignment among NWs not within the surface of the substrate but along the normal direction of the template. A thermoelectric microgenerator proposed by Wang et al.<sup>157</sup> utilises this unique feature. In this study, arrays of n-type and p-type Bi<sub>2</sub>Te<sub>3</sub> NWs are patterned into micro arrays and sandwiched between electrodes and thermally conductive layers. Patterning of the NWs is carried out by consecutive deposition of n-type and p-type NW material into nanopores by selective masking. NWs separated by insulating alumina walls are then electrically connected by a second lithographic step on both surfaces of the template, which is followed by the deposition of Si on both sides as the thermally conductive layer. Although the characterisation of the microgenerator is not supplied in the report, this constitutes an interesting approach to microsystem fabrication with the nanoscale template serving as the substrate. A similar approach is also utilised for CNTs to fabricate vertical field effect transistors,<sup>145,158</sup> where CNTs were catalytically grown inside the pores of an anodic alumina membrane and integrated with electrical contacts through subsequent lithographic steps.

Up to this point, we have covered some techniques where nanostructures have a certain alignment due to either the application of an external field or the nature of the nanofabrication method. An alternative technique is introduced by Bockrath *et al.*<sup>159</sup> for single walled CNTs and then utilised in various studies<sup>160–164</sup> where CNTs have no alignment at all. Their suspension is deposited on to a substrate where coordinate markers were previously patterned. The position and orientation of each CNT or CNT rope with respect to the markers are recorded followed by the fabrication of electrical contacts through lithography and evaporation. Although highly serial and impractical for mass fabrication purposes, the technique eliminates the need for alignment for the fabrication of laboratory specimens for transport property measurements such as electrical measurements on PECVD grown multiwalled CNTs<sup>162</sup> and measurements of the response of single walled CNTs to mechanical stimulus.<sup>163,164</sup> In a remarkable series of studies on nanoelectromechanical system integration, the same technique was employed to align a multiwalled CNT that serves as a support shaft for a rotor plate.147,148

And finally, in some instances, a two-dimensional, blanket deposited network of nanostructures with no alignment would suffice as it is the case with some sensor applications. The collective behaviour of the network becomes the key issue regarding the operation of the device. The microfluidic patterning approach adapted by Fu and Liu represents an example.<sup>165</sup> A planar network of single walled CNTs is coated with a 40 nm thick gold film. Gold is patterned in the form of thin lines and CNT's not covered by the gold coating are removed from the surface. Then microchannels with three merging inlets are formed using PDMS such that they run perpendicular to the gold lines. The two outer inlets are used to feed water, whereas the middle inlet serves as the gold etchant supplier. Depending on the flow rates of water and etchant, a gap is formed in the gold lines bridged by the exposed network of CNTs. The same microfluidic structure can then be used to supply analytes to the sensor.

#### **Directional growth**

Directional growth involves synthesis of one-dimensional features at desired sites on a microstructure through catalytic reactions. Vapour-liquid-solid growth technique is among the most widely used techniques for NWs based on the epitaxial growth of the structure on a lattice matched substrate.<sup>166</sup> In this process, metal nanoparticles serve as a catalytic medium for the formation of a metal/semiconductor alloy above its eutectic temperature with a vapour phase semiconductor source. With further transfer of the semiconductor, the alloy droplet supersaturates, and the semiconductor precipitates in the form of an NW, whose size is dictated by the diameter of the nanoparticle. With proper selection of the substrate orientation, the growth will occur perpendicular to the substrate. The NW integrated substrate then serves as a starting point for device fabrication. Usually a CVD setting is required for the feeding of the semiconductor. Carbon nanotubes are

also grown in a directional fashion using CVD, either thermal or plasma assisted. Plasma assisted CVD produces better directionality, while obtaining single walled CNTs becomes more challenging compared to thermal CVD.<sup>167</sup> The ability to define integration sites lithographically and good adhesion between micro- and nanostructures are some of the major advantages of this approach. However, the demanding requirements of thermal and/or chemical resistance of the growth platform limit the choice of integrated devices. In this section, various growth techniques for both NWs and CNTs will be discussed (Figs. 8 and 9). After some of the reported applications are reviewed, the section will be concluded with a discussion on the limiting scenario of growing a single nanostructure.

#### Growth studies

Single-terminal epitaxial growth studies with relaxed requirements on NW spatial density but with decent directionality are reported including single crystalline NWs of Si,<sup>179</sup> Ge<sup>180</sup> and III–V semiconductors<sup>181</sup> among others. Similar growth studies are carried out on CNTs using mainly the CVD technique.<sup>182,183</sup> Selective growth can be achieved when the VLS<sup>176</sup> or CVD<sup>184</sup> techniques are combined with lithography.

The first demonstration of a two terminal NW growth was provided by Islam et al.,<sup>168</sup> where the growth of Si NWs bridging the vertical {111} walls of a trench etched in a {110} oriented Si substrate was studied. NWs are observed to grow epitaxially along <111> directions. The initiation sites of NWs are dictated by either Ti or Au spots deposited on one of the {111} walls through evaporation. The samples were annealed in hydrogen at  $\sim 625^{\circ}$ C leading to the formation of Au–Si or TiSi<sub>2</sub> nanoparticles. The temperature was then increased to 635°C and the samples were exposed to a mixture of SiH<sub>4</sub> and HCl to grow the NWs, where Si adatoms from silane diffuse through the nanoparticles and precipitate on the underlying Si substrate. Continued precipitation leads to the epitaxial growth of a Si nanowhisker, whose diameter is determined by the size of the nanoparticle. In the case of a gold catalyst, NWs are observed to have a diameter of  $180 \pm 20$  nm with an average length of ~8  $\mu$ m. For trenches with a width <8  $\mu$ m, the NW impinges almost perpendicularly on the opposite Si wall leading to the spreading of the catalytic Au-Si droplet. This spreading leads to further deposition of an anchor in the form of a disc. In the case of Ti catalyst, on the other hand, the catalytic TiSi<sub>2</sub> retains its solidity and hence, no disc formation is observed. The bond between the NW and the opposite Si wall is observed to be a Si-Si bond leading to a two terminal NW.<sup>185</sup>

The requirement for a metallic catalyst can also be lifted as reported by Conley *et al.*<sup>186</sup> where ZnO NWs are grown using a ZnO thin film as the seed layer. Furthermore, similar to the above study, it is demonstrated that ZnO nanobridges spanning two structures can be formed in this way. For this purpose, undercut Si islands on SOI wafers or trenches etched in Si layer on an SOI wafer are used along with the atomic layer deposition of a ZnO seed layer.

Another approach for NWs and also for CNTs was proposed by Englander *et al.*<sup>187</sup> where suspended Si microstructures were selectively heated by passing an electric current. After a maskless evaporation of a 5 nm thick catalyst layer (Au–Pd or Ni–Fe), microstructures



8 Examples of in-plane alignment using directed growth. (a) A Si NW grown inside a trench of {111} Si side walls. Ti was used as catalyst. The arrow indicates the growth direction. (reprinted with permission from Ref. 168, copyright 2004, Institute of Physics) (b) Single walled CNTs bridging Si pillars. (reprinted with permission from Ref. 169, copyright 2000, Wiley-VCH Verlag GmbH & Co. KGaA) (c) Directed growth of Si NWs in an electric field. (reprinted with permission from Ref. 170, copyright 2005, American Chemical Society) (d) Growth of CNTs between two Mo electrodes in the absence (upper micrograph) and in the presence (lower micrograph) of an electric field. (reprinted with permission from Ref. 171, copyright 2002, American Institute of Physics) (e) A suspended single walled CNT between a Si cantilever and an anchor. (reprinted with permission from Ref. 172, copyright 2002, American Chemical Society) (f) Suspended CNTs between the grips of a MEMS test platform (reprinted with permission from Ref. 173, copyright 2006, Elsevier)

were exposed to  $SiH_4$  or  $C_2H_4$  to grow Si NWs or CNTs respectively. Growth mechanism is similar to the one explained above. It is based on the catalyst layer breaking down into nanoparticles under the effect of heating. After each nanoparticle is saturated with the decomposed element of interest from the feedgas, precipitation at the solid/liquid interface leads to a one-dimensional growth. Nanostructures were observed to grow selectively on suspended structures. The nonuniformity regarding their size and growth rate is found to be a direct indication of temperature distribution along the suspended structures. Growth is observed to initiate preferentially at those locations where a sufficiently high temperature is obtained. Nevertheless, local



9 Examples of epitaxially aligned, out-of-plane growth. (a) ZnO NWs grown from Au catalyst islands on Si terminated SiC epilayer. (reprinted with permission from Ref. 174, copyright 2004, American Chemical Society) (b) In<sub>2</sub>O<sub>3</sub> NWs grown on a-sapphire substrate. Catalytic Au particles at NW tips are visible in the inset. (reprinted with permission from Ref. 175, copyright 2004, American Chemical Society) (c) Si NWs on {111} Si substrate. (reprinted with permission from Ref. 176, copyright 2005, American Chemical Society) (d) Same conditions as (c) with catalyst patterning. (reprinted with permission from Ref. 176, copyright 2005, American Chemical Society) (d) Same conditions as (c) with catalyst patterning. (reprinted with permission from Ref. 176, copyright 2005, American Chemical Society) (e) A single walled CNT growing from a catalyst nanoparticle. (reprinted with permission from Ref. 177, copyright 2002, Springer) (f) Blocks of CNTs growing from Fe catalyst islands (reprinted with permission from Ref. 178, copyright 1999, The American Association for the Advancement of Science)

heating facilitates NW integration with microsystems, since the rest of the device is now protected against high temperatures.

Further improvement in directionality can be achieved by growing NWs within an electric field utilising their high anisotropy in polarisability.<sup>170</sup> In addition to the suspended structures on which NW growth is initiated, secondary structures, situated 5 to 10  $\mu$ m away from the growth structures, are now employed to create a local DC electric field. Intrinsic Si NWs with a relatively low charge carrier

concentration are observed to align themselves according to the electric fields and the degree of alignment is enhanced at higher electric field strengths. Bending of NWs along fringe field lines or preferred initiation of growth near sharp corners is evident. A similar growth study on Cr NWs is also reported.<sup>188</sup>

A similar technique utilising local heating and electric fields is also proposed for CNTs,<sup>189</sup> with the first demonstrations under electric fields carried out in 2001.<sup>190,191</sup> These early studies utilised parallel electrodes between which a DC field is applied. For single

walled CNTs<sup>191</sup> lower electric field strengths are observed not to be sufficient to suppress randomisation of CNT orientation during growth. In addition to thermal vibrations, build-up of van der Waals forces due to interaction with nearby surfaces is a contributing factor to the randomisation. This is usually avoided by bridging the electrodes away from the substrate surface. However, it is observed that thermal oxide or quartz surfaces provide a growth platform with reduced interaction, where CNTs can make contact and still grow along the electric field lines.<sup>171,191</sup> In addition to a sufficient electric field, it is still important that CNTs grow to a certain length before they land on the substrate surface for further growth.<sup>171</sup> Both parameters are important in the determination of the alignment torque acting on the CNT, and hence, for overriding the randomisation effect. More in-plane alignment studies will be discussed later in this section.

A higher precision for positioning the CNTs is obtained when the intensity of the electric field is increased by utilising sharp Si pyramids.<sup>192</sup> A tetramethyl ammonium hydroxide (TMAH) based etching step is used to anisotropically etch Si and create the pyramids. This is followed by thermal oxidation and catalyst layer (iron and cobalt) deposition. During the CVD process in ethanol vapour, an electric field is applied between the pyramids and a nearby plate electrode. The presence of an electric field is reported to increase the number of pyramids with a CNT at the tip and also the number of nanotubes that grow in a straight fashion.

Out-of-plane growth of single walled CNTs is usually obtained through patterning of the growth site by simple lithography and lift-off followed by the catalytic growth with CVD using gases such as methane as the carbon source.<sup>21</sup> The growth in the presence of methane is observed to be very sensitive to  $H_2$  flow.<sup>193</sup> A careful study of the growth mechanism was carried out by preparing discrete catalytic nanoparticles (Fe<sub>2</sub>O<sub>3</sub>) of tunable diameters and growing isolated CNTs from them in methane and hydrogen at 900°C.194 It was concluded that the growth mechanism can be explained by the base growth model. The growing end of the CNT remains attached to the substrate together with the catalytic particle. During growth, carbon atoms are absorbed into the particle and form a solution. Once the solution is supersaturated, carbon precipitation leads to a single walled CNT growing out of the surface. The other end of the CNT is dome closed. Tip growth mechanism is also reported for single walled CNTs in CVD with fast heating conditions.<sup>195</sup> Smaller particles (1-2 nm) are observed to be better in producing single walled CNTs, whereas bigger particles ( $\sim 7 \text{ nm}$ ) produce no single walled CNTS. The diameters of the resulting CNTs are closely related to the size of the nanoparticles.<sup>177</sup> Using a porous substrate, such as porous silica, enhances the diffusion of the feedgas to the catalytic particle and leads to higher growth rates.<sup>178,196</sup> Among the applications reported one can mention various sensors including NO<sub>2</sub> detectors,<sup>197</sup> field emitters,<sup>198–200</sup> field effect transistors,<sup>174,201</sup> energy storage media<sup>202</sup> and AFM tips.<sup>203</sup>

Dating back to 1998,<sup>204–206</sup> PECVD is established as a technique to further increase the directionality and reduce the process temperature. Various PECVD techniques available for CNT growth are listed in Ref. 207. In the process, acetylene is used as the carbon source and ammonia as the etching gas for the Ni catalyst. Additions to the technique including the formation of curly segments<sup>208</sup> and elimination of amorphous carbon from the surface through the optimisation acetylene/ammonia ratio<sup>209</sup> followed. An example of PECVD with better control on the number of CNTs as compared to CVD is the field emission source with an integrated poly-Si gate electrode,<sup>210</sup> where CNTs are grown vertically on Ni islands inside the microcathodes. Resulting devices exhibit stable emission characteristics after an initial shift in the turn-on voltage.

As far as in-plane alignment studies of single walled CNTs are concerned, first demonstrations were reported by Dai group. In addition to alignment enhancement, obtaining long single walled CNTs was one of the early challenges. An example is the bridging of Si towers with their tops exposed to catalysts.<sup>211</sup> This was a purely geometric approach with no external fields applied. Any CNT that would not reach a neighbouring tower would fall down and stick to the sidewalls of the tower, while those who could reach a neighbouring tower would survive and assume the shape of a suspended bridge. The yield associated with the process was later enhanced by placing a bulk amount of conditioning catalyst upstream of the growth platform.<sup>169</sup> Another early example is the bridging of catalytic islands on SiO<sub>2</sub> defined by e-beam lithography.<sup>212</sup> Other examples of inplane CVD growth are reported on a variety of structures such a pair of electrodes facing each other,<sup>213</sup> a specific part of MEMS<sup>173</sup> or inside vias.<sup>214</sup> Among the prototypes reported one can mention the single electron memory, <sup>215</sup> field effect transistor, <sup>203</sup> NO<sub>2</sub> and NH<sub>3</sub> detectors, <sup>216,217</sup> biological sensors<sup>218,219</sup> in addition to electrode CNT set-ups for transport property measurements.<sup>220</sup>

Although there is no control on the number of resulting CNTs, Franklin et al.<sup>213</sup> reported a yield of 30% over 4 inch substrates, where opposing Mo electrodes are bridged by a single SWCNT. In addition to Mo, Ta, W, Au, Cu, Co, TaN and TiN are reported as electrode materials if coated by a very thin Al film acting as the catalyst support.<sup>221</sup> The study with Mo electrode <sup>213</sup> utilises alumina supported iron catalyst in a methane and hydrogen environment at 900°C. Grown nanotubes are observed to have lengths of 3-10 µm. If one of the electrodes is patterned on a cantilever with the opposing electrode located on a fixed structure, electrical characterisation of the CNT can also be carried out under mechanical loading by bending the cantilever. Catalyst thickness is also reported to be a parameter determining the number of resulting CNT bundles in hot filament CVD.<sup>203</sup> The randomness associated with the process can be reduced by growing the CNTs in an electric field on the order of  $1 \text{ V} \mu \text{m}^{-1}$  as discussed previously.171

Alternatively, inductive heating is also proposed for catalytic nanotube growth with higher growth rates allowing smaller reaction chambers.<sup>167,222</sup>

Instead of growing CNTs from a catalyst surface, a two terminal approach is also proposed utilising a multilayered catalyst structure that leads to low contact resistance and high adhesion strength<sup>223</sup> similar to two terminal NWs by Islam *et al.*<sup>168</sup> A catalytically active, 2 nm thick iron layer is sandwiched between two Al layers on both sides, which are again sandwiched by Ni layers. The substrate is a p-type Si and the whole structure is capped with a Cu layer. It is suggested that during preheating of the structure in hydrogen plasma, a Fe–Al compound layer forms which is then bombarded by various CH including free radicals when the plasma is switched to a methane plasma. Radicals are also absorbed by the Ni layers. When the radicals recombine on metal surfaces, the corresponding bond energy is released which heats the catalyst droplets. It is argued that a eutectic C–Fe alloy is formed within the droplets leading to CNT growth toward upper and lower Ni plates, thereby lifting the Cu cap.

Another technique that can be classified as directed growth is proposed by Cheng and Haynie,<sup>224</sup> where Pd NWs are grown at the sharp tips of Au electrodes in an aqueous solution of Pd acetate. NWs form upon the application of an alternating electric field and are observed to follow the field lines. The growth is also observed between parallel electrodes.<sup>225</sup> Similarly, indium NWs are obtained in an aqueous solution of indium acetate by applying an alternating voltage between two electrodes with sharp tips.<sup>2</sup> Solidification starts at the tip of the electrode with the alternating potential, where the reduction rate of cations is elevated.<sup>227</sup> Nanowires are observed to grow form there towards the grounded electrode. A concentration versus voltage window is established over which the dendritic growth is suppressed and linear wires are obtained.<sup>226</sup> The frequency of the alternating voltage is reported to be the main parameter controlling the NW diameter.<sup>227</sup> The same electrochemical growth mechanism is also demonstrated between conductive particles polarised in an external electric field, where no physical connection between the growth platform and the power source is necessary.<sup>228</sup> However, in a crowded device configuration with multiple contact points this approach would be difficult to implement. Finally, the growth of NWs using field emission in the presence of an organometallic precursor can be considered as the 'dry' version of the aforementioned technique.<sup>229</sup> The growth takes place inside the chamber of an environmental SEM, where precursor gas molecules introduced through a nozzle are dissociated by emitted electrons at a sharp tip. Using this technique a tungsten NW is grown at the tip of a CNT. The path of the growing NW can be dictated by the motion of an externally driven anode.230

#### Applications

Among the self-assembly techniques directional growth is the most widely utilised one in various device applications (Fig. 10). One major advantage is, of course, the combination of fabrication and assembly stages with the capability of defining growth sites through lithography. This opens up new possibilities including the integration of CNTs with standard Si MOS technology.<sup>231</sup>

A direct application of directed growth to device fabrication is the vertical field effect transistor reported by Nguyen *et al.*,<sup>175</sup> where  $In_2O_3$  NWs are epitaxially grown on a self assembled  $In_2O_3$  film on a {1120} optical sapphire. After the growth of NWs to a length of

 $\sim 2 \ \mu m$  is accomplished, they are buried underneath a CVD Si dioxide. A chemical mechanical polishing step is carried out removing excess oxide and the gold catalyst particle at the NW tip exposing In<sub>2</sub>O<sub>3</sub> tips in the SiO<sub>2</sub> coating. This is followed by the deposition and patterning of a Pt film as the drain electrode. A blanket deposition of HfO<sub>2</sub> serving as the gate dielectrics is followed by the deposition of a second Pt electrode as the gate. In this configuration, NWs serve as electron channels between the bottom source, the In<sub>2</sub>O<sub>3</sub> thin film on the substrate, and the Pt drain.

A similar device is proposed by Schmidt *et al.*<sup>201</sup> with the addition of a surround gate. Si NWs are grown epitaxially on a (111) p-type Si substrate. The deposition of SiO<sub>2</sub> as the gate insulator is followed by the deposition of Al, the gate material. The whole structure is then embedded in SiO<sub>2</sub> by spin coating of spin-onglass and curing. The top surface is etched via reactive ion etching until Au catalytic particles are revealed. They then serve as electrical contacts to a subsequently deposited layer of Al or Ti as the source. The same system was previously reported by Ng *et al.*<sup>174</sup> with a ZnO NW, SiO<sub>2</sub> gate dielectrics and Cr surround gate and source.

Field emission related applications widely utilise vertically grown NWs and CNTs. The work of Pirio et al.<sup>210</sup> was already mentioned in regard to the better control on nanostructures in PECVD. A Ni catalyst layer is deposited inside a 1 µm diameter trench etched in SiO<sub>2</sub>. A doped poly-Si gate was previously formed around the trench opening. Nickel elsewhere is removed by a lift-off process. The subsequent PECVD in acetylene and ammonia leads to vertically aligned CNTs inside the trench serving as field emitters in this triode type cathode with a low voltage requirement. A similar device was later reported<sup>200</sup> with a single CNT (multiwalled) or Si NW deposited inside the trench. The number of resulting nanostructures is determined by the size of the catalyst. The size of the photoresist opening through which the catalyst spot is deposited is confined to 300 nm using e-beam lithography. This leads to a single CNT growth in 90% of all the apertures. Using Au catalyst and VLS technique, the same method is also demonstrated for growing Si NWs. A list of early work can be found in Ref. 233. In an alternative approach, the CNTs are grown first on a blank substrate and covered with various layers for spacer and gate structure.<sup>234</sup> A chemical mechanical polishing step removes protrusions on the surface exposing CNT locations. In this 'selfaligned' technique, registration is required for further integration.

A micromechanical application of the directed growth is presented in the form of a device mimicking the gecko effect.<sup>235</sup> For this purpose, large and compliant Si dioxide platforms are created and coated with polymeric NWs. Instead of epitaxial growth, formation of polymeric NWs occurs through the transformation of a photoresist layer under inductively coupled oxygen plasma by adjusting etching conditions. Complete coverage of platforms with NWs is achieved, and the adhesion force between a flat Al punchtip and the device is measured using a nano-indenter.

The vertical catalytic growth of CNTs is utilised by Takei *et al.*<sup>236</sup> for the fabrication of a force sensor. CNTs are grown between a Si beam and its underlying Si



10 Various examples of systems integration utilising directed growth. (a) CNT decoder. (reprinted with permission from Ref. 231, copyright 2004, American Chemical Society) (b) Vacuum microtriode. (reprinted with permission from Ref. 232, copyright 2002, American Institute of Physics) (c) Field emission microcathode. (reprinted with permission from Ref. 210, copyright 2002, Institute of Physics) (d) Vertical surround gate field effect transistor. (reprinted with permission from Ref. 201, copyright 2006, Wiley-VCH Verlag GmbH & Co. KgaA) (e) Pressure sensor (reprinted with permission from Ref. 164, copyright 2006, American Chemical Society)

substrate with a thermally oxidised surface. The resulting structure is a suspended beam that is connected to the substrate through CNTs. The structure is then coated with PDMS, which, by infiltrating between the nanotubes, imparts structural rigidity to the device. When the Si bridge embedded in PDMS is exposed to an external loading, it deflects and thereby deforms the underlying nanotubes. The load is then measured by monitoring the resistance change of the deforming nanotubes.

Gas sensors based on semiconducting CNTs are also reported, where conductance change of the nanostructures upon exposure to gas molecules is monitored. NO<sub>2</sub> and NH<sub>3</sub> sensor of Ref. 217 is one such example. In this particular example, multiple CVD grown CNTs are used to bridge electrodes with the method introduced in Refs. 21 and 213. Fluctuations in conductance measurements are observed to decrease significantly (from  $\sim 10$ to  $\sim 1\%$ ) when multiple nanotubes are used as opposed to single CNT devices. Functionalisation of CNTs through adsorption of polyethyleneimine leads to an increase of sensitivity to  $NO_2$  (better than 1 ppb). They are at the same time observed to be insensitive to NH<sub>3</sub>. On the other hand, functionalisation of CNTs with Nafion leads to the reverse effect of enhanced sensitivity to NH<sub>3</sub> and reduced sensitivity to NO<sub>2</sub>.

Biosensors constitute another application area of CNTs grown directionally such as the amperometric glucose biosensor where metallic MWCNTs were grown on a platinum substrate via CVD.<sup>237</sup> Closed ends of nanotubes are first etched open and an enzyme is immobilised. Electrons created due to enzymatic reactions taking place at the nanotube tip are then transferred to the Pt electrode. The quality of immobilisation is observed to be a direct consequence of etching procedure. The best limit of detection obtained is 0.19 mM.

Directional growth is also suitably applied to AFM probe fabrication with CNT tips. Marty *et al.*<sup>203</sup> grew single walled CNT bundles at the apex of conventional Si tips using Co as the catalyst in a methane environment. The catalyst thickness is reported to play a crucial role in determining the number of growing CNT bundles. A yield of 20-30% is attained when Co thickness is kept within 5–8 nm.

#### *Limit: growing single nanostructure*

It should be noted that in all of the mentioned examples, the dimensions of the site, on which the micro-/nanointegration is realised, is determined by lithography. Hence, the resolution of the lithographic step determines the extent and quality of the integration. Integration sites are either in the form of (111) sidewalls of etched trenches in Si,<sup>168</sup> suspended Si bridges<sup>170,187</sup> or photoresist coated silica platforms.<sup>235</sup> Furthermore, the dissociation of the catalytic coating into nanoparticles<sup>168,187</sup> or formation of polymeric nanorods out of the photoresist layer<sup>235</sup> are self-assembly processes allowing little control on the number of resulting NWs. Therefore most of the reported work is suitable for blanket coating of microstructures with a known density of NWs of a chosen size range, and it is out of the reach of these processes to place a certain number of NWs at certain spots on a microdevice. However, compared to many other self-assembly techniques, the ability to work directly on devices is still an advantage for individual addressability of specific parts. The growth of CNTs on

a hinged polysilicon panel is one of the most sophisticated examples of MEMS integration.<sup>232</sup> Hence, for applications where a blanket coating is required, this family of integration techniques is preferable over topdown methods, which are still unmatched in precision when it comes to placing a single NW at a specified location.

If the dimensions of the integration platform can be further reduced, growing a single nanostructure can be achieved in the fundamental limit (Fig. 11). To an extent this was realised by growing single and multiple walled CNTs within pores etched on a Si AFM tip.<sup>240</sup> The AFM tip is first flattened by scanning it on an abrasive surface under a high load. The flat platform is then anodised and etched in aqueous KOH leading to 100 nm diameter pores etched vertically to a depth of  $\sim 1 \ \mu m$ . Subsequent catalyst deposition in the pores and CVD growth leads to both singled walled and mutliwalled CNTs growing perpendicular to the flattened tip. The type of CNT depends on the type of the catalyst and growth conditions. The resulting structure is used as an AFM probe with a nanotube tip. A similar approach is utilised by Duesberg *et al.*,<sup>241,242</sup> where nanopores are created not by anodisation but by photolithography and subsequent amorphous Si deposition. Each nanopore is observed to accommodate a single CNT up to the pore diameter of 60 nm, beyond which catalyst film at the bottom of the pores dissociate and multiple CNT growth takes place. Since the pore diameter is controllable as opposed to the previous case and a one-to-one correspondence is obtained between pore diameter and CNT size, the method provides a better control of the resulting CNT structure. Besides, this is a true batch process, where all fabrication steps involved are in compliance with other semiconductor processes.

E-beam lithography, of course, lies on the other extreme.<sup>238,243</sup> Considering again AFM tips, PECVD is used to grow a single CNT on a catalyst island defined by e-beam lithography.<sup>244</sup> During the e-beam step, alignment marks are also defined for registration. After the deposition of a 20 nm thick Ni serving as the catalyst, Si microcantilevers are fabricated such that each cantilever has a catalyst spot at its tip. Finally CNTs of a 3-5 µm length are grown under NH<sub>3</sub> and C<sub>2</sub>H<sub>2</sub> flow as usual for PECVD growth. It is observed that catalyst islands of 50 to 200 nm diameter lead to the growth of a single CNT, whereas multiple growth takes place when 250 and 300 nm sizes are used. The explanation lies in the break-up of a large catalyst particle or a continuous film into nanoclusters and was observed for multiple CNT growth in DC PECVD<sup>239</sup> and InP<111>B NW growth via VLS.<sup>245</sup> Owing to the non-uniformity of cluster size formed this way, the resulting structures also lack uniformity in diameter and size. The study of Teo et al.<sup>239</sup> revealed that even for 200 nm diameter clusters, there is a slight distribution of CNT number per cluster. At 100 nm scale the distribution vanishes and a single structure is obtained. The structures obtained with cluster sizes <300 nm exhibit an enhanced uniformity in size.

Similar observations on the effect of the catalyst size were reported previously on CNTs,<sup>246</sup> GaAs NWs,<sup>238</sup> and ZnO NWs.<sup>247</sup> These specific examples represent a hybrid approach to micro-/nano-integration issue where



11 The effect of catalyst patterning on nanostructure growth. Growth of GaAs NWs on GaAs (111)B surface using a Au catalyst island of dimensions (a) 200 × 200 nm, and (b) 1 × 1 μm. (reprinted with permission from Ref. 238, copyright 1995, American Institute of Physics) (c) CNT growth from 100 nm diameter Ni catalyst islands. (reprinted with permission from Ref. 239, copyright 2003, Institute of Physics) (d) The number of resulting CNTs using different catalyst sizes –mentioned on the micrographs under the same growth conditions as (c) (reprinted with permission from Ref. 239, copyright 2003, Institute of Physics)

self-assembly sites are determined by high resolution lithography. One can similarly classify the field emitter with a single PECVD grown CNT<sup>248</sup> as an example of hybrid fabrication. Self-aligned counterparts of the same device<sup>200,210</sup> were already discussed in the section on 'Applications' with the aspect of reducing the number of CNTs. With the emergence of batch processes such as Ref. 241, such an approach will slowly loose its technological relevance in the future. In addition to e-beam lithography, one can also mention template techniques,<sup>249</sup> phase shifting photolithography,<sup>247</sup> nanoimprint lithography,<sup>250</sup> nanosphere lithography,<sup>251</sup> and manipulation with AFM<sup>252</sup> among the demonstrated techniques of catalyst patterning.

A similar accomplishment is reported by Mueller-Falcke *et al.*,<sup>253</sup> where a polymeric pellet of  $5-15 \,\mu\text{m}$ 

diameter including a single CNT protruding at its end is mechanically transferred to the tip of a microscale electrostatic actuator to serve as an in-plane AFM probe. The fabrication technique is based on catalytically growing NWs at the bottom of Si grooves.<sup>254</sup> The grooves are then filled with an epoxy based polymer (a convenient choice is SU-8).<sup>253</sup> After planarisation the polymeric pellets are released and become ready to be transferred and attached to other devices.

In an attempt to enhance the control on the orientation and the spatial distribution of NWs in VLS technique, the growth can also be confined within encapsulated nanochannels patterned within the plane of the substrate.<sup>255</sup> In this case, the gold slug fabricated in the middle of the channel serves as the catalyst for Si NWs and depending on its size, it might initiate the

growth of either one or two NWs that take the shape of the nanochannel. In this technique, nanopatterning is again required to define the locations and orientation of growth.

#### Integration with top-down methods

#### Manipulation

Manipulation at the nanoscale traditionally played a key role in transport property measurements<sup>256</sup> and proto-type device fabrication<sup>257,258</sup> with the possibility of atomic scale resolution.<sup>259–261</sup> Processes such as choosing a single NW or CNT from an ensemble and alignment of it with respect to contacts, usually in an SEM, constitute fundamental tasks for integration. However, its applications in systems development remain confined to pilot fabrication due to the lack of parallel assembly techniques.<sup>262</sup> Common manipulators range from scanning probe microscopes,  $^{263-267}_{268,269}$  optical  $^{268,269}$  and mechanical tweezers  $^{67,270-275}$  to nanorobotic manipulation systems with additional degrees of freedom.<sup>276,277</sup> Fabrication of mechanical tweezers also poses an interesting integration problem, where nanoscale end effectors, either NWs or CNTs, are to be attached to a platform that provides high precision motion control. It can be concluded that the lack of parallel assembly among other nanoscale related phenomena such as stiction remains a key challenge for all manipulation techniques to gain technological relevance for systems integration. A comparison of assembly rates measured in the number of assembled parts per hour shows a significant decrease (around three orders of magnitude) when printed wiring board assembly is compared to assembly via scanning probe microscopy.<sup>278</sup> The level of difficulty associated with this kind of assembly becomes clear when one compares the attachment of a CNT on to a Si tip through manipulation inside an SEM<sup>66</sup> and the growth of CNTs on multiple Si tips through CVD in an electric field.<sup>192</sup>

# High resolution lithography and serial deposition techniques

Since its introduction in the sixties, the development of UV lithography has been the workhorse of semiconductor industry. Its principle is based on inducing chemical changes in a photoresist material by exposing it to UV radiation through a mask. The pattern on the mask is thus transferred on to the photoresist in the form of regions of altered and unaltered chemistry.

Early day exposure tools were contact printers where the mask and the photoresist coated wafer were brought into contact. The low process yield due to defects introduced by this physical contact was overcome by the introduction of the proximity printing. This was followed by projection printing, where a high resolution lens is used to project the image of the mask on the photoresist layer. This progress was fuelled by the miniaturisation trend in microelectronics – as predicted by the Moore's Law – for high speed, high performance and low power consumption. Major focus through the development of the UV lithography was centred around a continuous pursuit of enhanced resolution. Resolution can be defined as the smallest linewidth to be consistently patterned. In projection printing, Rayleigh criteria can be adopted to define the resolution R (Ref. 279)

$$R = k_1 \frac{\lambda}{NA} \tag{2}$$

where  $k_1$  is the so called 'k-factor', a process parameter,  $\lambda$  is the illumination wavelength, and NA is the numerical aperture of the lens system. Reduction in both the exposure wavelength and the k-factor and increase in the numerical aperture constitute requirements for enhanced resolution and set the path for technology development.

Historically, each transition to a shorter wavelength was accompanied by major challenges for various fields ranging from mask making to lens manufacturing. For example, the comparably recent introduction of 157 nm DUV lithography required the development of an  $F_2$ excimer laser, lens materials with high concentrations of CaF<sub>2</sub> and, of course, new resist materials. Although these issues could be resolved, due to a lack of solutions to transmittance problems associated with the mask material, 157 nm lithography could not gain any commercial significance, since it was introduced in 1998.<sup>280</sup> For 193 nm lithography, the period from the time of first industry sale to market dominance lasted for 10 years due to similar challenges. This period required for maturity on the commercial level is observed to increase with increasing complexity of the lithographic techniques.<sup>280</sup> Each transition was also accompanied by an exponential increase in equipment costs. While a contact printer of early seventies was worth around \$100 000, 193 nm step-and-scan exposure tools of early 2000s were worth over \$12 million.281 The overall progress regarding the resolution has been astonishing and to an extent beyond expectations. For example, although 180 nm node seemed to be an optimistic limit for UV lithography almost 15 years ago,<sup>282</sup> 32 nm node is now considered to be the limit with the use of the state-of-the-art exposure tools and photoresist materials. At this node, half of the pitch of first level interconnect will be 32 nm.

A chronological list of exposure wavelengths with market dominance is given in Table 2. Currently 193 nm immersion technology is expected to be the next generation along this list to meet the requirements for the 45 nm and possibly 32 nm node. In this technique the gap between the lens and the wafer surface is filled with a fluid to increase the numerical aperture in equation (2).

Similarly, the pursuit of a low *k*-factor resulted in a variety of technologies addressing any parameter other than the wavelength and the numerical aperture. These technologies are known as resolution enhancement technologies and include phase shift masking, antire-flective coating, correction for optical proximity effects and off-axis illumination. In the period from 1995 to 2007, a reduction in  $k_1$  from about 0.7 to 0.3 has been obtained.<sup>283,284</sup>

Beyond this point, EUV technology ( $\lambda$ =13·5 nm) is the most likely candidate to replace DUV and reach beyond 32 nm.<sup>28</sup> Prototype EUV exposure tools by EUV LLC were already available by early 2000s with their own list of challenges<sup>281</sup> and the first full field systems were installed in 2006. The limit along this line of decreasing exposure wavelengths would be the X-ray lithography introduced in 1978. However, it also did not meet the expectations in surviving the competition on the commercial scale, mainly due to infrastructure related challenges.

In addition to UV lithography, there are other parallel/batch compatible lithographic techniques available including microcontact printing<sup>285–287</sup> and nanoimprinting,<sup>288,289</sup> which can be collectively described as soft lithography.<sup>290</sup> Most of the soft lithographic techniques can be applied to non-planar, large scale surfaces and are especially suitable for introducing chemical functionalities and establishing interface with biological species.

In nano-imprint lithography, pattern transfer takes place by embossing a resist material, usually a thermoplastic or a curable material, and inducing a thickness contrast rather than a chemical change. The deformed resist layer is then solidified. Since optics is not utilised, issues related to the resolution limit for photolithography mentioned above are not relevant. Solidification technique and mechanical strength of the resist material become important. Imprint lithography is listed among potential solutions for 32 nm node and beyond in the 2007 Edition of the International Technology Roadmap for Semiconductors.<sup>291</sup> So far 12 nm pitch was obtained with photocurable nano-imprint lithography.<sup>284</sup>

In microcontact printing, the patterning principle is based on the transfer of a SAM, usually alkanethiols, to a substrate using an elastomeric stamp. Poly-(dimethylsiloxane) is the most commonly preferred material for the stamp. High resolution lithography is needed only once to pattern a master, on which the PDMS stamp is cast. The quality of pattern transfer to the substrate can be enhanced by overcoming stamp distortions through improving mechanical properties of PDMS. The ability to rapidly replicate the master and print constitutes one of the major advantages of the technique.

The issue of the suitability for systems integration requires, of course, multiple printing with registry among the different layers. The extension of soft lithography to device studies was demonstrated as early as 1997 by integrating optical mask aligners into the printing process.<sup>292,293</sup> A more recent example is a 60 nm, four-mask MOSFET process demonstrated with nano-imprint lithography on 4 inch wafers.<sup>294</sup> Nano-imprint lithography was used in all of the four steps for active area, gate, via and metal contact definitions. An overlay accuracy of ~500 nm was reported over the entire 4 inch wafer.

In microcontact printing, overlay accuracy is strongly affected by stamp related issues. First of all, having a low elastic modulus and a high coefficient of thermal expansion made pattern transfer prone to mechanical and thermal distortions. Furthermore swelling of the stamp during its exposure to the molecular ink solution and sagging of unsupported areas were major obstacles.<sup>295</sup> By utilising a hard backplate, a 1  $\mu$ m overlay

accuracy over an area of  $5 \times 5 \,\mu\text{m}$  was reported in 2002 with a homemade aligner.<sup>295</sup> In a recent systematic study, the dependence of the overlay accuracy on the elastic modulus of the stamp material was studied.<sup>296</sup> A registration accuracy of  $4.96 \pm 0.02 \,\mu\text{m}$  obtained with Sylgard 184 PDMS (elastic modulus of 1.8 MPa) was reported to decrease to  $0.50 \pm 0.01 \,\mu\text{m}$  in the case of TOPAS (elastic modulus of 2600 MPa) over an area of  $50 \times 50 \,\mu\text{m}$ .

One challenge associated with such registration studies is the need to carry out alignment while the mould and substrate are brought into contact unlike proximity printing, where a fixed gap facilitates alignment. One solution to this issue was reported by Li et al. for the specific case of nano-imprint lithography.<sup>297</sup> In this study two sets of diffraction gratings, one on the substrate and the other one on the mould, were utilised giving rise to Moiré fringes. If two gratings with slightly different periods are superimposed, the resulting Moiré fringe has a much larger period. This amplification makes the detection of nanoscale shifts between the two layers possible. Moreover, since the fringe pattern is not dependent on the gap size, the aforementioned problem is eliminated. In the present study, double gratings were used in each layer with the matching pair located in the other layer. This increased the alignment accuracy even further. As a result sub-500 nm alignment accuracy was obtained over the 4 inch wafer.

In addition to parallel techniques discussed so far, serial top-down methods, such as e-beam lithography, electron beam deposition, focused ion beam (FIB) CVD or FIB milling, scanning probe techniques and laser direct write are also widely utilised for nanofabrication. They provide perfect control on the direction, location and number of nanostructures. However, in all these integration techniques, while the fabrication of the microstructure is carried out in a batch process, the final touch, i.e. the addition of nanoscale extensions, requires a time consuming and serial step, limiting their use to low volume and high end applications (Fig. 12). The capabilities, limitations and applications of these high resolution lithography and deposition techniques will be discussed in the remainder of this section.

E-beam lithography has traditionally been the leading integration technique and played a key role in the development of solid state devices based on nanoscale phenomena.<sup>301,302</sup> It is fairly well characterised, and although the width of the resulting NW is limited by electron scattering from the substrate, linewidths below 10 nm can be obtained.<sup>303,304</sup> The usual practice in integration has mostly been confined to bridging already existing electrodes by e-beam lithography. The mask is either utilised as an etch mask, where anisotropic etching leads to an NW below the mask or the mask is used to etch a channel which is then filled by electrodeposition,

Table 2 Chronological list of exposure wavelengths with market dominance (compiled with data from Ref. 280)

Exposure wavelength	Illumination source	Year of market dominance	Initial resolution, $\mu m$
g-line, 436 nm	Hg lamp	1982	1.25
i-line, 365 nm	Hg lamp	1991	0.80
Deep UV, 248 nm	KrF excimer laser	1998	0.45
Deep UV, 193 nm	ArF excimer laser	2006	0.15
Deep UV, 193 nm, immersion	ArF excimer laser	Emerging	0.04

lift-off or another technique. Finally, the structure is released from the substrate. Nanobridges of single crystal Si,<sup>305,306</sup> Pd and polypyrrole<sup>307</sup> among other materials are reported. An alternative deposition technique introduced by Yun *et al.*<sup>307</sup> involves delivering electrolytes into nanochannels with Si dioxide sidewalls and Si nitride bottom. The nanochannel spans two Au electrodes, and the electrodeposition of NWs takes place in this confined space as opposed to etching or lift-off. Accompanied by a suitable delivery method for the electrolyte, the technique can potentially lead to NWs of different compositions at desired locations.

Scanning probe lithography is another planar technique that has a variety of derivatives:

- (i) the anodic oxidation process, where a negative bias is applied to the scanning probe leading to an electrochemical reaction between substrate and water in air (fabrication of magnetic tunnel junctions<sup>300</sup>
- (ii) patterning mostly biomolecules and polymers by:
   (a) nanografting<sup>308-312</sup>
  - (b) (direct write) dip pen nanolithography.<sup>313–320</sup>

Near field electrospinning, a recently introduced technique,<sup>321</sup> can also be used for polymeric NW fabrication and integration with microstructures. The technique overcomes the instability issue associated with the liquid jet, which constitutes the major obstacle in the way of controlled electrospinning. This is achieved by bringing



12 Top-down integration. (*a*) SiC nanoresonators clamped at both ends fabricated through e-beam lithography. (reprinted with permission from Ref. 298, copyright 2005, American Institute of Physics) (*b*) Growing end effectors at the tip of a gripper through e-beam induced deposition. (reprinted with permission from Ref. 273, copyright 2001, Institute of Physics) (*c*) Examples of FIB milling and e-beam induced deposition in the fabrication process of a thermal nanoprobe. (reprinted with permission from Ref. 299, copyright 2001, Elsevier) (*d*) A gripper fabricated by FIB CVD. (reprinted with permission from Ref. 271, copyright 2004, American Institute of Physics) (*e*) A multiple tunnel junction fabricated through scanning probe lithography (reprinted with permission from Ref. 300, copyright 2004, Institute of Physics)

the substrate (collector) into the close vicinity of the electrode (500  $\mu$ m–3 mm) where the liquid jet is still stable. By moving the substrate in a controlled manner, NWs can be deposited. The technique is recently used to establish chip-to-chip connections.<sup>322</sup> Some of the previous alignment efforts associated with electrospinning include the introduction of a micromachined dispension tip and a rotating collector, <sup>323,324</sup> aligning nanofibres utilising a secondary electric field on the substrate via a pair of electrodes<sup>325,326</sup> with the possibility of multiple depositions for crossed structures<sup>327</sup> – this technique actually falls under the electric field alignment – and using a sharp counterelectrode.<sup>328</sup>

And finally, one can mention laser direct write technique based on thermal desorption of a SAM, usually alkanethiols on gold substrate, upon focusing a laser beam along a desired path.<sup>329</sup>

As opposed to e-beam and scanning probe lithography or other direct write techniques, e-beam deposition is used to grow NWs on released microstructures. Rather than planarity, e-beam deposition possesses an inherent three-dimensionality that is very suitable for MEMS applications. The technique will also be discussed later in the section on 'Adhesion issues' in the context of adhesion promotion between self-assembly products and microstructures. It is based on focusing the e-beam on a surface where the dissociation of residual organic species in the vacuum chamber and their subsequent deposition take place. Additionally, organic contaminants on the surface are also reported to migrate toward the electron beam and dissociate.<sup>330</sup> The path of the slowly moving e-beam determines the ultimate shape of the growing structure where continuous adjustment of focus and correction of astigmatism are required.<sup>273</sup> There is an optimum beam current associated with the process beyond which growth rates are observed to decrease.<sup>273</sup> The procedure is carried out using a conventional electron gun or specifically designed electron emitters such as the multiwalled CNT emitter reported by Dong et al.331

Early applications of the technique can be found in a review paper by Koops *et al.*<sup>332</sup> Initially, straight structures such as AFM tips,<sup>333,334</sup> four-point probes<sup>335</sup> and field emitters<sup>336</sup> were reported as products of the technique. This was followed by more intricate structures and integration studies. For example, rotation of the substrate can lead to three-dimensional structures such as octahedral frames.<sup>337</sup> In the study of Ooi *et al.*,<sup>337</sup> hook shaped nanoprobes are used to extract and unfold DNA from a human chromosome, followed by a variety of reports on e-beam deposited nanoprobes integrated with microactuators. One of these devices is the nanotweezers reported by Bøggild et al.,<sup>273</sup> consisting of metallised Si dioxide cantilevers on Si substrate that can be actuated electrostatically. These cantilevers are then utilised as platforms where the tips are grown by e-beam deposition. The shape of the tips including their approach angle can be adjusted for better gripping capability. The resulting tips exhibit considerable mechanical strength and prove to be suitable for manipulation.<sup>338</sup> Similar devices with enhanced measurement capability of the tip deflection are reported later.<sup>339</sup> In addition to these structural applications, the carbon deposit can also be used as a masking medium as demonstrated in a study on the electrical characterisation of individual microtubules.340

E-beam deposition can also be utilised to deposit metallic nanostructures in the presence of an organometallic precursor gas, while inorganic precursors are also reported.<sup>341</sup> Koops et al.<sup>336</sup> reported gold and platinum NWs deposited in the presence of dimethylgold-(trifluoro)acetylacetonate and cyclopentadienylplatinum-trimethyl precursors, respectively. Resulting structures consist of nanocrystalline metals embedded in an amorphous matrix due to carbon incorporation. Pt based nanoprobes are also demonstrated by Rangelow et al.,<sup>299</sup> where methylcyclopentadienyl dimethyl platinum is used as the precursor gas and two microstructures are bridged by a Pt nanoprobe with a high degree of carbon contaminants. Resistivity of the resulting structure is measured to be 900  $\Omega$  cm. The structure is utilised as a thermal sensing element. Similarly, Pt probes are grown at the tip of microfabricated grippers as nanoscale end effectors.<sup>274</sup>

Solid gold nanostructures are also obtained when  $Au(CH_3)_2(C_5H_7O_2)$  is used as a precursor gas in the presence of nitrogen or water vapour.<sup>342</sup> When nitrogen is used, the resulting nanotip consists of gold nanocrystals dispersed in an amorphous carbon matrix. When water vapour is used instead, the core of the tip consists of solid polycrystalline gold, and the whole structure is covered by a coating similar to the amorphous carbon matrix with gold crystals obtained in the case of nitrogen. It is also reported that any nearby deposition that takes place later leads to a thick contamination layer accumulating on the sides of neighbouring NWs facing the deposition site. This asymmetrical deposition also results in bending of the structures.

The necessity of the presence of environmental gases such as  $H_2O$  and  $O_2$  in addition to the organometallic precursor gas is based on the removal of carbon residues in the metallic structures.<sup>343,344</sup> Oxygen radicals formed during the process help the formation of CO and CO<sub>2</sub> molecules and thereby reducing the C content in the deposit. Increasing the deposition temperature is also observed to lead to a reduced carbon content and enhanced conductivity.<sup>336</sup>

Similarly, FIB CVD can be carried out in commercially available FIB systems to grow nanostructures at desired locations. The deposition rate is much higher compared to e-beam induced deposition.<sup>345</sup> Furthermore, the reduced penetration depth of ions compared to electrons facilitates the fabrication of complicated, three-dimensional structures.<sup>345</sup> One of the demonstrations of the technique is reported on a smooth glass surface (the tip of a glass capillary), where carbon NWs are grown under a Ga<sup>+</sup> ion beam in the presence of a precursor gas (phenanthrene) provided by nozzles that are located 300 µm above the substrate.<sup>271</sup> In addition to NWs, claw-like structures or coils are also reported.

Finally, FIB milling provides a more direct way of machining nanostructures, where one starts with a microstructure and thins it down to nanoscale such as the nanoscale end effectors of the thermally activated grippers by Wang *et al.*.<sup>274</sup>

#### **Batch compatible techniques**

Among the integration techniques discussed upto this point, there are some that can be described as batch compatible. In other words, these techniques are suitable for parallel registration and alignment of various layers or fabrication steps. These are mainly techniques based on the combination of self-assembly with either high resolution photolithography or various soft lithographies.

Additionally, some of the top-down techniques are not based on any nanolithographic tool and are still compatible with batch processing. Avoiding high resolution lithography, these techniques constitute the most innovative integration approach and some of them will be highlighted in this section. In many cases, the approach is based on creating a mould by using conventional photolithography. Inherent to the mechanism of formation, the mould itself may end up having nanoscale dimensions or further processing of the mould leads to a nanostructure. Two such examples were already mentioned previously in the section on 'Directional growth', where nanopores were created by photolithography and each pore was utilised as a growth site of a single CNT<sup>241</sup> or CNTs were grown on top of Si pyramids formed through TMAH etching.<sup>192</sup>

Let us start with timed etching as a natural extension of photolithography, although the level of control on dimensions is rather limited. Starting with a micrometre sized etch mask, one can etch the structural layer with a careful timing for achieving a certain undercut. If the structural layer in question is Si, dimensions of NWs can be further reduced by oxidation and subsequent oxide etch.<sup>346</sup>

Oxide etch becomes especially important in the case of pattern dependent oxidation (PADOX). It is well known that oxidation properties of structured Si is quite different from those of planar Si.347 Oxidation is observed to exhibit heavy retardation due to the buildup of viscous stresses if Si patterns such as cylinders are oxidised,<sup>348</sup> an effect that is widely employed to obtain Si nanodots within Si NWs.<sup>349</sup> The same principle can be utilised for NW fabrication. Si pillars with a diameter of  $\sim 2$  nm are reported as a result of PADOX using dry oxidation at 800°C for 30 min.350 Similarly, planar Si NWs are also reported.<sup>351</sup> It is important to carry out the oxidation at a low enough temperature where selflimiting oxidation can be obtained. Although both of the NW studies<sup>350,351</sup> started with the creation of a template NW using e-beam lithography, the technique offers a promising aspect for batch compatibility if the template can be created by conventional lithography.

'Size reduction lithography'<sup>352</sup> is another batch compatible technique. Although the name implies a close relationship to the aforementioned batch compatible methods, it is quite different. The technique is based on creating vertical walls in a sacrificial layer and performing a conformal deposition leading to a uniform coverage of the sidewalls. Once the sacrificial layer is preferentially etched, the coating on the sidewalls remains as a vertical structure whose thickness is determined by the deposition technique. Choi et al.<sup>352</sup> used poly-Si as the sacrificial layer and a low temperature oxide (LTO) as the conformal layer. After the removal of poly-Si, vertical lines with a thickness of 200 nm are obtained. Those structures are then employed as an etch mask for further anisotropic removal of the underlying wet oxide layer and patterning the Si substrate. The end result is Si NWs of 20 nm width. Further oxidation and wet etching reduce the width down to the order of 12 nm. An application area of the technique is the spacer patterning for CMOS

FinFET,<sup>353</sup> where a conformal deposition of phosphosilicate glass on vertically etched SiGe islands is used to fabricate 10 nm wide fins.<sup>353</sup> The variation of the critical dimension obtained with the technique is reported to be better than that obtained with e-beam lithography.<sup>354</sup> Similar techniques based on conformal deposition and etching exist.<sup>355</sup>

Crack formation technique offers another batch compatible method, where the mould formation is triggered by patterned microscale stress raisers and the end product, i.e. the crack, provides the mould at nanoscale. Early examples of the technique involve cracks initiating in a SiO<sub>2</sub> coating with stress raisers in the form of sharp corners etched in the underlying Si substrate.<sup>356,357</sup> When the coating is subjected to thermal loading, cracks are observed to initiate at sharp corners and propagate along the axis of symmetry of the stress raising feature overriding any effects associated with the crystalline anisotropy of Si. The method supplies the additional capability of diverting the crack from its straight path by employing free edges as crack attractors/terminators. Cracks are then filled with a material of choice by electroless deposition or electroplating. Crack filling proved to work down to 10 nm scale with a variety of materials.<sup>358,359</sup> There is no constraint on the direction of NWs, and for the aforementioned Si/SiO2 system, cracks are screened from each other's effect, i.e. their paths are still determined by the layout rather than neighbouring cracks, as long as their spacing is  $>20 \ \mu\text{m}$ ,<sup>360</sup> a suitable scale for MEMS.

A similar approach, where one starts with micrometre level lithography and ends up with nanostructures, is proposed by Partridge *et al.*<sup>361</sup> This technique is based on etching V-grooves in single crystalline Si with KOH and using them as moulds for NW fabrication. Two alternatives exist both utilising momentum driven assembly of Sb clusters in an inert gas aggregation source with Ar. The first one involves thermal oxidation (120 nm thick) of the Si surface after KOH etching. Thermally oxidised surface is then exposed to Sb cluster deposition with Ar flow rate adjusted to impart enough momentum to clusters so that they are reflected from the plateaus and accumulate at the bottom of the grooves. The resulting one-dimensional structure resembles an NW. However, Sb is observed to oxidise easily during the process. Therefore, an alternative use of the technique is proposed where Sb deposition takes place on the same Si structure, but this time with a Ti/ Au coating. The resulting Sb NWs are then utilised as an etch mask in Ar plasma, where exposed regions of the Ti/Au coating are removed. Finally, after removal of Sb clusters one ends up with Au NWs at the bottom of the grooves. As a result, the technique provides parallel NWs whose direction is dictated by substrate anisotropy.

A similar technique based on anisotropic etching is proposed for GaAs,<sup>362</sup> where a SiO<sub>2</sub> layer patterned in the form of parallel lines is used as an etch mask on the (100) GaAs wafer. GaAs is etched anisotropically using an aqueous solution of  $H_3PO_4$  and  $H_2O_2$ . Etched profiles resemble reverse mesas, and when neighbouring reverse mesas meet with prolonged etching, the bordering GaAs assumes a triangular cross-section, whose width depends on the etch time. Therefore, similar to the previous method, using micrometre level lithography leads to NWs, this time with careful timing of the etch process. The direction of the NWs is once again dictated by substrate anisotropy. After the process the wires are transferred on plastic substrates for printing purposes. However, the suspended GaAs structures can easily be kept on the wafer and integrated with microstructures as well.

Another interesting batch compatible technique is proposed by Elibol et al.,363 where NW growth takes place along channels defined by the collapse of a membrane upon sacrificial layer etch. Si NWs are fabricated at precise locations and the resulting device is used as a gas sensor. The fabrication takes place on a p-type, low doped Si wafer. A sacrificial amorphous Si layer of 10 nm thickness is sandwiched between a lower wet oxide (200 nm thick) and an upper PECVD oxide layer (400 nm thick). Upon wet etching of the amorphous Si layer, the upper oxide membrane collapses and forms contact with the lower oxide layer leaving nanochannels along its anchoring edges. These nanochannels are then filled through epitaxial growth of Si. The resulting NWs have a diameter of 50 nm. The integration of the NWs with electrical contacts and their use as gas sensors are also demonstrated.

The scalloping effect during deep reactive ion etching (DRIE), can also be utilised as a means for NW fabrication.<sup>364</sup> It is well known that sidewalls of etched trenches exhibit scallops due to repeating isotropic etch steps interrupted by passivation. If two neighbouring trenches are separated by a 1 to 2  $\mu$ m wide photoresist line, two trenches meet along this thin boundary due to the scalloping effect. Airgaps are created that are separated by nanoscale bridges, whose spacings can be changed by changing DRIE recipe. The structure looks like a cage between two trenches with its parallel bars spanning the whole length of the trenches. The crosssections of these NWs can be rounded and their diameters can be further reduced upon thermal oxidation thinning. The integration of such structures with MEMS can take place easily, as it is the case with the proposed thermoelectric and biomedical applications.

### Adhesion issues

Although high adhesive forces pose a problem during object manipulation at micro- and nanoscales, some of the mentioned micro-/nano-integration studies suffer from a lack of adequate adhesion. This is not an important issue if the microstructure is deposited on top of existing nanostructures giving rise to an adequate level of adhesion.<sup>365</sup> However, especially when external fields are used to align NWs with respect to microstructures or NWs are placed on microstructures using manipulators, the affinity between the two structures becomes the critical parameter.<sup>39</sup> Most of the time it is necessary to increase the strength of adhesion between the two after the integration process is over. The earliest examples involve the use of acrylic adhesives when integration was carried out manually under a microscope.<sup>270,366–368</sup> Apart from mechanical integrity, reduction of contact resistance is also an important motivation in electrical applications.

Electron beam deposition, a common technique for adhesion promotion, is based on depositing a carbonaceous solid film on the area of integration. This is carried

out by scanning an electron beam over a specific area repeatedly in an SEM as explained earlier among serial top-down techniques. An example where requirements on adhesion strength are especially strict involves the tensile testing of CNTs,<sup>330,369</sup> where a tensile testing apparatus is made of AFM cantilever probes connected to high resolution motors. Carbon nanotubes are attached on to the AFM tips using the electron beam deposition technique. It is reported that half of the samples failed prematurely at deposition sites. Tensile testing is also performed on gold NWs attached to a MEMS tensilometer by electron beam deposition.370 Another reported test is a buckling test, where a CNT is again attached to an AFM probe using electron beam deposition and is moved against a more compliant cantilever in an SEM.<sup>371</sup> The point at which buckling occurs is recorded and a value for the elastic modulus is obtained.

On the other hand, in the previous example about the extraction of inner shells,<sup>69</sup> where the nanotube is already weakened by the electrical breakdown and the small sliding force due to van der Walls interaction between shells is measured, such an adhesion promotion was not necessary. There are similar examples utilising an AFM tip to retrieve a nanotube from a cartridge.<sup>6</sup> The nanotube is then placed onto a MEMS stage and attached to it at its tips in the form of a suspended bridge using electron beam deposition. Lateral loading by an AFM tip is observed to lead to the failure of the welded spot instead of breaking the nanotube indicating a weak connection as opposed to the tensile testing study mentioned previously.<sup>330</sup> Nanotweezers with nanotube end effectors are just another example of controlled placement of multiple nanotubes on an AFM cantilever with electrical connections.<sup>67,372</sup>

In addition to AFM tips other types of manipulators including electrochemically sharpened tungsten wires are also utilised. Kim *et al.*<sup>373</sup> demonstrated the possibility of attaching bundles of nanotubes on to the tungsten tip through electron beam deposition and then using the bundle itself as a means of manipulating single nanotubes.

For electrical characterisation studies the use of an organometallic gas is required to reduce the contact resistance between the microelectrodes and the nanos-tructure to be attached. Madsen *et al.*<sup>374</sup> reported a decrease of contact resistance by two orders of magnitude when dimethylacetylacetonate gold(III) with water vapour is used during the attachment of CNTs with e-beam deposition.

Similarly, FIB CVD, previously discussed in the context of serial top-down techniques, also serves as an adhesion promoting technique. For example, in the gas sensor reported by Yu *et al.*<sup>65</sup> the tin dioxide nanobelts are attached to the electrodes using FIB method to deposit a thin Pt film on the integration site. In fact, the quality of the contact is reported to be extremely important for the prevention of the poisoning of the sensor.

Ultrasonic welding is yet another technique recently demonstrated on nanotubes spanning Ti electrodes.<sup>375</sup> Nanotubes are aligned in an AC field and a wire bonder with a welding head of an alumina single crystal is used to clamp and vibrate nanotube ends. It is observed that contact resistance decreases with increasing ultrasonic

power. Moreover, obtained transistors exhibit a high transconductance, and when nanotube bridges are loaded under an AFM tip, nanotubes are observed to break before the welded joints fail.

Soldering is also reported for NW/contact pad bonding.<sup>76</sup> This work was already mentioned in the section on 'Magnetic alignment', where Au capped Ni NWs are aligned with respect to Ni pads. In fact, the pads are made up of a stack of materials, where Ni is coated with Cu and Sn/Pb solder, where Cu serves as a wetting platform. Once Au caps of NWs make contact with the pads, the wafer is heated up to 180°C for a short time allowing the reflow of solder. A short time is required to prevent the formation of intermetallic compounds. The solder wets NW caps and establishes a solid contact with the pad as it is slowly cooled down accompanied by a substantial reduction in contact resistance. An analog integrator in the form of an RC circuit with an integrated NW is also demonstrated.

# Conclusions

The issue of micro-/nano-integration derives its importance from the necessity of building systems with nanoscale components. When used as extensions on micromechanical systems, NWs and CNTs can serve as digits for manipulation and handling, as sensing elements or as agents for the modification of surface properties such as the adhesive strength. Similar achievements are demonstrated in microelectronics and photonics where nanostructures serve as integrated field emitters, transistors or laser sources to name just a few application fields. Although individual components can be fabricated with great precision regarding dimensions, structure and properties, the success of a system level approach heavily depends on the nanofabrication technique's capability of providing a means of integration with higher order structures. Therefore the classification employed in this review is based on the fabrication method of the nanostructure. Integration techniques can be summed up under two headings: Selfassembly products and Top-down methods.

#### Self-assembly products

- 1. The original approach to integration was based on removing NWs and CNTs from their original fabrication sites and dispersing them in a solvent, where the microstructure of interest is also placed. The coming together of these two components with proper alignment can be carried out by two different means:
  - assembly can take place under electric, magnetic or fluidic fields thanks to the shape anisotropy of NWs and CNTs. The capability of this technique can be extended to include hierarchical assembly. Adhesion promotion for structural rigidity or for the reduction of contact resistance might be necessary. Various techniques such as electron beam deposition, FIB CVD and nanoscale counterparts of welding and soldering are reported
  - functionalisation of nano- and microscale components also provides an alternative means of attraction and assembly. Selectivity inherent to biological species is especially promising. A lithographic technique is usually required for the definition of functionalisation sites.

- 2. Contrary to the first technique, integration can be realised at the original fabrication site of NWs and CNTs:
  - most of the time, integration takes advantage of the fact that nanofabrication occurs with reasonable alignment. For example, for in-plane growth, crystalline surfaces provide a good platform for step edge decoration or directional catalytic growth. Similarly, anodic alumina templates provide out-of-plane alignment. An interesting alternative is a highly serial approach, where nanostructures exhibit no alignment within the plane. After synthesis they are registered with respect to prepatterned markers followed by the definition and fabrication of individual contacts
  - some applications, usually sensors, require a network rather than a well aligned set of nanostructures. If measurement of the collective response of the network would suffice for the operation of a specific microsystem, integration can again take place on the original nanofabrication platform.
- 3. Directional growth involves synthesis of nanostructures at desired sites on a microstructure through catalytic reactions. Vapour-liquid-solid technique in the case of NWs and CVD based techniques in the case of CNTs are widely utilised. Lithographic definition of integration sites might be necessary depending on the application. Hybrid approaches where high resolution lithography is accompanied by directional growth provide excellent demonstration of the limiting case where a single nanostructure is grown. Although demanding requirements of thermal and/or chemical resistance of the growth platform limit the choice of materials for the growth platform, directional growth is the most widely used technique among all self-assembly techniques in actual device applications.

#### **Top-down methods**

- 1. Manipulation is the most direct way of handling NWs and CNTs. Its applications to systems development remain confined to pilot production due to the lack of parallel assembly.
- 2. Parallel fabrication is the soft spot of high resolution charged particle beam lithographies and serial deposition techniques. As far as batch compatible lithographic techniques are concerned, DUV is pushed into its limits with immersion and resolution enhancement technologies, whereas soft lithographies such as microcontact printing and nanoimprinting have their own set of challenges including overlay accuracy.
- 3. Finally, batch compatible techniques that do not depend on high resolution lithography exist. Most of these techniques take advantage of special aspects of fabrication processes. These aspects range from side wall coverage in conformal deposition to scalloping effect associated with ICP DRIE. Other techniques utilise crystalline anisotropy or cracking to form nanoscale moulds by conventional UV lithography. Since original pattern definition is achieved through UV lithography, nanostructures are automatically registered.

From a comparison of the integration approaches discussed in this review, one can conclude that the lack

of deterministic assembly sometimes overshadows the capability of tight control on transport properties that is inherent to self-assembly, where significant improvements were reported regarding dimensions, crystalline orientation and doping process. Therefore hybrid techniques become important, where the sites of synthesis and growth are determined by lithography. Hence, one can take advantage of the structural control of selfassembly and spatial control of lithography. This is especially true for directional growth and explains the dominant role of the technique for device development. Top-down techniques such as manipulation, high resolution lithography and serial deposition methods suffer from expensive and/or non-parallel working principles that hinder large scale production. Batch compatibility of top-down integration is an important issue under study, whose success is crucial for the ultimate success of micro-/nano-integration.

Of course, one should remember that these statements provide a rather general assessment, whereas the suitability of any integration technique is ultimately judged by the targeted application. For a niche field with low throughput, a serial top-down method can be the best solution, while catalytic growth whose extent is defined by crude lithography can provide the easiest integration in another application, where a statistical placement suffices. For anything in the middle with requirements of increased throughput, reduced cost and deterministic placement of nanostructures, batch compatibility will be the most sought-after criterion in the near future.

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