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Nanowire Chemical/Biological Sensors: Status and a Roadmap for the Future

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1 International Edition: DOI: 10.1002/anie.201505308 1
Cerman Edition: DOI: [10.1002/ange.201505308](http://dx.doi.org/10.1002/ange.201505308) 2 **Sensors**

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Nanowire Chemical/Biological Sensors: Status and $\frac{6}{7}$ a Roadmap for the Future 3 3 3 4 -Nanowire Chemical/Riological Sensors: Status and 4 - 4 - 4 $\frac{5}{5}$ \ldots 7×7

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Sébastien Rochat, Katherine A. Mirica, Jens B. Ravnsbæk, and $\frac{11}{12}$ Timothy M. Swager* 10 Sébastien Rochat, Katherine A. Mirica, Jens B. Ravnsbæk, and **10** 10

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 Chemis Chemie Keywords: metal oxides · nanocarbons · 15 15 nanowires · sensors · transductionmechanism 2 www.angewandte.org © 2015 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim Angew. Chem. Int. Ed. 2015, 54, 2-17 \blacksquare κ These are not the final page numbers! 13 13 $\frac{1}{4}$ 14 $\frac{1}{6}$ <u>neduction mechanism and the control of the control of</u> 18 18 19 and 19 20 20 21 \sim 21 \sim 21 $\begin{array}{|c|c|}\n 20 & 20 \\
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 24 & 23\n\end{array}$ **for Sensor Devices 23** 23 24 24 25 / 25 **Functionalization** 26 $/$ 26 $27 / 27$ 28 28 29 29 $\frac{3}{2}$ 30 $\frac{30}{2}$ 30 $\frac{30}{2}$ 30 $\frac{30}{2}$ 30 $\frac{30}{2}$ 30 $\frac{30}{2}$ 30 $\frac{30}{2}$ $\frac{3}{2}$ $\frac{12}{2}$ 32 33 33 34 NW Assembly/ $\sqrt{2}$ CPs \leftarrow CPs \leftarrow 34 35 35 36 and $\frac{1}{2}$ and $\$ $\frac{37}{2}$ 37 $\frac{37}{2}$ 37 38 38 $\frac{39}{2}$ 39 $\frac{39}{2}$ 39 40 40 41 41 42 42 $\overline{43}$ $\overline{43}$ $\overline{43}$ 44 44 $\frac{45}{46}$ Device Performance $\frac{1}{46}$ $\frac{1}{46}$ $\frac{1}{46}$ $\frac{1}{46}$ $\frac{1}{46}$ 46 and 46 and 46 and 46 and 46 and 46 and 46 47 47 48 and 48 and 48 and 48 and 48 and 48 and 48 49 49 50 50 50 51 51 51 51 51 52 52 53 53 54 54 55 55 56 56 $\frac{1}{3}$ $\frac{1}{3}$ **Externational Edition Chemic** 59

Dateiname: A505308e Pagina: 2 Pfad: L:/daten/Verlage/VCH/ACH/hefte/pool/ Seite: 1 te von 18 Status Neusatz Umfang (Seiten): 18 Datum: 47 KW., 20. November 2015 (Freitag) Zeit: 8:21:49 Uhr

Sensors

 $\sum_{n=1}^{\infty}$ **C**hemiresistive sensors are becoming increasingly important as they **From the Contents** $\frac{2}{3}$ offer an inexpensive option to conventional analytical instrumenta- $\frac{3}{4}$ tion, they can be readily integrated into electronic devices, and they $\frac{1}{4}$ **htroduction** $\frac{3}{4}$ 5 have low power requirements. Nanowires (NWs) are a major theme in $\frac{1}{2}$, Sensory Device Performance $\frac{5}{2}$ 6 chemosensor development. High surface area, interwire junctions, and **Parameters Parameters** 4 6 *T* restricted conduction pathways give intrinsically high sensitivity and \blacksquare $\frac{8}{\circ}$ new mechanisms to transduce the binding or action of analytes. This 3. Nanowire Types 6 8 Review details the status of NW chemosensors with selected examples $\frac{9}{10}$
Review details the status of NW chemosensors with selected examples 11 from the literature. We begin by proposing a principle for under-
 $\frac{1}{2}$ $\frac{11}{2}$ 12 standing electrical transport and transduction mechanisms in NW and the contract of the standing electrical transport and transduction mechanisms in NW 13 sensors. Next, we offer the reader a review of device performance $\frac{13}{2}$ sensors. Next, we offer the reader a review of device performance $\frac{13}{2}$ $\frac{14}{15}$ parameters. Then, we consider the different NW types followed by **Sensor Devices** 8 $\frac{14}{15}$ $\frac{1}{6}$ a summary of NW assembly and different device platform archi-
6. Functionalization Methods for 17 *tectures. Subsequently, we discuss NW functionalization strategies.* Applications **Applications** 17 ¹⁸ Finally, we propose future developments in NW sensing to address **18** selectivity, sensor drift, sensitivity, response analysis, and emerging $\frac{1}{20}$ 7. Future Developments 11 19 applications. 4 and, they can be readily integrated this electronic devices, and they are all the second function of 4 10 Review details the status of two chemosensors with selected examples 4. NW Assembly and Sensor 10 15 parameters. Then, we consider the ufficient two types followed by 20 embations 20 21 applications. 21

1. Introduction

The omnipresence of wireless devices, cloud data, and electronic chemical sensors. These sensors enable govern-31 well as physical quantities. Over the last decade, modern For many systems, reductions in conductivity are observed 31 electronic chemical sensors have contained nanowires (NWs). 33 The assertion that NWs offer advantages in sensors is often heterogeneous potentials that result from analytes. However, 33 34 made without justification. However, we contend that it is analytes can also enhance conductivity by facilitating charge 34 35 generally warranted. The simplest reason is their high surface transfer across resistive interfaces, lowering barriers, or 35 36 area to volume ratios, which allows more interactions with injecting carriers through doping. 36 37 analytes. However, monolayers of 2D materials such as That a "turn-on" response is more sensitive than "turn- 37 als is how they transport current. 32 electronic chemical sensors have contained nanowires (NWs). as coherence in electronic transport is destabilized by 32

In a NW, electrical transport is primarily along the NW 42 axis. If the NW is small enough in diameter, high sensitivity is conductive upon exposure to an analyte. If this activation is 42 43 achieved because analytes can bind anywhere along the NW achieved by carrier injection, high sensitivity is obtained when 43 44 to perturb its entire conductivity. This effect is similar to what the NW has very low carrier density in the absence of the 44 45 our group reported with semiconducting polymers, where we analyte. Alternatively, NW networks can be assembled 45 46 demonstrated signal gain by wiring receptors in series.^[8] slightly below a percolation threshold such that they are 46 47 Although this principle was studied for exciton rather than highly resistive, and an analyte-triggered formation of new 47 48 charge transport, there was a correlation between the carrier conduction pathways can produce a large turn-on response. 48 49 path length and signal amplification. Thus, long single NWs Sensitivity without selectivity, however, is simply noise. 49 50 are attractive because sensitivity increases with the length Systems should respond strongly only to desired analytes. 50 available for interacting with analytes. However, they are difficult to fabricate. 4 In a NW, electrical transport is primarily along the NW analogy for NWs is an insulating device that becomes 4 5 available for interacting with analytes. However, they are Coupling molecular processes to conductivity changes for 5 S2 difficult to fabricate. Selectivity often involves careful molecular constructions. 52

53 Alternatively, disordered NW networks can be readily 53 54 deposited by solid transfer, printing, spraying, or drop-casting $\frac{1}{2}$ 55 a dispersion. Random networks of NWs, as opposed to $^{[8]}$ J. F. Hennell II, S. F. Liu, J. M. Azzarelli, Dr. J. G. Weis, Dr. S. Rochat, 55 56 densely packed aligned networks, have the advantage of 56 μ is the state of changes of 56 57 porosity with high surface area as well as limited contact
Massachusetts Institute of Technology 58 between NWs to give restricted pathways that preserve the ϵ cambridge, MA (USA) \sim 58 59 1D character. The contacts between NWs are critical: For E-mail: tswager@mit.edu 59

From the Contents

27 printable electronics is an extraordinary opportunity for tubes (SWCNTs) in Figure 1. Amongst all the NWs, our group 27 29 ments, businesses, and individuals to satisfy an ever-expand- exceptional aspect ratios, and numerous methods available 29 30 ing appetite to measure chemical and biological processes as for functionalization. 30 30 NWs with high carrier mobilities, these junctions are con-24 24 ductivity-limiting. Various intra- and inter-NW mechanisms 25 25 26 The omnipresence of wireless devices, cloud data, and of transduction are illustrated for single-walled carbon nano- 26 tubes (SWCNTs) in Figure 1. Amongst all the NWs, our group 28 electronic chemical sensors. These sensors enable govern- favors SWCNTs because of their excellent conductivity, 28 exceptional aspect ratios, and numerous methods available for functionalization.

> For many systems, reductions in conductivity are observed heterogeneous potentials that result from analytes. However, analytes can also enhance conductivity by facilitating charge transfer across resistive interfaces, lowering barriers, or injecting carriers through doping.

38 graphene^[1,2] and MoS₂^[3-7] also have high surface areas. The off" is often stated in fluorescence; however, this assertion is 38 39 most important difference between 1D NWs and 2D materi- only true with zero background fluorescence in the absence of 39 That a "turn-on" response is more sensitive than "turnoff" is often stated in fluorescence; however, this assertion is only true with zero background fluorescence in the absence of 40 als is how they transport current. conductive upon exposure to an analyte. If this activation is achieved by carrier injection, high sensitivity is obtained when the NW has very low carrier density in the absence of the analyte. Alternatively, NW networks can be assembled slightly below a percolation threshold such that they are highly resistive, and an analyte-triggered formation of new conduction pathways can produce a large turn-on response.

> Sensitivity without selectivity, however, is simply noise. Systems should respond strongly only to desired analytes.

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^[*] J. F. Fennell Jr., S. F. Liu, J. M. Azzarelli, Dr. J. G. Weis, Dr. S. Rochat, Dr. K. A. Mirica, Dr. J. B. Ravnsbæk, Prof. Dr. T. M. Swager Department of Chemistry and Institute for Soldier Nanotechnologies Massachusetts Institute of Technology Cambridge, MA (USA) E-mail: tswager@mit.edu

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 22 ented network of SWCNTs spanning two metallic electrodes has 22 a current (yellow cloud) flowing through it when a voltage is applied. 24 The presence of an analyte (red sphere) can inhibit current flow (red in a device successively exposed to increasing concentrations of 24 cloud) through: B) modulation of the Schottky barrier at electrode– $\frac{25}{26}$ cloud) through: B) modulation of the Schottky barrier at electrode—
23 25 28 SWCNT junctions, C) charge transfer between the analyte and SWCNT, $\frac{26}{25}$ or D) increasing the SWCNT–SWCNT junction distance by intercala-27 tion or swelling of the SWCNT network. 23 a current (yellow cloud) flowing through it when a voltage is applied. **Figure 2.** Graphical representation of selected performance parameters 23

Chemistry has provided decades of innovations in molecular tions by analyte binding. 30 Chemistry has provided decades of innovations in molecular The limit of detection (LOD) is the minimum amount of 30

In this Review, we survey the area of NW sensors. First, we progress. We then move on to compositions, fabrication 38 Rather than a comprehensive review, we discuss selected The LOD can be influenced by receptor-analyte inter- 38 status of the field, and opportunities for going forward, which we hope will provide a basis for future innovations.

Authors, from left to right: Prof. Dr. Timothy M. Swager, Dr. Jonathan G. Weis, Sophie Liu, Dr. Jens Ravnsbæk, John F. Fennell, Jr., Dr. Katherine Mirica, Joseph M. Azzarelli, Dr. Sébastian Rochat.

The performance of a chemical sensor is a product of the 3 A a surrent allowed physical form of the sensor material, strength of the analyte 4 transduction event, selectivity of the response to a given 5 5 \sim analyte, and the sensor's stability. Figure 2 shows a represen- $\frac{7}{2}$ intra-SWCNT $\frac{1}{2}$ and $\frac{1}{2}$ tative sensing trace that may assist in graphically visualizing $\frac{7}{2}$ key terms. 8 8

in a device successively exposed to increasing concentrations of analyte.

28 **28 2.1. Sensitivity, Dynamic Range, Limit of Detection** 28 **2.1.**

31 recognition to guide the development of chemical sensors. analyte that can be detected at a known confidence level.^[9] 31 32 The challenge is how to produce specific electronic perturba- The target LODs in sensors for environmental safety are 32 35 lay out performance parameters employed to evaluate National Institute of Occupational Safety and Health 35 37 methods, device architectures, and operational modalities. And Health at Work.^[12] 37 analyte that can be detected at a known confidence level.^[9] The target LODs in sensors for environmental safety are 33 tions by analyte binding. The same of the state of 34 In this Review, we survey the area of NW sensors. First, we the United States Environmental Protection Agency,^[10] the 34 National Institute of Occupational Safety and Health 36 progress. We then move on to compositions, fabrication $(NIOSH)$,^[11] and the European Union Agency for Safety 36 and Health at Work.^[12]

39 examples to highlight the advantages of NW sensors, the actions, surface area, functionalization, and signal amplifica- 39 The LOD can be influenced by receptor–analyte interactions, surface area, functionalization, and signal amplifica-40 status of the field, and opportunities for going forward, which tion. A low LOD is closely tied to high sensitivity (response $\frac{40}{2}$ 41 we hope will provide a basis for future innovations. \sqrt{p} per unit concentration). The extent to which an analyte 41 **influences the electronic properties of NWs is a major factor** 42

 56
 57 Authors from left to right: Prof. Dr. Timothy M. Swager. Dr. Ionathan G. FETs by an electric field. A) SEM image of a Si NW FET. B,C) Sche- 57 Authors, from left to right: Prof. Dr. Timothy M. Swager, Dr. Jonathan G.
The Weis Sophie Liu Dr. Jens Raynshock John F. Fennell Ir. Dr. Katherine matic illustrations of the possible APTES molecular structures before 58 Weis, Suprile Liu, Dr. Jens Ruvisblek, John F. Fennell, Jr., Dr. Rumerine
Mirica Joseph M. Azzarelli, Dr. Sébastian Rochat sion. Copyright 2013 American Chemical Society. 59 59

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1 modified Si NW field-effect transistor (FET) achieved LODs 2.2. Selectivity 2 of 0.1 fm for ssDNA and 0.5 ppm for alcohols. The authors 2 β deduced that the field creates structural order to increase the Selectivity is measured as the ratios of the sensitivity of β 4 efficiency of molecular reactions, strengthen the molecular the target analyte to that of interferents. Specificity should be \sim 4 5 dipoles, and consequently improve sensitivity. The reorgan- reserved for cases of ultimate selectivity. Researchers must 5 \acute{o} izations are likely more complicated than shown in Fig- choose relevant interferents to effectively demonstrate selec- \acute{o} 7 ure 3 B,C, as APTES generally produces multilayer, polymer- tivity. ized coatings.

An increase in surface area offers more sites for analyte– sensor interactions and lowers the LOD. Yue et al .^[17] 11 obtained high surface areas in ZnO NW devices by growing Increased selectivity can also be achieved by using a separate 11 12 them on 3D graphene foam, thereby increasing the surface device to bind and preconcentrate an analyte before thermal 12 13 area from 6 to 33 m^2g^{-1} and with a LOD of 1 nm for uric acid desorption onto the sensor.^[20-22] Many sensors derive selec- 13 and dopamine, indicators of Parkinson's disease. An increase in surface area offers more sites for analyte- analyte size, affinity, or permeation time.^[19] The operating $\frac{9}{2}$ 10 sensor interactions and lowers the LOD. Yue et al.^[17] temperature of a sensor will also affect the selectivity. 10

Signal amplification of molecular interactions can reveal 16 otherwise undetectable events. Russell et al.^[18] employed how functionalization can impart selectivity. 16 17 a rolling circle amplification (RCA) technique to create DNA 17 18 strands to template the formation of NWs between two 18 19 electrodes (Figure 4). This turn-on method detected synthetic **2.3. Stability** 19 20 and bacterial DNA at 100 pm and 66 fm, respectively. 20 20 15 Signal amplification of molecular interactions can reveal interact selectively with an analyte. In Section 6, we discuss 15

Chemical Society.

56 prevent saturation) and by lowering the LOD. Figure r Creation of a highly stable SWCNT sensor by nolymerization 56

2.2. Selectivity

Selectivity is measured as the ratios of the sensitivity of the target analyte to that of interferents. Specificity should be reserved for cases of ultimate selectivity. Researchers must choose relevant interferents to effectively demonstrate selectivity.

8 ized coatings. The same coating control of the Membrane coatings can exclude interferents based on 8 8 8 8 12 12 13 13 14 15 16 17 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 17 18 Increased selectivity can also be achieved by using a separate device to bind and preconcentrate an analyte before thermal desorption onto the sensor.[20–22] Many sensors derive selec-14 and dopamine, indicators of Parkinson's disease. tivity from functionalization with recognition elements that 14 how functionalization can impart selectivity.

2.3. Stability

21 Stability is determined by the sensor's ability to produce 21 22 the same output for an identical input over time, and is 22 23 \overrightarrow{A} $\overrightarrow{PEG}\substack{Surface oligo}{PEG}\substack{shape}$ quantified as a ratio of the response of the aged device 23 24 **relative to that of a new device. A stable sensor should remain** 24 25 **and the lifespan of the device.** 25

26 SAM of oligonucleotide and PEG NW functionalization has been explored to improve 26 stability. Our group demonstrated how SWCNTs functional-27 27 ized with trialkoxysilane moieties exhibit increased robust-28 28 ness (Figure 5). Hydrolytic polymerization affixes the 29 29 SWCNTs in place on the glass substrate. These sensors can 30 S1 survive sonication in methanol.^[23] Alternatively, coatings can 31 32 Hybridization and ligation of padlock probe be used to stabilize devices by functioning as a barrier to 32 33 **D CONFIDENTIAL CONSTRUCTED CONFIDENT CONSTRUCTED CONSTRUCTED CONFIDENTIAL CONFIDENTIAL 33** 33

Figure 5. Creation of a highly stable SWCNT sensor by polymerization $\frac{2}{57}$ of a functional selector around the deposited NW network. Reprinted $\frac{57}{57}$ 58 from Ref. [23] with permission. Copyright 2013 American Chemical 58 Society. 59 Society. Society.

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2.4. Drift

Drift is the stimuli-independent change of a measurable output over time. It can lead to uncertain results, false alarms, 5 and the need for frequent recalibration or replacement of metallic electronic state; most NWs in chemical sensors are in 5 6 sensors. Furthermore, there is a dearth of discussion on drift fact semiconductors. Semiconducting polymers, which can be 6 in the literature. There can be both short- and long-term drift, 8 and the difference between the two can in some cases allow affording signal amplification prior to the extensive efforts 8 one to deconvolute specific contributions.[28–30] Some poten- 10 tial causes include reorientation of particles and domains, a more conventional description of NWs: high aspect ratio 10 segregation of mixtures, sublimation/evaporation of compo-12 nents, adsorption of species, doping/de-doping, and charge and often polydisperse in terms of length, diameter, and 12 13 migration. The high surface area of NWs creates a challenge composition. 13 14 for drift, and how to mitigate these factors without passivation 14 15 needs further exploration. 15 4 output over time. It can lead to uncertain results, false alarms, capable of charge transport. Wire does not imply an intrinsic $\overline{4}$ \overline{z} in the literature. There can be both short- and long-term drift, considered as molecular wires, were first recognized as \overline{z} 9 one to deconvolute specific contributions.^[28-30] Some poten- on NW sensors.^[41-43] However, we will restrict ourselves to 9 11 segregation of mixtures, sublimation/evaporation of compo- materials that are larger than a conventional polymer chain 11

 $\frac{1}{6}$ Drift can be addressed either by in-device recalibration^[31] **3.1. Elemental Semiconductor NWs** 16 17 or algorithmically during data processing and/or workup, for 17 However, many applications cannot sustain intensive compudrift must be addressed at the device level.

2.5. Hysteresis

Hysteresis is the difference between outputs when an 27 analyte concentration is approached from an increasing and \qquad ity.^[48] $\qquad \qquad$ 27 When this suppression is not possible, the best one can do is characterize the hysteresis of the sensor.

2.6. Response Time, Dead Time, Rise Time

 36 Response time is determined at 90% of its final amplitude 36 37 after analyte exposure,^[33] dead time is the time it takes to **3.2. Metal Chalcogenide or Pnictide NWs** 37 38 reach the first 10% of its final signal, and rise time is the 38 difference.^[34] The response time is critical, as many applications employ sensors to activate systems in response to temperature,[33] and catalysis.[36]

2.7. Reversibility and Recovery Time

Reversibility is the extent to which the signal is restored to 49 its initial state prior to analyte exposure.^[37] The recovery time selectivity and high operating temperatures.^{[55,59,60}] Nitrogen 49 50 is the time to decrease to 10% of the peak amplitude after oxides (NO_x) have been analytes of choice to assess the 50 removing the analyte. Although irreversibility can be 52 exploited in dosimetry, incomplete, sluggish recovery is oxides can be synthesized by methods^[52] that are variations on 52 often undesirable. A 8 Reversibility is the extent to which the signal is restored to However, their use in sensing is often hampered by poor 48 5 removing the analyte. Although irreversibility can be performance of devices based on metal oxides. $[61-64]$ NWs of 5

54 Remedies to promote complete recovery include heating oxidation.^[57,66,67] Remedies to promote complete recovery include heating oxidation.^[57,66,67] 55 the sensor^[14,38,39] or exposing the sensor to ultraviolet (UV) 55 56 light.^[40] However, heat and UV treatment create more 56 57 complex systems, with difficulties such as baseline noise and 57 58 attenuated device lifetimes. Further investigations into alter-
58 59 nate solutions for irreversibility are needed. 59

$\frac{1}{2}$ 2.4. Drift 2.4. $\frac{1}{2}$ 2.4. $\frac{1}{2}$ 2.4. $\frac{1}{2}$ 3. Nanowire Types

3 Drift is the stimuli-independent change of a measurable We define a NW as a high aspect ratio nanostructure 3 metallic electronic state; most NWs in chemical sensors are in fact semiconductors. Semiconducting polymers, which can be affording signal amplification prior to the extensive efforts a more conventional description of NWs: high aspect ratio and often polydisperse in terms of length, diameter, and composition.

3.1. Elemental Semiconductor NWs

18 example, by using principal component analysis $(PCA).^{[30]}$ NWs fabricated from Si and Ge have been extensively 18 20 tational solutions to sensor drift. Therefore, the challenge of popular as a result of their compatibility with Si electronics 20 NWs fabricated from Si and Ge have been extensively 19 However, many applications cannot sustain intensive compu- studied in sensors, especially in FETs.^[44–47] Si NWs are 19 popular as a result of their compatibility with Si electronics 21 drift must be addressed at the device level. \Box and because Si doping and functionalization are mature 21 technologies. However, they have limited stability and oxidize 22 22 quickly, thereby resulting in surface passivation. Silica NWs 23 23 24 **2.5. Hysteresis** Superior Section 24 (SiO₂ NWs), however, have been substantially documented as 24 and 24 an excellent material for sensing applications because of their 25 25 26 Hysteresis is the difference between outputs when an ease of modification, functionalization, and biocompatibil- 26 ity.[48]

28 decreasing range.^[32] It is important to minimize as a result of Si/Ge NWs can be synthesized through methods such as 28 29 the challenges it poses to reversibility and dynamic range. thermal evaporation.^[49,50] A common technique for growing 29 Si/Ge NWs can be synthesized through methods such as thermal evaporation.^[49,50] A common technique for growing 30 When this suppression is not possible, the best one can do is various inorganic NWs is the vapor-liquid-solid (VLS) 30 31 characterize the hysteresis of the sensor. method by using chemical vapor deposition (CVD) process- 31 $es^{[51,52]}$ Laser ablation can be used with the VLS method.^[53] 32 Si/Ge NWs can also be grown from solution using a super-33 **2.6. Response Time, Dead Time, Rise Time** critical fluid-liquid-solid growth method.^[54] 34

3.2. Metal Chalcogenide or Pnictide NWs

4 changing analyte levels.^[32] The factors that govern response as GaN or AlGaN have been used extensively in chemiresis-4 42 time mirror those of reaction kinetics: surface area,^[35] tive gas sensors^[36,55–57] and FETs.^[47,58] Their sensor function 42 39 difference.^[34] The response time is critical, as many applica-
NWs of chalcogenides such as SnO_2 , ZnO , TiO_2 , In_2O_3 , 39 40 tions employ sensors to activate systems in response to WO_3 , V_2O_5 , CuO , Cr_2O_3 , Nb_2O_5 , and Fe_2O_3 and pnictides such 40 as GaN or AlGaN have been used extensively in chemiresistive gas sensors $[36, 55-57]$ and FETs. $[47, 58]$ Their sensor function 43 temperature,^[33] and catalysis.^[36] often relies on redox reactions between the analyte and the 43 surface, thereby generating variations in carrier concentra-44 44 tions, or surface trapping. They are inexpensive, robust to 45 45 46 **2.7. Reversibility and Recovery Time temperature and resistant to caustic environments, easily 46** d integrated into electronic circuits, and give high sensitivity.^[59] 47 selectivity and high operating temperatures.^[55,59,60] Nitrogen oxides (NO_x) have been analytes of choice to assess the oxides can be synthesized by methods^[52] that are variations on 53 often undesirable. the VLS method in $CVD^{[44,57,58,65]}$ or catalyst-free thermal 53 oxidation.^[57, 66, 67]

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3.3. Carbon Nanotubes (CNTs)

been a widespread effort to exploit their properties. CNTs can be considered as long hollow tubes of rolled-up graphene and the tube diameter determine whether a CNT is semiconducting or metallic. CNTs are either single-walled (SWCNT) or multi-walled (MWCNT). MWCNTs consist of $\frac{12}{2}$ multiple concentric layers of SWCNTs. In defect-free tubes, chemiresistor architectures have revolutionized sensing by $\frac{12}{2}$ the bonds between carbon atoms in the sidewalls are sp^2 hybridized, and noncovalent van der Waals forces or π stack- $\frac{15}{15}$ ing dominate the intermolecular interactions. CNTs are tion.^[120] The CPs can serve as a receptor layer, transducer, $\frac{15}{15}$ 16 synthesized by laser ablation,^[72] arc discharge,^[71] CVD, and protective coating, or electronic circuit.^[122] Sensing with 16 17 combustion.^[73–75] CVD is the premier method, with mild nonfunctionalized CPs is mostly limited to small molecules 17 18 conditions, high yield, simplicity, and facile mediation of (e.g. HCl, NH₃, hydrazine, chloroform, acetone, acetonitrile, 18 physical characteristics.[76, 77] Recent reviews can be found on CNTs in biotechnology and drug delivery, $[69,78-80]$ electronand the tube diameter determine whether a CNT is semi-electrophoretic deposition, and thermal evaporation.^[120,121] 10 conducting or metallic. CNTs are either single-walled Sensors based on FETs (organic field-effect transistors, 10 1 (SWCNT) or multi-walled (MWCNT). MWCNTs consist of OFETs, and organic electrochemical transistors, OECTs) and 11 13 the bonds between carbon atoms in the sidewalls are sp^2 using CPs with short response times, high sensitivity, easy 13 14 hybridized, and noncovalent van der Waals forces or π stack- device integration,^[108,120] and room-temperature opera- 14 19 physical characteristics.^[76,77] Recent reviews can be found on alcohols, and benzene)^[123,124] and cations (e.g. Cu^{2+} ,^[125,126] 19 20 CNTs in biotechnology and drug delivery,^[69,78-80] electron- $Ag^{+[127]}Pb^{2+}$ and $Cd^{2+,[128]}$ and K^+ and $Ca^{2+}[129]}$ in solution 20

23 because of the aforementioned properties, compatibility with sensor properties.^[130] Long-term instability, irreversibility, and 23 organic chemistry for functionalization,[87] and easy integra-25 tion into electronic circuits.^[88] CNTs have been used to sense 25 26 biological molecules^[89–95] and vapors of industrial gases and 26 explosives.[23, 96–102] 24 organic chemistry for functionalization,^[87] and easy integra- poor selectivity are the main drawbacks of CPs in sensing.^[120] 24 27 explosives.^[23,96–102] 27 **4. NW Assembly and Sensor Fabrication** 27

3.4. Transition-Metal NWs

Metal-based NWs such as Ni, Pt, Pd, Au, Ag, Pb, and Co discribed applications in chemical sensing compared and the sensing compared and sensing compare

3.5. Conducting Polymer NWs

 48 materials that are semiconducting upon oxidation or reduc- Figure 6D printed from Ref. [144] with permission from the American 48 49 tion (doping).^[108] Their molecular construction allows for Chemical Society 2003; Figure 6F printed from Ref. [145] with permis- 49 50 intimate integration of receptor units into the backbone. Sign from the Royal Society of Chemistry 2014. 51 Sensing relies on inducing modification into the band 51 structure or structure, which results in changes in the electronic properties.[108] Many reviews exist on these matericonstructions created from CPs.

Polymers in 1D structures are formed by oxidative polymerization and can have improved electrochemical capacities.[113, 114] Templated synthesis relies on physical supports (hard template) to create nanostructures or on molec-56 Polymers in 1D structures are formed by oxidative single-crystalline NWs^[132] and n- and p-doped Si and Ge 56 57 polymerization and can have improved electrochemical nanowires^[133,134] in FETs^[135] for the detection of DNA,^[136] 57 58 capacities.^[113,114] Templated synthesis relies on physical sup- single viruses,^[137] cancer markers,^[138] and interactions 58 59 ports (hard template) to create nanostructures or on molec- between small molecules and proteins.^[139] The Nuckolls 59

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3 CNTs are excellent sensor candidates due to their spinning of nanofibers allows control over the size, alignment, 3 4 mechanical and electrical properties.^[68–70] Since Iijima^[71] morphology, and surface functionalization.^[116,117] Nanolithog- 4 5 significantly raised the profile of CNTs in 1991, there has raphy methods allow reproduction of a pattern on a sur-8 sheets. The angles at which they are rolled (the chiral vector) inkjet printing, dip-coating and spin-coating, drop-casting, 8 **3.3. Carbon Nanotubes (CNTs)** Ular self-assembly to guide the growth of CP-based NWs (soft 1 2 template). Template-free syntheses also exist.^[115] Electro- 2 spinning of nanofibers allows control over the size, alignment, morphology, and surface functionalization.^[116,117] Nanolithography methods allow reproduction of a pattern on a sur-6 been a widespread effort to exploit their properties. CNTs can face.^[118,119] CP NW films can also be deposited by layer-by- $\%$ be considered as long hollow tubes of rolled-up graphene layer (LbL) methods, Langmuir–Blodgett (LB) techniques, inkjet printing, dip-coating and spin-coating, drop-casting,

2 ics,^[81] energy production and storage,^[82–84] and catalysis.^[85,86] and reactive gases (e.g. H₂, N₂H₄, NH₃, H₂S, HCl).^[122] CPs can 21 22 CNTs have found a special place in the sensor community be functionalized and included in formulations to improve 22 chemiresistor architectures have revolutionized sensing by tion.[120] The CPs can serve as a receptor layer, transducer, protective coating, or electronic circuit.^[122] Sensing with nonfunctionalized CPs is mostly limited to small molecules (e.g. HCl, NH3, hydrazine, chloroform, acetone, acetonitrile, alcohols, and benzene)^[123,124] and cations (e.g. Cu^{2+} ,^[125,126] $Ag^{+, [127]} Pb^{2+}$ and $Cd^{2+}, [128]$ and K^+ and $Ca^{2+}, [129]$ in solution and reactive gases (e.g. H_2 , N₂H₄, NH₃, H₂S, HCl).^[122] CPs can be functionalized and included in formulations to improve sensor properties.[130] Long-term instability, irreversibility, and

29 NW devices can be classified by the number of NWs (one 29 30 **3.4. Transition-Metal NWs** α or many) and orientation (aligned, unaligned). Single NW 30 devices have a NW bridging the gap between two electrodes 31 32 Metal-based NWs such as Ni, Pt, Pd, Au, Ag, Pb, and Co (Figure 6A,B) and offer high sensitivity, fast response to 32

47 Conducting (or conjugated) polymers (CPs) are a class of Ref. [143] with the permission from 2006 Nature Publishing Group; 47 external and the set of **15 3.5. Conducting Polymer NWs**
images): A,B) Device with a single nanowire; C,D) device with horizontally aligned NWs; E,F) device with a Single handwick, C,B) device with her control and the single handwick of the single handwick of the single handwick of the single mathematic and the single mathematic of the single mat Ref. [143] with the permission from 2006 Nature Publishing Group; Figure 6D printed from Ref. [144] with permission from the American Chemical Society 2003; Figure 6 F printed from Ref. [145] with permission from the Royal Society of Chemistry 2014.

54 als as sensors.^[109–112] We restrict this discussion to NW equipment to fabricate, and the low yield of functional 54 52 structure or structure, which results in changes in the changing analyte concentrations, and high spatial resolu- 52 53 electronic properties.^[108] Many reviews exist on these materi- tion.^[131] However, single NW devices require specialized 53 equipment to fabricate, and the low yield of functional devices increases the cost. The Leiber research group has used 55 55

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Figure 7. Electrical detection of methyl transferase (M.SssI) binding at a DNA-bridged CNT device. Reprinted from Ref. [142] with permission. Copyright 2012 Royal Society of Chemistry.

22 research group (Figure 7) has shown novel NW junctions, $[140]$ carrier densities through modulation of the applied gate 22 23 where SWCNTs are cut and then reconnected by small voltage and provides insight into the sensory mechanism 23 24 molecules with probes to observe binding events such as the (Figure 8B). 24 25 methylation of DNA.^[141] They also bridged SWCNT-SWCNT Changes in capacitance can also be analyte-specific. The 25 26 junctions with DNA, thus illustrating the ability of DNA- high surface area and prospects for polarizing interfaces are 26 nanowire devices to serve as sensors for biochemical events.[142] 27 nanowire devices to serve as sensors for biochemical well-suited to creating large capacitive signals (Figure 8C). 27

Aligned multi-NW devices (Figure $6C$,D) have multiple 30 NWs arranged in a single orientation. Compared to NW electrophoretic effects from an applied directional voltage, 30 31 networks, aligned NW devices have longer mean-free paths which can reduce drift and enhance reversibility. Moreover, 31 32 for conducting electrons and meet percolation thresholds for simultaneously examining changes in conductance and capac- 32 33 connectivity with fewer NW–NW contacts. Accordingly, it ance can help increase the specificity. 33 34 aligned NW devices can have lower resistance and greater The analog nature of chemiresponsive circuit components 34 29 • Aligned multi-NW devices (Figure 6C,D) have multiple can reduce $1/f$ noise, ensure rapid response, and avoid 29

Devices based on NW networks (Figure $6E$, F) are those 37 in which the orientation of the NWs is random. The sheer antennas. Resonant circuits are comprised of inductive, 37 38 number of NWs results in statistics mitigating the effects of resistive, and capacitive elements, so chemiresponsive ele- 38 39 electronic heterogeneity arising from polydispersity in NW ments directly influence their resonant frequency and Q fac- 39 40 length, diameter, and structure. Network devices diminish the tor. Our group made use of these features and developed 40 4 need for precise position or orientation of any individual NW, SWCNT sensors that are wirelessly powered and read by 4 \vert which lowers the difficulty in fabricating devices. On the other 43 hand, network devices have shorter mean-free paths for 43 44 electrons, thereby leading to higher resistance and lower 44 36 Devices based on NW networks (Figure 6E,F) are those resonant circuits and radio-frequency identification (RFID) 36 42 which lowers the difficulty in fabricating devices. On the other smartphones^[146] (Figure 9). This method uses the smart- 42

56 (source and drain) on an insulating support connected by $\frac{1}{2}$ 57 NWs (Figure 8A). The resistance can be measured by $\frac{12}{2}$ municating with a passive RFID tag. Reprinted from Ref. [146] with 57 58 monitoring changes in the current with a fixed voltage bias permission. Copyright 2014 Proceedings of the National Academies of 58 59 across the electrodes. 59 Science.

 $\frac{13}{14}$ chemicapacitor^[15] devices and corresponding circuit diagrams and $\frac{13}{14}$ data readouts. $\frac{1}{4}$ $\frac{1}{4}$

 $\frac{17}{2}$ Figure 7. Flectrical detection of methyl transferase (M Sssl) binding at It can be advantageous to control the carrier concentra-18 a DNA-bridged CNT device. Reprinted from Ref. [142] with permission. tion when transduction results from charge-transfer interac- 18 19 Copyright 2012 Royal Society of Chemistry. The contract of the service of the semiconducting NW and an analyte. An 19 additional electrode (gate) underneath the support converts 20 20 a resistor into a FET and introduces additional control of the 2 carrier densities through modulation of the applied gate voltage and provides insight into the sensory mechanism (Figure 8B).

Changes in capacitance can also be analyte-specific. The high surface area and prospects for polarizing interfaces are 28 events.^[142] **The use of an AC field for measurements in chemicapacitors** 28 electrophoretic effects from an applied directional voltage, which can reduce drift and enhance reversibility. Moreover, simultaneously examining changes in conductance and capacitance can help increase the specificity.

 35 current density, but require complicated assembly processes. allows for facile integration into wireless devices such as 35 The analog nature of chemiresponsive circuit components allows for facile integration into wireless devices such as antennas. Resonant circuits are comprised of inductive, resistive, and capacitive elements, so chemiresponsive elements directly influence their resonant frequency and Q factor. Our group made use of these features and developed SWCNT sensors that are wirelessly powered and read by

Figure 9. Illustration of a smartphone wirelessly powering and communicating with a passive RFID tag. Reprinted from Ref. [146] with permission. Copyright 2014 Proceedings of the National Academies of Science.

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circuit of a passive sensor tag and reconnecting it with chemiresponsive SWCNTs.

6. Functionalization Methods for Applications

NWs display useful intrinsic sensor properties, but functionalization is generally necessary to improve the processability, sensitivity, selectivity, operating conditions, and stability.

6.1. Functionalization of Inorganic Semiconductor NWs 6.1.1. Noncovalent Functionalization of Inorganic Semiconductor NWs

19 sputtering metal films (Pt or Pd, typically 100 Å) on top of $\frac{19}{2}$ 20 GaN,^[147] InN,^[148] or Si.^[149] In the presence of a Pt or Pd 6.2. Functionalization of Metal Oxide NWs 20 21 catalyst on the NWs, H_2 binds and dissociates, thereby 6.2.1. Compositional Mixtures with Metal Oxide NWs 21 22 modifying the carrier concentrations as evidenced by FET 22 23 measurements.^[150] Coating NWs with metals allows for faster, Metal oxides possess many desired properties, but rarely 23 enhanced response and shortened recovery time. Au nano-25 particles (AuNPs) were deposited onto GaN NWs by plasma- and carrier density can be modulated by preparing mixed- 25 enhanced chemical vapor deposition (PECVD), and physisorption of gases such as N_2 and CH₄ reduced the conductivity of the NW network.^[151] CO₂ sensing was achieved by coating GaN NW transistors with poly(ethyleneimine) (PEI). 30 In the presence of CO_2 and humidity, carbamic acid groups CuO NWs allows the detection of NH₃ at room temper- 30 31 form and ionize to create new charges that affect the transport ature.^[178] The presence of highly environment sensitive nano-
31 of the NWs.[152] 24 enhanced response and shortened recovery time. Au nano- does one exhibit all the desired attributes.^[167] Work function 24 26 enhanced chemical vapor deposition (PECVD), and phys- metal oxides. Considerable effort has been made to function- 26 27 isorption of gases such as N₂ and CH₄ reduced the con-alize SnO₂.^[58] Gaseous analytes such as H₂S,^[168] EtOH,^[169–172] 27 28 ductivity of the NW network.^[151] CO₂ sensing was achieved by CO₂^[173] H₂^[174] and formaldehyde^[175–177] have been detected 28 29 coating GaN NW transistors with poly(ethyleneimine) (PEI). using mixed metal oxides. Integrating SnO₂ nanocrystals with 29

Specific detection of biological species is achieved by the In_2O_3 NWs functionalized with antibodies through phos-45 detection limit by electrostatically adsorbing complementary AuNP-decorated SnO₂ NWs provide enhanced sensing 45

6.1.2. Covalent Functionalization of Inorganic Semiconductor NWs

modification of Si NWs with functional groups (amines, often etched away, and the exposed surface is covalently 58 thiolation, or arylation.^[158] 58 59 59

1 phone's near-field communication (NFC) by cutting the One of the earliest reports on Si-based FET sensing 1 18 **A** typical method for functionalizing NWs involves 18 One of the earliest reports on Si-based FET sensing 2 circuit of a passive sensor tag and reconnecting it with described pH monitoring by APTES-functionalized NWs,^[133] 2 3 chemiresponsive SWCNTs. The same type of NWs were found to respond to 3 trinitrotoluene (TNT) in sub-fm amounts in solution and 4 4 sub-ppt concentrations in air through formation of Meisen-5 5 6 **6. Functionalization Methods for Applications** heimer complexes.^[45] Si NWs modified with thiol groups 6 $\frac{1}{2}$ showed a response to Cd²⁺ and Hg^{2+ [159]} Peptides were $\frac{1}{2}$ 8 NWs display useful intrinsic sensor properties, but func- attached to Si NWs and used in an array to detect Cu^{2+} and 8 9 tionalization is generally necessary to improve the process- Pb^{2+} in low nm concentrations,^[160] and Na⁺ was detected by Si 10 ability, sensitivity, selectivity, operating conditions, and sta- NWs functionalized with crown ethers.^[46] Volatile organic 10 11 bility. bility. Compounds (VOCs) and small molecules (NH₃, acetone, 11 12 NMe₃, acetic acid) were detected orthogonally by oligopep- 12 13 tides appended to Si NWs.^[161] Many biosensors were devel-14 **6.1. Functionalization of Inorganic Semiconductor NWs** oped by appending receptors to Si NWs,^[162, 163] such as biotin 14 15 6.1.1. Noncovalent Functionalization of Inorganic Semiconduc-

for streptavidin recognition,^[164] antigens for recognition of 15 $\frac{1}{6}$ **tor NWs their respective antibody**,^[137,165] and DNA strands for the $\frac{1}{6}$ 17 recognition of complementary oligonucleotides.^[166] 17

6.2. Functionalization of Metal Oxide NWs 6.2.1. Compositional Mixtures with Metal Oxide NWs

 34 functionalization of NWs with receptors possessing high non-enzymatic determination of biologically important mol- 34 35 selectivity (enzymes or antibodies).^[153] Glucose oxidase was ecules. CuO NWs have been used as the active sensing 35 36 immobilized in a ZnO NW matrix on top of a GaN transistor. component to determine glucose concentration in concert 36 37 In the presence of glucose, the enzyme catalyzes the with a Nafion film^[179] and in a Cu-CuO NW composition.^[180] 37 38 formation of gluconic acid and peroxide, which translates /In both cases, the NWs electrocatalyze the oxidation of 38 39 into a change in the charge on the NWs, thereby allowing for glucose; both devices display high sensitivity and linearity in 39 40 real-time glucose monitoring with a LOD of 0.5 nm.^[154] the biological range of blood glucose (3–50 mm) and environ- 40 4 Approaches have also been reported for the detection of mental stability. Electrochemically synthesized Cu₂O NWs 4 42 biomarkers such as prostate-specific antigen (PSA) by using have been employed with Nafion to determine the concen-42 44 phonic acid binding.^[155] DNA sensing was achieved at a 10 pm 5.0 mm) with a low LOD (0.12 μ m).^[181] 44 Metal oxides possess many desired properties, but rarely and carrier density can be modulated by preparing mixed-CuO NWs allows the detection of $NH₃$ at room temperature.[178] The presence of highly environment sensitive nano- 32 of the NWs.^[152] sized p-n junctions was given as an explanation for the 32 33 Specific detection of biological species is achieved by the sensitivity. Copper oxide NWs have garnered attention for the 33 non-enzymatic determination of biologically important molecules. CuO NWs have been used as the active sensing component to determine glucose concentration in concert with a Nafion film^[179] and in a Cu-CuO NW composition.^[180] In both cases, the NWs electrocatalyze the oxidation of glucose; both devices display high sensitivity and linearity in the biological range of blood glucose (3–50 mm) and environmental stability. Electrochemically synthesized Cu₂O NWs have been employed with Nafion to determine the concen- 43 In₂O₃ NWs functionalized with antibodies through phos- tration of H₂O₂ over a wide and relevant range (0.25 µm to 43 5.0 mm) with a low LOD (0.12 μ m).^[181]

46 ssDNA onto Si NWs functionalized with amine side chains.^[156] performance for NO₂^[182] and aromatic gases vapors? (i.e. 46 52 If an oxide layer is present, silane reactions allow the where the (doped or mixed) metal oxide is decorated with 52 54 aldehydes) for further functionalization.^[138] However, the In₂O₃ NWs functionalized with metal NPs (Au, Ag, or Pt). 54 55 passivating oxide layer lowers the sensitivity,^[157] so the SiO₂ is Used in an array at room temperature, the sensors were able 55 57 functionalized by hydrosilylation, halogenation, alkylation, with the LOD for CO being 0.5 ppm. $^{[186]}$ 57 AuNP-decorated $SnO₂$ NWs provide enhanced sensing performance for $NO₂^[182]$ and aromatic gases vapors? (i.e. 47 benzene, toluene).^[183] Similarly, SnO₂ NWs decorated with 47 AgNPs displayed improved sensitivity and selectivity for 48 49 **6.1.2. Covalent Functionalization of Inorganic Semiconductor** ethanol over other gaseous analytes,^[184] and PtNP-decorated 49 50 NWs Next structure $SnO₂$ nanofibers detected acetone in sub-ppm concentra- 50 films.^[185] More complex functionalization schemes exist 51 where the (doped or mixed) metal oxide is decorated with 53 modification of Si NWs with functional groups (amines, other nanostructures. A recent example reported Mg-doped 53 In_2O_3 NWs functionalized with metal NPs (Au, Ag, or Pt). Used in an array at room temperature, the sensors were able 56 often etched away, and the exposed surface is covalently to discriminate vapors of CO, ethanol, and H₂ at 100 ppm, 56 with the LOD for CO being 0.5 ppm.^[186]

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6.2.2. Covalent Functionalization of Metal Oxide NWs 1 1

for biofunctionalization. ZnO can be modified with carbox-APTES was used to functionalize ZnO NWs with uricase to GaN NWs are also amenable to organosilane chemistry, thereby allowing covalent functionalization to afford sensors with high stability.^[188] 2 and \sim 2

6.3. Functionalization of Carbon Nanotubes

CNTs offer some of the best opportunities for precise molecular assembly; however, they have considerable imprecision in structure and are mixtures with different diameters, chiralities, and lengths. They also have aperiodic functional groups (defects) that influence their properties. There is still optimal NW sensors. 21 21

Figure 10. Optimized structure of copper(I) scorpionate bound to 6,5-SWCNT. Reprinted from Ref. [193].

selectors and either MWCNTs or SWCNTs have been produced by solid-state mechanical mixing for chemiresis- 57 tors.^[98] This rapid prototyping method allows the screening of $NO₂$.^[208] CNT composites as sensor materials.[98] 56 produced by solid-state mechanical mixing for chemiresis- FET sensors with detection limits at the ppb level for NH₃ and 56 58 CNT composites as sensor materials.^[98] Vlandas et al. used the addition of phenyl radicals to 58

6.3.2. Noncovalent Functionalization of CNTs

3 Covalent functionalization has been investigated mostly Noncovalent modification is less invasive than covalent 3 5 ylic acid or amine functions for further functionalization.^[148] Waals interactions between molecules and CNTs. Noncova- $\overline{7}$ develop a FET sensor for uric acid in aqueous solution.^[187] achieved through solution-based mixing, in situ polymeri-Noncovalent modification is less invasive than covalent 4 for biofunctionalization. ZnO can be modified with carbox- functionalization as it relies on π interactions and van der 4 Waals interactions between molecules and CNTs. Noncova- δ APTES was used to functionalize ZnO NWs with uricase to lent interactions are often integral to CNT dispersions δ achieved through solution-based mixing, in situ polymeri- δ GaN NWs are also amenable to organosilane chemistry, zation, or melt mixing.^[194] Wei et al. showed the detection of δ Thereby allowing covalent functionalization to afford sensors trinitrotoluene (TNT) by using aminopyrene as a selector.^[195] 10 with high stability.^[188] Frazier and Swager used a trifunctional molecule containing 10 an aromatic unit (hexafluoroxylene), a selector unit (a 11 $\frac{12}{2}$ thiourea), and an anchoring unit (a silane; Figure 5)^[23] to $\frac{12}{2}$ 13 6.3. Functionalization of Carbon Nanotubes **Exercise 20** noncovalently functionalize CNTs with superior durability 13 14 and good sensitivity for the detection of cyclohexanone. 14

20 much to learn on how to best functionalize SWCNTs to create used in sensors to discriminate between different xylenes 20 15 CNTs offer some of the best opportunities for precise A common strategy is to use polymers. The active 15 16 molecular assembly; however, they have considerable impre- materials are typically obtained by drop-casting,^[27,196] dip-16 17 cision in structure and are mixtures with different diameters, coating,^[197] solution mixing,^[198] or in situ polymeri- 17 18 chiralities, and lengths. They also have aperiodic functional zation.^[199–201] CPs are excellent for dispersing SWCNTs.^[202] 18 19 groups (defects) that influence their properties. There is still Polythiophenes with receptor-based side chains have been 19 used in sensors to discriminate between different xylenes (Figure 11).[203]

Figure 11. Schematic view of the SWCNT/polymer sensor that selec-36 36

 \sim 42 \sim Covalent modification disturbs the π -electron system and 42 43 adds defects, but can increase the stability of dispersions. 43 Common side-wall reactions include dipolar cycloadditions 44 44 45 **1999 WINTER WELL AND STATE AND STATE WAY** with an azomethine ylide and reductive reactions with 45 $\frac{1}{2}$ aliazonium ions.^[194, 204–206] Covalent functionalization can also 46 47 be conducted at CNT termini. A common strategy relies on 47 ⁴⁸ a vidation to produce carboxylic acid moieties at defect sites, ⁴⁸ A 9 49 which can be further functionalized. Weizmann et al. dem- 49 onstrated the exclusive regioselective functionalization of 50 50 51 Figure 10. Optimized structure of copper(I) scorpionate bound to 6,5. SWCNT termini for the detection of single-stranded DNA 51 52 SWCNT. Reprinted from Ref. [193]. \sim (ssDNA) using a chemiresistor with detection limits of 10 fm 52 and discrimination of single, double, and triple base-pair 53 53 $54 \text{ mismatches (Figure 12)}$.^[207] Oxidation at the end of the tube 54 55 selectors and either MWCNTs or SWCNTs have been followed by polymer modification has also been used to build 55 $\mathrm{NO}_2.^{[208]}$

demonstrate the covalent functionalization of SWCNT side 59 59

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Figure 12. DNA-CNT NWs hybridized with an HRP probe for visualization of a DNA junction. Reprinted from Ref. [207] with permission. Copyright 2011 American Chemical Society.

 17 walls with boronic acids for the detection of glucose at 5- 6.4.3. Covalent Functionalization of CP NWs 17 18 30 mm.^[209] Huang et al. utilized a similar approach to func-

18 tionalize double-walled CNTs (DWCNTs) with aromatic 20 carboxylic acids as a selective sensor for NH_3 ^[210] The solubility, processability, and sensing. These modifications 20 DWCNTs allowed higher degrees of functionalization compared to SWCNTs. Our group has demonstrated the covalent 23 functionalization of SWCNTs by thermal aziridination to patible receptors onto polymers to enhance the stability 23 24 introduce amino groups that were further functionalized with under aqueous conditions.^[230] Polypyrrole (PPy) is particu-
24 25 hydrogen-bond donors for the detection of cyclohexanone larly amenable to covalent functionalization because the 25 26 and nitromethane.^[96] The covalent functionalization of nitrogen atom can be functionalized. NWs functionalized with 26 27 MWCNTs by using a modular zwitterionic functionalization PPy-NTA (nitrilotriacetic acid) chelators were found to 27 strategy allowed the formation of diverse densely functionalized materials. Arrays of these materials were used for the 19 tionalize double-walled CNTs (DWCNTs) with aromatic Covalent modifications have been used to improve 19 2 DWCNTs allowed higher degrees of functionalization com- include side-chain, copolymer, and graft functionalization.^[120] 2 22 pared to SWCNTs. Our group has demonstrated the covalent Many schemes have been developed to immobilize biocom-22 28 strategy allowed the formation of diverse densely function- detect Cu²⁺ cations at sub-ppt concentrations, and the Cu²⁺ 28 29 alized materials. Arrays of these materials were used for the complex could be used to detect His-tagged proteins.^[231] 29

6.4. Functionalization of CP NWs

As discussed earlier, CPs are not, by our definition, intrinsically NWs. However, they can be used to create NWs by templated synthesis or assembled in composite structures with nanofibers or CNTs. 38 with nanofibers or CNTs. 38 bacterial spores,^[235] or human serum albumin.^[236] 38

\sim 6.4.1. Physical Mixtures of CP NWs \sim 40 and the set of CP NWs

42 Composites with CPs can produce materials with superior 42 43 properties, such as high selectivity and sensitivity, enhanced The last two decades have produced many innovations in 43 44 resistance to humidity, low detection limits, low sensing nanofabrication, and we are in an opportunity-rich environ-44 45 temperatures, and enhanced stability.^[212] The rational design ment for the creation of functional NW sensors. The sensor 45 46 of materials that combine CPs with nonconductive poly- designer must continue expanding the range of options to 46 47 mers,^[213,214] carbon-based materials,^[215–218] metal nanoparti- tackle problems of selectivity, drift, sensitivity, and stability. 47 48 cles^[219,220] and oxides,^[221] or biological materials^[222,223] has Improved understanding of the basis of analyte detection in 48 49 been reported. CPs can serve as a matrix for a secondary NW sensors is needed to guide this development. We suggest 49 material or can be decorated with nanostructures in an 50 material or can be decorated with nanostructures in an areas of need and opportunities for the inspired sensor 50

52 Polyaniline (PANI) has been widely investigated and 52 53 combined with carbon-based materials^[226] such as $CNTs^{[215,216]}$ 54 or metal oxides.^[221] "One-pot" procedures are used where **7.1. Improving Selectivity** 54 $55\overline{\smash{\big)}\,}$ aniline is oxidatively polymerized in the presence of CNTs.^[218] blending materials together avoids complex syntheses, and composite materials may have improved morphology, parti-

 \overline{Aq}^+ 1.22 tion coefficients toward analytes, swelling behavior, as well as mechanical or conductive properties. An additive can also 2 2 **help maintain the properties of the polymer and increase the** 3 $\frac{1}{4}$ lifetime.^[120] Polymers enable the production of electrospun 4 \mathcal{F} \mathcal{A} $\mathcal{G}^{\mathcal{O}}$ high surface area nanofibers for gas sensors with enhanced 5

10 Noncovalent functionalization can be accomplished by 10 the deposition of NPs onto the surface of CP fibers. For 1 $\frac{12}{2}$ **Eigure 12** DNA CNT NW_s by byterized with an HPB probe for visual example, AgNP-decorated PEDOT NWs were found to $\frac{12}{2}$ 13 ization of a DNA junction. Reprinted from Ref. [207] with permission. detect NH_3 with a detection limit (1 ppm) that was fivefold 13 14 Copyright 2011 American Chemical Society. I lower than that of pristine PEDOT NWs,^[228] and AuNP- 14 15 decorated PANI NWs show an excellent response to H_2 S.^[229] 15

6.4.3. Covalent Functionalization of CP NWs

30 detection of a number of volatile organic compounds.^[211] Polypyrrole (CPPy) NWs functionalized with carboxylic 30 solubility, processability, and sensing. These modifications patible receptors onto polymers to enhance the stability under aqueous conditions.[230] Polypyrrole (PPy) is particularly amenable to covalent functionalization because the nitrogen atom can be functionalized. NWs functionalized with PPy-NTA (nitrilotriacetic acid) chelators were found to Polypyrrole (CPPy) NWs functionalized with carboxylic 31 acid groups were found to improve the immobilization onto 31 APTES-functionalized surfaces through covalent linking, 32 32 33 **6.4. Functionalization of CP NWs** with the remaining carboxylic acid moieties available for 33 34 bioconjugation. FET sensors based on this scheme were 34 35 As discussed earlier, CPs are not, by our definition, developed for proteins.^[232] The nitrogen position of PPy was 35 36 intrinsically NWs. However, they can be used to create NWs functionalized with biomolecules, and the materials were used 36 37 by templated synthesis or assembled in composite structures to detect cancer antigens (CA125),^[233] bacteriophages,^[234] 37

7. Future Developments 41 41

5 organized manner.^[130,224,225] exercises researcher. 51 The last two decades have produced many innovations in nanofabrication, and we are in an opportunity-rich environment for the creation of functional NW sensors. The sensor designer must continue expanding the range of options to tackle problems of selectivity, drift, sensitivity, and stability. Improved understanding of the basis of analyte detection in NW sensors is needed to guide this development. We suggest researcher.

7.1. Improving Selectivity

56 For example, PANI-SWCNT composites are sensors for NH₃ To many critics, a fundamental limitation is that chemical/ 56 57 and HCl.^[218] Compared to chemically modified polymers, biological sensors often lack the ability to identify an analyte 57 To many critics, a fundamental limitation is that chemical/ biological sensors often lack the ability to identify an analyte 58 blending materials together avoids complex syntheses, and unambiguously.^[237] It is true that high-resolution mass spec- 58 59 composite materials may have improved morphology, parti- trometry can give near-perfect identification of small, readily 59

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volatilized molecules. Even so, front-end separation by gas or liquid chromatography is often necessary. There has been massive investment in creating portable spectrometers, but these systems generally sacrifice precision for portability and provide useful information at a fraction of the cost by eliminating the need for expensive electronics, power supplies, and physical structures. 1 volatilized molecules. Even so, front-end separation by gas or pling of biological recognition/catalysis, and nanostructures.

Broad adoption of NW chemical/biological sensors will a combination of innovations in the integration of molecular and the dimensionality of sensing data.

7.2. Mitigating Sensor Drift

The effects of drift can be mitigated through data manipulation^[29,30] such as baseline correction or reference 21 device normalization but remains a fundamental limitation optimal fields and frequencies. 21 22 that hinders adoption in applications, especially for continu-

22 23 ous monitoring and for ultralow-cost devices without the 23 24 power or computational budget to execute these techniques. 7.4 . Mechanistic Analysis of Responses 24 19 The effects of drift can be mitigated through data positioning (orienting) of NWs, controlling the electrical 19 20 manipulation^[29,30] such as baseline correction or reference transport and dielectric coupling between NWs, and choice of 20

The key to minimizing thermally induced drift is devel-25 25 26 oping new techniques that limit nanoscopic rearrangements Ideal designs exploiting the aforementioned mechanisms 26 27 of sensor components in thin films. Covalent methods, such as require comprehensive understanding of their contributions 27 cross-linking, that bind NWs to themselves or the substrate 29 should continue to be developed, and matrices may also tion of sensor-response contributors is a challenge that holds 29 decrease the drift and improve device lifetimes. 28 cross-linking, that bind NWs to themselves or the substrate to NW sensor responses, which remains elusive. Deconvolu- 28 30 decrease the drift and improve device lifetimes. great promise for the field. In addition to informing rational 30

When the NW transducer and selector are distinct, drift 32 can be minimized by strengthening the binding of the selector NW sensors, the ability to extract multidimensional informa- 32 to the NW to mitigate phase segregation. Tethering can be 34 accomplished by covalent attachment^[96,211] or by increasing NW FETs and resonant circuits^[241] hold promise as device 34 35 the number or strength of noncovalent binding moieties.^[238] architectures capable of accomplishing this task, but advances 35 31 When the NW transducer and selector are distinct, drift design of molecular recognition and their implementation in 31 33 to the NW to mitigate phase segregation. Tethering can be tion out of a single NW sensor device is an attractive prospect. 33

Drift is also induced electrically and is dependent upon 37 the strength of the applied field. This contribution to drift can 37 be minimized by decreasing the voltage bias, performing 38 38 39 a medium-to-high voltage AC pretreatment to accelerate π , **5. Current and Emerging Applications** 39 40 equilibration processes, or using thermal or UV treatments to 40 um. Indeed, mucus protects the mammalian olfactory epitransport analytes to sensors.

7.3. Improving Sensitivity

Strategies for enhanced sensitivity include the following: 1) The design of molecular recognition elements where 56 analyte interactions are intimately coupled to carrier trans-
sensor will be in a durable or expendable form α ? 57 port/generation/depletion. Innovations here can be made by Next, one must consider how the analytes are likely to 57 bottom-up chemical designs by employing molecular recognition, designer NWs with engineered work functions, cou-54 Strategies for enhanced sensitivity include the following: power transfer is relatively new, but early reports are 54 55 / 1) The design of molecular recognition elements where promising.^[242,246–250] One must also consider whether the 55 58 bottom-up chemical designs by employing molecular recog- encounter the sensing element. Point surveillance is often 58 59 nition, designer NWs with engineered work functions, cou- sufficient for object-level monitoring, while area surveillance 59

 5 only offer incremental advances. In most cases, NW sensors been introduced as important mechanisms. A major oppor- \acute{o} will not provide superior identification. However, they tunity lies in placing responsive materials/molecules in the \acute{o} 11 require improvements in selectivity. Solutions will involve photodiodes. 3) Complex signals can be used to minimize 11 13 recognition as well as in increasing computational innovations need to be understood. Advances in capacitive sensing are 13 2 liquid chromatography is often necessary. There has been $\,$ 2) Developing sensors that leverage junctions between NWs $\,$ 2 3 massive investment in creating portable spectrometers, but and electrodes. Schottky barriers^[36, 103, 149] and swelling- $\frac{3}{2}$ 4 these systems generally sacrifice precision for portability and induced expansion of tunneling barriers^[113, 122, 216, 240] have $\overline{4}$ been introduced as important mechanisms. A major opportunity lies in placing responsive materials/molecules in the $\overline{7}$ provide useful information at a fraction of the cost by tunnel junction that can be chemically triggered to have $\overline{7}$ δ eliminating the need for expensive electronics, power sup-
resonant electronic states with the NWs. Rectifying interfaces δ P plies, and physical structures. The settlement of the sett 10 Broad adoption of NW chemical/biological sensors will amplification in analogy to gain produced by avalanche 10 photodiodes. 3) Complex signals can be used to minimize 12 a combination of innovations in the integration of molecular noise, and combinations of resistive and capacitive responses 12 need to be understood. Advances in capacitive sensing are 14 and the dimensionality of sensing data. possible by utilizing charge polarization along the length of 14 15 **NWs isolated by resistive junctions. Space–charge contribu-** 15 16 tions to a material's dielectric constant are much larger than 16 7.2. Mitigating Sensor Drift **Subset Constant Const** changes in chemical/biological sensors will involve careful 18 18 optimal fields and frequencies.

7.4. Mechanistic Analysis of Responses

Ideal designs exploiting the aforementioned mechanisms require comprehensive understanding of their contributions tion of sensor-response contributors is a challenge that holds NW sensors, the ability to extract multidimensional informa-NW FETs and resonant circuits^[241] hold promise as device architectures capable of accomplishing this task, but advances 36 Drift is also induced electrically and is dependent upon in multidimensional experimental design will be required. 36

7.5. Current and Emerging Applications

4 release trapped charges. Devices that operate under instanta-
The first consideration when designing a NW sensor 4 42 neous applied voltage instead of a static electric field should should be its environment. Common environmental problems 42 43 drift less per unit time; passive radiofrequency (RF) devices include temperature, humidity, interferents, EM effects, and 43 44 are a promising platform. Less drift should lead to longer biofouling. One must also consider how data will be retrieved 44 45 device lifetimes. It may also be possible to mitigate drift by and how often, which leads us to consider power requirements 45 46 creating dynamic fluid environments that maintain equilibri- and sensor stability. If the sensor operates for a long period 46 48 thelium,^[239] and designer fluids could selectively partition and termined data logging time interval may be optimal. Con-48 The first consideration when designing a NW sensor should be its environment. Common environmental problems include temperature, humidity, interferents, EM effects, and biofouling. One must also consider how data will be retrieved and how often, which leads us to consider power requirements and sensor stability. If the sensor operates for a long period 47 um. Indeed, mucus protects the mammalian olfactory epi- and operates passively, then a coin-cell battery and a prede- 47 termined data logging time interval may be optimal. Con-49 transport analytes to sensors. The versely, if data are required on demand, a larger on-board 49 power source may be required. Novel approaches to powering 50 50 sensors include triboelectric methods,^[242–245] remote photo- or 51 52 7.3. Improving Sensitivity **Superintensity** thermal power harvesting, and resonant inductive coupling. 52 The concept of coupling NWs into devices capable of wireless 53 sensor will be in a durable or expendable form \bullet ok?

Next, one must consider how the analytes are likely to

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Sensors

1 may be necessary for situational awareness in a large 3D 2 space. Area surveillance requires more than one sensor and, $[1]$ F.R. Baptista, S.A. Belhout, S. Giordani, S. J. Quinn, Chem. $\frac{2}{3}$ 3 therefore, the total cost of goods should be considered. $\frac{3606 \text{ KeV}}{121 \text{ N}} \times \frac{1215 \text{ MeV}}{121 \text{ MeV}} \times \frac{3606 \text{ MeV$

5 sensor design. Analytes are categorized as VOCs, oxidizers $\frac{1}{21}$ D L Late Y-K Huang B Liu L Acharya S N Shirodkar L $\frac{1}{2}$ and reducers, particulates, biological macromolecules, and $\frac{1}{2}$ Luo, A. Yan, D. Charles, U. V. Waghmare, V. P. Dravid, et al., 7 viruses and bacteria. The ability to detect VOCs continues to $ACS Nano$ **2013**, 7, 4879–4891. improve as molecular design principles are introduced and P coupled to NWs. Oxidizers and reducers are often detected by *Interfaces* **2015**, 7, 7809–7832. 10 exploiting their influence on carrier concentrations and the $[5]$ B. Cho, M. G. Hahm, M. Choi, J. Yoon, A. R. Kim, Y.-J. Lee, S. Schottky barrier. Greater efforts are needed to detect G. Park, J.-D. Kwon, C. S. Kim, M. Song, et al., Sci. Rep. 2015, 2 particulates because of their vast size, shape, and charge $\frac{1}{16}$ I.S. Kim H W Yor H O Chai H T Jung Nano Latt 2014 13 dispersity. Biological macromolecules can be targeted by $\frac{10}{14}$ 5941 = 5947 14 leveraging nature's biomolecular machinery, but this is not [7] F.K. Perkins, A.L. Friedman, E. Cobas, P.M. Campbell, G.G. 14 15 a general design approach. Viruses and bacteria interact by Jernigan, B. T. Jonker, Nano Lett. 2013, 13, 668–673. 16 multivalent processes, and translating these collective pro- [8] Q. Zhou, T. M. Swager, J. Am. Chem. Soc. 1995, 117, 12593- 16 17 cesses into robust signals that are discernable from other 12602. 18 events is needed. Robust inexpensive trace virus and bacterial [9] D.C. Harris, *Quantitative Chemical Analysis*, W. H. Freeman, 18 19 detection has far-reaching implications for human health and $\frac{\text{New York, 2006}}{\text{[60] (4)}}$ safety. 8 improve as molecular design principles are introduced and [4] C. N. R. Rao, K. Gopalakrishnan, U. Maitra, [ACS Appl. Mater.](http://dx.doi.org/10.1021/am509096x) 8 20 safety.
²⁰ 2011 Participal and the found under http://www.compan.com/cont/acci¹/2012

The fidelity of information is of critical importance in $\frac{11.1 \times 10^{10} \text{ N}}{111 \times 10^{10} \text{ N}}$ and the land under http://www. 22 chemical/biological sensing. The vast majority of chemical $\frac{1}{2}$ che gov/niosh/regulations html 2013 23 sensors have low specificity, and practitioners often assume $\frac{12}{2}$ Exposure to chemical agents and chemical safety" can be 23 24 that more data from, for example, a large sensor array is found under https://osha.europa.eu/en/legislation/directives/ 24 25 better. However, Occam's razor is generally the best exposure-to-chemical-agents-and-chemical-safety/osh-related- 25 26 approach, with inspired designs that produce high specificity aspects/council-directive-91-414-eec, 2015. 27 in individual sensors. Such sensors can serve their purpose $\begin{bmatrix} 13 \end{bmatrix}$ R.J. Chen, N.R. Franklin, J. Kong, J. Cao, T. W. Tombler, Y. 27 28 without an unnecessarily expensive or cumbersome design. $\frac{208 \text{ m/s}}{142 \text{ s}}$ ang, H. Dai, Appl. Phys. Lett. 2001, 29, 228

30 cations. However, for many situations where crossing a thresh-
 $\frac{276}{151}$ F.S. Snow F.K. Perkins, F.J. Houser, S.C. Badescu, T.J. 31 old is relevant, an irreversible dosimeter can be employed

Reinecke Science 2005, 307, 1942-1945. 32 that conveys information about the history of the device. [16] C.-J. Chu, C.-S. Yeh, C.-K. Liao, L.-C. Tsai, C.-M. Huang, H.-Y. 32

34 a single sensor or device are beginning to emerge.^[251] By $2564-2569$. 34 35 combining multivariate extraction of orthogonal parameters [17] H.Y. Yue, S. Huang, J. Chang, C. Heo, F. Yao, S. Adhikari, F. 35 36 with statistical techniques such as PCA, discrimination Gunes, L.C. Liu, T. H. Lee, E. S. Oh, et al., ACS Nano 2014, 8, 36 $\frac{37}{20}$ between analytes with an array is possible.

There is a new sensor paradigm on the horizon. The trend $\frac{1}{2}$ and $\frac{1}{2}$ $\frac{3}{2}$ is moving from discrete comprehensive data collection to $\frac{1}{19}$ Handbook of Membrane Separations: Chemical Pharmaceut-40 continuous, parsimonious data collection. Such a move will *ical, Food, and Biotechnological Applications* (Eds.: A.K. 40 41 require the deployment of wireless distributed sensor net-
Pabby, S. S. H. Rizvi, A. M. Sastre), CRC, Boca Raton, 2009. 41 42 works that are linked directly to cloud storage. Chemists, [20] S.T. Hobson, S. Cemalovic, S.V. Patel, Analyst 2012, 137, 42 43 material scientists, physicists, and practitioners will need to $1284-1289$. 43 44 work together to develop new modalities that minimize cost $[21]$ C.J. Lu, E. T. Zellers, Anal. Chem. 2001, 73, 3449–3457. 45 per sensor and cost per sensed event. Additionally, oppor- $[22]$ F. Blanco, X. Vilanova, V. Fierro, A. Celzard, P. Ivanov, E. 45 46 tunities will unfold for fusing chemical information with other 46 $\frac{1}{20}$ and $\frac{1}{2}$ and $\frac{1}{$ 47 inputs to derive new insights about our environment and $\frac{B2000, 122, 50-50}{121 \text{ K M}}$ Frazier T M Swager Anal Chem 2013 85 7154-7158 47 behavior. dependence Sci. 2002, 245, 311 – 48

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Nanowire Chemical/Biological Sensors: Status and a Roadmap for the Future

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