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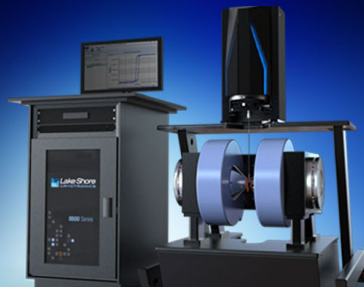
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Field-effect-based chemical sensing using nanowire-nanoparticle hybrids: The ion-sensitive metal-semiconductor field-effect transistor

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A new class of nanoscale devices called ion-sensitive metal-semiconductor field-effect transistors (nano-IS-MESFET) for sensing applications is reported. Nanoparticle-nanowire hybrids with active metal-semiconductor regions are operated as ion-sensitive field-effect transistors (ISFETs) in liquids, where 0D metal gates induce quasi-spherical charge depletion regions in 1D transport channel producing stronger field-effects. As a proof-of-concept, we present ZnO nanowire-Pd/Au nanoparticle IS-MESFETs that show increased transconductance in comparison to ZnO nanowire ISFETs. As demonstrated further, ISMESFETs may also provide strategies for site-specific immobilization of receptor molecules paving way towards a novel electrical biosensing platform operable at low voltages with improved selectivity and sensitivity. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4775579>]

One-dimensional nanostructures (1D-NS) such as nanowires (NWs) and nanotubes are proposed as active components of various kinds of electrical devices and circuits.^{1–3} The use of 1D-NS-based field-effect devices for sensing chemical and biological species in solution is an attractive route for the design of highly sensitive detection platforms due to the high surface-to-volume ratio of 1D-NS.⁴ Field-effect transistors as sensors in liquids were first demonstrated in the form of ion-sensitive field-effect transistors (IS-FETs) for measuring solution pH.^{5,6} With the drive for miniaturization and advent of new process technologies and surface chemistries, numerous prototype 1D-NS-based sensing platforms for label-free electrical detection have been demonstrated in recent years.^{7–14} Most of these devices have been predominantly based on the principle of an ISFET either in a direct or an indirect manner. Two key factors that determine the performance of such devices are sensitivity and selectivity. Sensitivity is related to device characteristics such as transconductance and field-effect mobility.¹⁵ Common methods for improving the sensitivity include the use of different geometries of gate around NWs.^{16–18} However, inaccessibility of the NW surface for sensing, diminished surface-to-volume ratio, and inaptness for electrochemical gating render these devices unsuitable for chemical sensing applications. On the other hand, selectivity is determined by the ability to controllably attach analyte-sensitive receptors on the nanowire surface. Silanes are the most common molecules used to tune the properties of ZnO and other NW-based devices in a deliberate way for many applications.^{19,20} Cross-linking silanes are used to form a linker self-assembly on NW surfaces in order to attach receptor molecules subsequently.^{21,22} Although novel silanization methods allow the

efficient functionalization of hydroxylated nanowire surfaces, these protocols inevitably also lead to the modification of the underlying SiO₂ or glass substrate, which are also rich in similar functional moieties such as hydroxyl groups. The resulting high density of receptors also in areas that do not form the active element of the device may cause concerns over the loss of sensitivity, when operating in liquids.¹⁵ In this work, we aim at an exclusive functionalization of the nanowire surface.

Here, we demonstrate a new device concept utilizing nanowire-nanoparticle (NW-NP) hybrids, whereby the NPs present a versatile handle to tune both the sensitivity and selectivity of 1D-NS-based chemical and biological sensors. Specifically, the devices comprise of ZnO NWs that are decorated with Pd or Au NPs. The metal nanoparticle-semiconducting nanowire ensemble contacted by electrodes works as a metal-semiconductor field-effect transistor (MESFET). When deployed using an electrochemical gate, this device combines an ISFET configuration to form an ion-sensitive metal-semiconductor field-effect transistor (IS-MES-FET). In such a device, the NPs have a dual role by functioning both as efficient gates to improve the FET characteristics in liquid, while at the same time allowing for the attachment of analyte-specific receptors on their surface. The general principle of a nano-IS-MES-FET is shown in Figure 1 along with the experimental setup for its operation. Here, the active element is a semiconducting nanowire (*s*-NW), which is contacted by source and drain electrodes. Metal NPs (*m*-NPs) deposited on the surface of the nanowire act as gates analogous to the design of a MESFET. However, the gating itself is realized remotely using an Ag/AgCl reference electrode that is placed in contact with the solution, similar to the operation of an ISFET. The ion-sensitive membrane is realized using receptors that are chemically coupled to the *m*-NPs.

Until now, NW-NP hybrids have been demonstrated by fabrication of electrical devices based on Si NWs doped with metal NPs during CVD growth,²³ decorating ZnO or GaN nanowires with metal clusters using simple sputtering,^{24,25} or plasma enhanced CVD.^{26,27} Alternatively, the *m*-NPs may

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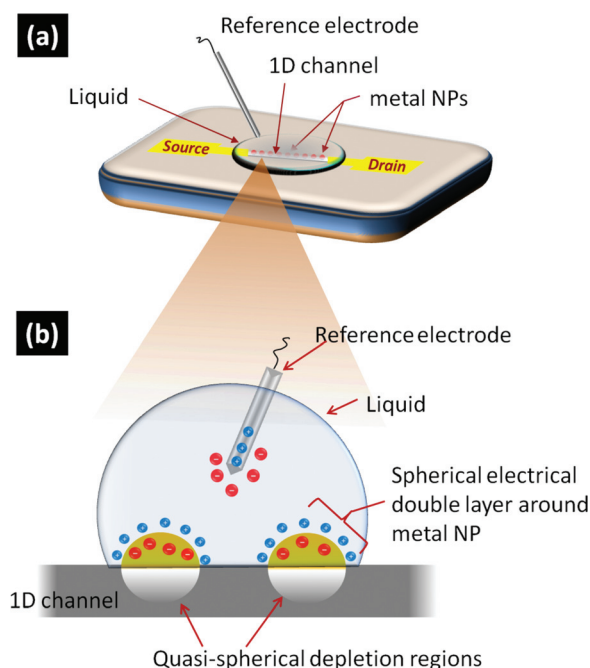


FIG. 1. Ion-sensitive metal semiconductor field-effect transistor: Schematic showing (a) the experimental setup and (b) the device architecture along with the principle of operation. Contacted NWs are decorated with metal NPs (*m*-NPs). The NPs function as gates whose potential is tuned by varying the voltage at a Ag/AgCl reference electrode. In (b), a schematic representation of the spherical double layer and the quasi-spherical depletion region is also shown.

be synthesized separately and subsequently self-assembled on the surface of the nanowires.²⁸ Here, we demonstrate a novel approach for the attachment of *m*-NPs on to ZnO NWs through electrochemical reduction of the desired metal salt. Electrochemical modification is a versatile technique that allows for the functionalization of nanostructures with a wide range of receptors from NPs to biomolecules, as has been demonstrated on carbon nanotube (CNT) and graphene

surfaces.^{29,30} Until now, this functionalization strategy has been seldom used on inorganic 1D-NS. In comparison to directly functionalizing bare ZnO surfaces, the use of NPs provides for a higher level of versatility and selectivity for the exclusive attachment of receptors on to the active area of the sensor surface. NPs are reported to be favorable candidates for attaching a variety of biomolecules.^{31,32} NPs also exhibit high efficiency for charge transduction that is important for translating analyte binding events into an electrical signal.^{33,34} Using NP anchors as a general strategy for immobilization of receptors in electrical devices may allow for developing a wide spectrum of label-free chemical sensors and biosensors.

From a device perspective, NW-NP hybrids have until now mainly been experimented for gas sensing, whereby the analyte gases bring some changes in the energy landscape of the NPs thereby locally affecting the resistance of the nanowire.³⁵ Here, we demonstrate for the first time that such NW-NP hybrids operating as an IS-MES-FET can be deployed as sensors in liquids. MESFETs based on unmodified CdS nanowires operating in air have been demonstrated wherein a microelectrode that is in contact with the nanowire serves as the gate electrode.³⁶ In general, MESFETs show improved gating efficiency, possibility to have a good control over the threshold voltage and operation at high frequencies.³⁷ Here, we combine the advantages of electrochemical functionalization and MESFET device architecture to show that ZnONW-NP hybrids can be operated as FETs in liquids. Use of NPs as metal gate in liquids improves the field-effect characteristics of ZnO NW ISMESFETs in comparison to bare ZnO NW devices. The device characteristics such as ON/OFF current ratios and transconductance values of ZnO ISMESFETs compare favorably with the best values reported for ZnO NW devices in different architectures.^{38–41}

Our approach starts with the realization of site-specific ZnO NW transistors using a solution-based method, leading

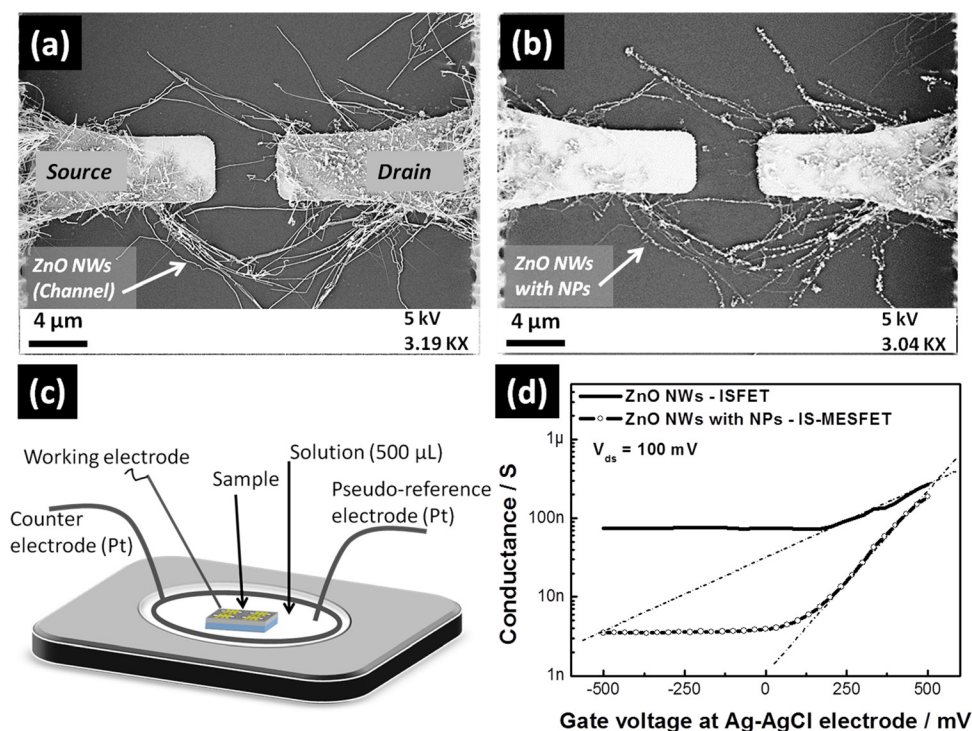


FIG. 2. ZnONW-PdNP-MESFETs – Fabrication and electrical characteristics. (a) and (b) SEM images of a typical ZnO nanowire device before (a) and after (b) electrochemical deposition of palladium NPs. (c) Schematic of the electrochemical set-up used for deposition of *m*-NPs. The ZnO NWs are functionalized through electrochemical reduction of 2 mM Na_2PdCl_6 in an aqueous solution with 0.1 M LiClO_4 as background electrolyte. (d) Field-effect characteristics of the device measured in the configuration of Figure 1 showing conductance at $V_{ds} = 100$ mV as a function of gate voltage at the reference electrode before (smooth black curve) and after (dotted curve) deposition of Pd NPs. The smooth black curve shows a weak field-effect in liquid gate (with conductance modulation less than an order of magnitude), while dotted black curve shows a clear improvement in field-effect.

to few nanowires in an electrode gap (Figure 2(a)).⁴² *m*-NPs (Au or Pd) are selectively deposited on the contacted nanowires through electrochemical reduction of an appropriate metal salt in solution (Figure 2(b)). The electrochemical cell used for performing this functionalization is shown in the schematic in Figure 2(c). Further details about the fabrication are presented in the supporting information.⁵¹ Figure 2(d) presents typical electrical transport characteristics in a citrate phosphate buffer solution (pH 8) measured in the configuration of Figure 1. Here, the conductance of the device is recorded as a function of the gate voltage (at the reference electrode) without (smooth black curve) and with (dotted black curve) Pd NPs. It is apparent that the sub-threshold swing of the FET characteristics shows a clear increase after the attachment of NPs, with an improved ON-OFF ratio. The conductance can be modulated by up to 2 orders of magnitude. This gives a first indication that the NPs play an important role in controlling the transport through the ZnO NW-NP hybrids. An ON/OFF ratio of 2 orders of magnitude appears to be somehow inferior to that obtained on other nanowire devices.^{38–42} This can, however, be improved by deploying VLS or CVD grown nanowires.^{19,20,38–41}

In order to obtain further confirmation for the gating occurring at the NPs, we selectively modify the surface characteristics of the deposited NPs with the help of thiolated receptors. A schematic of this functionalization procedure is shown in Figure 3(a). Specifically, the NP-NW hybrid devices are left in a 1% ethanol solution of substituted thiophenols for 90 min. This leads to the attachment of the desired end-group (denoted as X in Figure 3(a)) on the NPs surface through disulphide linkages.^{43–45} The devices are characterized in solutions of two different pH. Figures 3(b) and 3(c) show the electrical characteristics of a typical sample before and after functionalization with *p*-aminothiophenol (*p*-ATP, X = NH₂). While the unmodified IS-MES-FET shows little changes upon varying pH, the functionalized IS-MES-FET shows a clear shift in threshold voltage as a function of pH.

This can be understood by considering that the surface charge of bare NPs remains rather constant with small pH variations. On the contrary, hydroxyphenyl groups have an isoelectric point close to 9.9 thereby presenting a different charge distribution at the two pH values.⁴⁶ As a result of this, the threshold voltage shifts towards positive voltages with increasing pH. This shift is consistent with the direction of threshold shift observed for bare ZnO NWs that are directly functionalized with amino groups through silanization.⁴² This confirms the proposition that the field-effect in NW-NP hybrid devices is indeed dominated by the NPs. It is worth mentioning that the observed threshold shifts are much larger than the negligible hysteresis in field-effect characteristics seen in our devices. This is apparent from the transport curves for a typical sample shown in figure SI3 in the supporting information.

Further support for the improved gating due to the attachment of NPs is obtained by considering the statistics of transconductance values of various devices as shown in Figure 4. The transconductance values are extracted from the electrical characteristics of the device measured before and after attachment of Pd (Figure 4(a)) or Au (Figure 4(b)) NPs. It is apparent that the transconductance could be improved around an order of magnitude in most of the cases. The improvement in transconductance varies, however, from device to device. This could be attributed to the difference in the number of nanowires that are bridging the electrodes and the variation in the density of nanoparticles from sample to sample. We envision that by careful control of device fabrication and rigorous optimization of electrochemical parameters, it will be possible to obtain devices with more homogeneous device characteristics.^{47,48} The spatial variation of the chemical reactivity of the ZnO NWs due to defects may also play a role in the varying density of NPs as is known for carbon nanotubes.⁴⁹ The field-effect mobility (μ) values for the devices were calculated in a way similar to their ISFET counterparts given by $\mu = (dI_d/dV_{lg}) L / (c_{sdl} V_{ds})$, where I_d is the drain current, V_{ds} is the drain bias and V_{lg} is

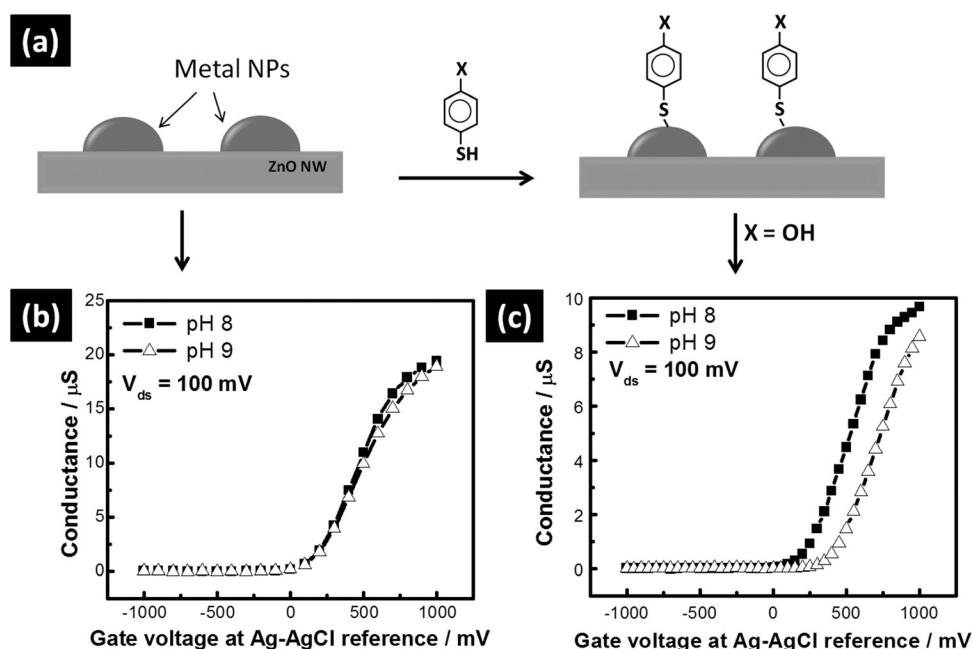


FIG. 3. Chemical functionalization of ZnONW-PdNP-IS-MES-FETs. (a) Schematic showing the functionalization strategy for the attachment of functional groups using substituted thiophenols. X corresponds to an arbitrary end-group. (b) and (c) IS-MES-FET characteristics in buffer solutions of pH 8 and pH 9 before (b) and after (c) functionalization with *p*-hydroxythiophenol (*p*-HTP); here X = OH. It is apparent that the bare IS-MES-FET is insensitive to pH, while the hydroxyfunctionalized IS-MES-FET shows a clear shift in threshold voltage towards positive gate voltages.

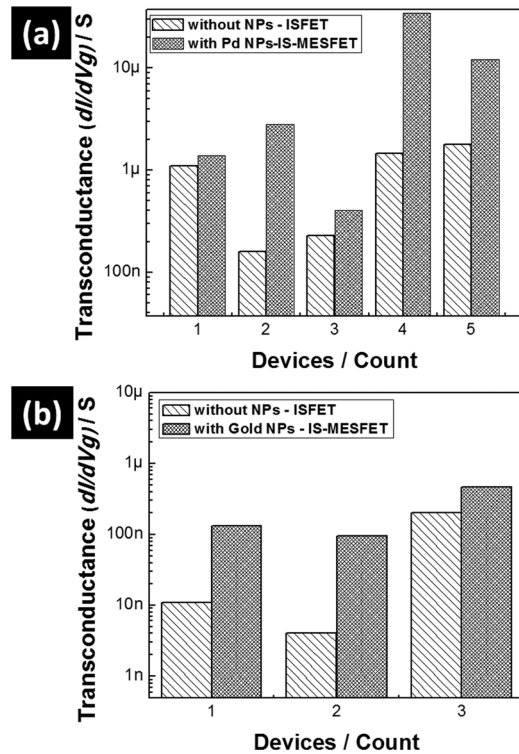


FIG. 4. Transconductance of ZnONW-NP-IS-MES-FETs. Distribution of transconductance values for Pd NP (a) and Au NP (b) decorated ZnO NW devices. The transconductance increases in all of the devices upon attachment of the NPs.

the liquid gate voltage, L is the channel length (2–5 μm in our samples), and c_{sdl} is the capacitance per unit area due to the spherical electrical double layer formed around m -NPs. The capacitance due to the electrical double layer (c_{edl}) would be a complex function of the cylindrical capacitances (at the nanowires) and the spherical capacitances (at the NP-NW interface). In order to obtain a rough estimate of the field-effect mobility, we assume here that the spherical capacitance dominates due to the strong coupling at the NW-NP interface. The field effect mobilities calculated in this way were as high as $7 \text{ cm}^2/\text{Vs}$ for such devices. Here, the spherical double layer capacitance is given by $c_{\text{sdl}} = 4 \pi \epsilon_r \epsilon_0$ (ab/a-b), where ϵ_r is the dielectric constant of the liquid (80 for water), “a” denotes the diameter of NPs and b is $(a + \lambda_D)$, λ_D being the Debye length as a function of ionic strength (around 0.5 nm for the phosphate-citrate buffer).

The improvement in the field-effect characteristics of NW-NP hybrids can be understood based on the Schottky model as presented in detail in the supporting information. In short, the presence of the m -NPs on the s -NWs leads to the formation of a Schottky barrier, which replaces the insulating oxide layer in a classical metal oxide semiconductor FET (MOSFET). The shape of the NPs enables the creation of a rather spherical depletion layer locally in the nanowires as shown in Figure 1(b). Furthermore, the double layer that is formed at the NP-liquid interface has the form of a spherical capacitor. By contrast, in bare ZnO-NW-ISFETs, the double layer is cylindrical and the depletion layer rather planar. We believe that the spherical double layer together with the quasi-spherical depletion layer provides for a higher capacitance and thereby an improved gating efficiency in the IS-

MES-FETs. The operation of our ISMESFETs is different from that of other NP-NW hybrids, where NPs do not form part of the total gate capacitance.²³

Next, we turn towards the use of IS-MES-FETs for chemical sensing. In principle, the pH-dependence shown in Figure 3(c) already gives an indication that such devices can indeed be deployed as sensors in liquids. In field-effect sensors, the sensitivity is determined by the FET characteristics such as field-effect mobility, ON-OFF ratio, and transconductance.⁵⁰ The possibility to improve these parameters in the case of IS-MES-FETs indicates that the NPs can be used as a handle to tune the sensitivity of 1D-NS-based field-effect sensors. In order to be deployable as sensors, the devices must also be selective to the analyte of interest. Here, we demonstrate indirectly that this is attainable by functionalizing the NP surface with different receptors. For this purpose, we modify the NP surface with two different receptors namely amino ($X = \text{NH}_2$) or hydroxyl groups ($X = \text{OH}$) using the same thiophenol-based functionalization strategy as outlined before. Figure 5(a) shows sensor responses obtained at the same pH for a ZnONW-PdNP-IS-MES-FET before and after functionalization with hydroxyl or amino groups. It is apparent that while for the amino groups, the threshold voltage shifts to positive voltages, and the hydroxyl groups bring a shift in the opposite direction. This

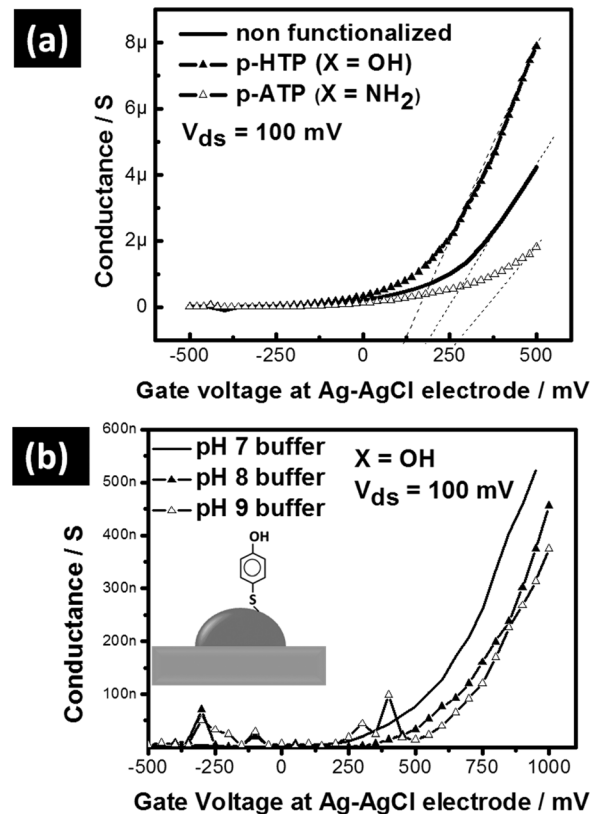


FIG. 5. ZnONW-PdNP-IS-MES-FETs as sensors. (a) Sensor response before (solid line curve) and after functionalization with amino ($X = \text{NH}_2$, line curve with hollow triangles) or hydroxyl ($X = \text{OH}$, line curve with solid triangles) groups in a buffer solution of pH 8. At this pH, the amino groups are predominantly positively charged, while the hydroxyl groups bear a net negative charge. This explains the shift in the threshold voltages in opposite directions. (b) pH response of an IS-MES-FET functionalized with p-hydroxythiophenol ($X = \text{OH}$), showing a threshold voltage shift as a function of pH.

is consistent with the isoelectric point of hydroxyl groups being lower than that of amino groups. This suggests that by functionalizing the nanoparticle surface with differing receptors that are specific to the analyte of interest, sensors with selectivity for a specific analyte can be realized. Furthermore, this confirms that the IS-MES-FET strategy constitutes an efficient mechanism for surface charge detection using NW-NP hybrids. This fact is underlined by the pH response of HTP-functionalized IS-MES-FETs shown in Figure 5(b), where a clear pH-dependence can be deciphered.

In summary, we have demonstrated a versatile electrochemical approach for the realization of ZnO nanowire-nanoparticle hybrids on-chip in a site-specific manner. This allowed us to realize a new device architecture in liquids namely the IS-MES-FET, showing improvements in field-effect performance. In order to realize sensors, an ion-sensitive layer could be obtained by facile functionalization of the nanoparticle surface through thiolated receptors. The NP surface can be used as a versatile anchoring point for the attachment of biomolecules thereby showing promise for a new class of on-chip biosensors.

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- ⁵¹See supplementary material at <http://dx.doi.org/10.1063/1.4775579> for Fabrication and characterization of ZnO ISMESFETs and the analysis of field-effect behavior of nanowires-nanoparticle hybrids using Schottky model.