

# Nitro-Aromatic Explosive Sensing Using GaN Nanowire-Titania Nanocluster Hybrids

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**Abstract**—Globalization of terrorist attacks in recent years calls for advanced technologies to detect the concealed explosives effectively. We demonstrate a highly sensitive and selective detection of traces of nitro-aromatic explosive compounds by functionalizing gallium nitride (GaN) nanowires with titania (TiO<sub>2</sub>) nanoclusters to address this issue. The ultraviolet light photo-modulated GaN/TiO<sub>2</sub> nanowire-nanocluster (NWNC) hybrids show remarkable selectivity to benzene and related aromatic compounds, with no measureable response for other analytes at room temperature. This paper presents the sensing characteristics of GaN/TiO<sub>2</sub> NWNC hybrids towards the different aromatic and nitroaromatic compounds at room temperature. The GaN/TiO<sub>2</sub> hybrids are able to detect trinitrotoluene (TNT) concentrations as low as 500 ppt in air and dinitrobenzene concentrations as low as 10 ppb in air in approximately 30 s. The noted sensitivity range of the devices for TNT is from 8 ppm down to as low as 500 ppt. The detection limit of dinitrotoluene, nitrobenzene, nitrotoluene, toluene and benzene in air is 100 ppb with a response time of ~ 75 s. The devices showed very sensitive and selective response to TNT when compared to interfering compounds like toluene with good reproducibility. Integration of different NWNC hybrids on the same substrate could lead to tiny, highly sensitive, selective, low-power and smart explosive detectors that could be manufactured cheaply in large numbers.

**Index Terms**—Explosives, functionalized nanostructures, GaN nanowires, nanoclusters, nanowire-nanoclusters, nitro-aromatics, photoconductivity sensors, trinitrotoluene (TNT).

## I. INTRODUCTION

**I**N RECENT years, there is an increased threat of explosive attacks worldwide, as the explosive-based weapons are

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easy to make, obtain and deploy [1], [2]. Detecting traces of explosives is a challenging task because of the low vapor pressures of most explosives [3]. The difficulty of the standoff explosive detection task is aggravated by reasons such as the noisy environment which can mask the signal from the explosive, the potential for high false alarms, and the need to determine a threat quickly so that action can be taken [4]. Trained canine teams remain as the most reliable means for the detection of explosive vapors to date; however, dogs are expensive to train, easily tired, and cannot be deployed for round the clock surveillance. Though ion mobility spectroscopy (IMS), which is used in airports for explosive detection, is highly sensitive, it still has limitations of being expensive, bulky, operator-dependent and is prone to false alarms. Nanotechnology offers a viable solution for trace detection of explosive compounds, by the use of nano-structured materials with high surface to volume ratio. Effective protection against explosive-based terrorism can be achieved by large-scale production and deployment of nano-sensor arrays that are inexpensive, highly sensitive and selective with low response and recovery times. In the present study, we demonstrated the selective response of GaN nanowire/TiO<sub>2</sub> nanocluster hybrids to the nitroaromatic explosives, including trinitrotoluene (TNT), dinitrotoluene (DNT), nitrotoluene (NT), dinitrobenzene (DNB) and nitrobenzene (NB) at room temperature. The sensor detected between 0.5 ppb and 8 ppm TNT with good selectivity against interfering compounds like toluene. The sensitivity of 1 ppm of TNT is 10% with response and recovery times of ~ 30 s. This study focuses on a novel fabrication methodology, which resulted in improved sensing performance characteristics at room temperature and room ambient conditions.

Although the semiconductor solid-state sensors are readily favored over direct analytical techniques like optical/mass spectroscopy and gas chromatography, they have significant limitations in terms of sensitivity, selectivity, and reliability [5]. It is well known that due to their high surface-to-volume ratio, the electrical and physical properties of nanoparticles and nanowires change significantly in response to their environments [6], [7]. In case of nanoparticles, direct electrical measurement of these changes is not feasible, which is often the fastest and most direct method to identify the reactions occurring at the surface. Nanowires, on the other hand, are well suited for direct measurement of changes in their electrical properties (e.g. conductance/resistance, impedance). There has been a vast amount of research demonstrating beyond doubt the enhanced sensitivity, reactivity, and catalytic efficiency of the

nanoscale structures. Unfortunately, for the nanowires, the surface-adsorbate interactions are limited due to availability of only one type of binding sites. Despite being highly sensitive, nanowires too suffer from the lack of selectivity as their bulk counterpart devices.

Our approach utilizes *n*-type (Si doped) GaN nanowires functionalized with TiO<sub>2</sub> nanoclusters for selectively sensing the nitro-aromatic explosive compounds. The GaN is a wide-bandgap semiconductor (3.4 eV), with many unique properties. Its chemical inertness and capability of operating in extreme environments (high-temperatures, presence of radiation, extreme pH levels) are highly desirable for sensor design. TiO<sub>2</sub> is a photocatalytic semiconductor with bandgap energy of 3.2 eV for the anatase phase. Photocatalytic oxidation of various organic contaminants over titanium dioxide (TiO<sub>2</sub>) has been studied for decades. The TiO<sub>2</sub> nanoclusters were selected to act as nanocatalysts to increase the sensitivity, lower the detection time, and most importantly, to enable tailoring the selectivity of these structures to various analytes.

In recent years, researchers have demonstrated the potential of NWNC hybrids for sensing many different chemicals [8], [9]. However, most of the hybrid devices developed so far require elevated working temperatures, have long response/recovery times, and operate in inert atmospheres, which limit their use in environmental, domestic, and industrial applications. Engel *et al.* demonstrated selective detection of TNT in solution by silicon nanowire arrays [10]. Detection of traces of nitro-aromatics by using TiO<sub>2</sub>-B nanowires at ambient conditions is recently shown by Wang *et al.*, but the devices lack selectivity amongst the nitro-aromatics [11].

We recently demonstrated the sensitivity and selectivity of GaN nanowire/TiO<sub>2</sub> nanocluster hybrid sensors for sensing environmental pollutants like benzene, toluene, ethyl benzene, xylene, and chlorobenzene mixed in air [12]. On the other hand, gases like methanol, ethanol, isopropanol, chloroform, acetone, and 1,3-hexadiene did not show any change in the electrical characteristics of the devices, which clearly demonstrated the selective response of these sensors to the aromatic compounds. We were able to detect xylene, ethylbenzene, benzene, and toluene at a concentration level of 50 ppb. These sensor devices were highly stable and able to sense aromatic compounds reliably for concentrations as high as few percents in air. In the present study, for the first time, the sensitivity of the GaN nanowire/TiO<sub>2</sub> nanocluster hybrid device to the most common explosives, trinitrotoluene (TNT) and other nitro-aromatics is explored.

## II. EXPERIMENTAL DETAILS

The GaN nanowires were grown by molecular beam epitaxy method as described elsewhere [13]. The nanowires are aligned on a pre-patterned substrate using dielectrophoresis. Details of the device fabrication have been reported in our earlier paper [12]. After fabrication of two-terminal GaN NW devices, the anatase TiO<sub>2</sub> NCs were formed on the GaN NW surface using the following procedure. First, RF magnetron sputtering deposition of TiO<sub>2</sub> was done at 325 °C with 50 sccm of Ar flow, and 300 W RF power with a deposition rate of about

0.24 Å/s. Later, thermal annealing of the complete sensor devices (i.e., GaN NW coated with 8 nm of TiO<sub>2</sub>) was done at 700 °C for 30 s in a rapid thermal processing system. Due to this heat treatment the TiO<sub>2</sub> film crystallized in the form of anatase nanoclusters. After this step, the device substrates i.e., the *sensor chips* were wire-bonded on a 24-pin ceramic package for the gas sensing measurements.

The microstructure and morphology of the sputtered TiO<sub>2</sub> films used for the fabrication of the sensors were characterized by high-resolution transmission and scanning transmission electron microscopy (HRTEM/STEM), selected-area electron diffraction (SAED), and field-emission scanning electron microscopy (FESEM). For the TEM characterization, the GaN NWs were dispersed on 10 nm thick carbon films supported by molybdenum mesh grids, followed by the deposition and annealing of TiO<sub>2</sub> for forming anatase NCs and subsequent Pt deposition. The samples were analyzed in a FEI Titan 80–300 TEM/STEM microscope operating at 300 kV accelerating voltage and equipped with S-TWIN objective lenses, which provided 0.13 nm (STEM) and 0.19 nm (TEM) resolution by points. The instrument also had a Gatan CCD image acquisition camera, bright-field (BF), ADF and high-angle annular dark-field (HAADF) STEM detectors to perform spot, line profile, and areal compositional analyses using an EDAX 300 kV high-performance Si/Li X-ray energy dispersive spectrometer (XEDS) {Certain commercial equipment instruments or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology nor does it imply that the materials or equipment identified are necessarily the best available for the purpose}.

The as-fabricated sensors were placed in a custom designed gas chamber for gas exposure measurements. Detailed description of the experimental setup and experimental conditions can be found in an earlier paper [12]. The device characterization and the time dependent sensing measurements were done using an Agilent B1500A semiconductor parameter analyzer. The gas sensing experiments have been performed by measuring the electrical conductance of the devices upon exposure to controlled flow of air/chemical mixture in the presence of UV excitation (25 W deuterium bulb operating in the 215 nm to 400 nm range). For all the sensing experiments with chemicals, breathing air (<9 ppm of water) was used as the carrier gas. After the sensor devices were exposed to the aromatic compounds, they were allowed to regain their baseline current with the air/chemical mixture turned-off, without purging or evacuating the test-chamber.

## III. RESULTS AND DISCUSSION

### A. Morphological and Structural Characterization of NWNC Hybrids

TEM imaging was conducted under minimal beam intensity conditions close to the Scherzer defocus at the highest available accelerating voltage of 300 kV using both stationary beam (bright-field TEM/SAED, phase-contrast high-resolution TEM) and scanning beam (STEM/XEDS) modes. Areas for analyses were selected near the NW edge and on the

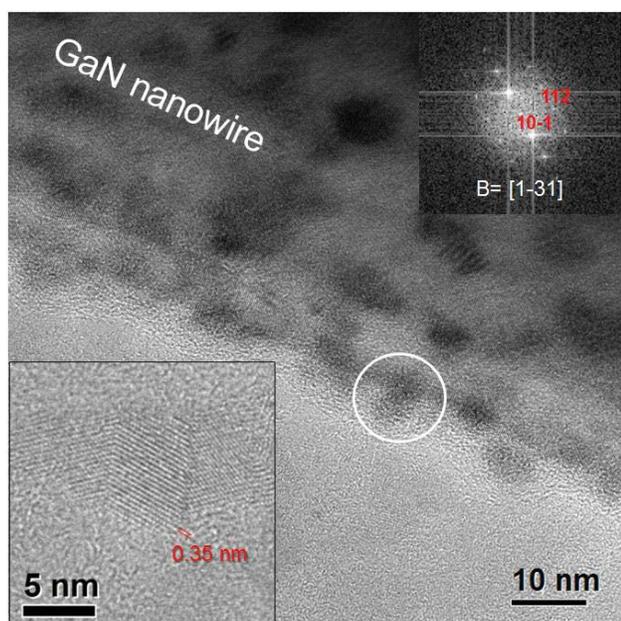


Fig. 1. HRTEM of GaN NW coated with TiO<sub>2</sub> nanoparticles. A typical titania nanoparticle at the NW edge is encircled. Insets show anatase lattice fringes (bottom) and corresponding FFT diffraction pattern (top).

amorphous carbon support film in the vicinity of the NW. Fig. 1 shows HRTEM micrographs of a TiO<sub>2</sub> coated GaN NW on a thin amorphous carbon support film. The deposited TiO<sub>2</sub> layer formed an array of islands, where 10 nm to 50 nm partially aggregated particles were often interconnected into extended 2-D networks. Detailed HRTEM and HR-STEM observations on these islands revealed 0.35 nm (101) hcp lattice fringes belonging to anatase phase (see lower inset in Fig. 1).

### B. Current–Voltage (*I*–*V*) Characteristics of NWNC Hybrids

Fig. 2 shows the *I*–*V* characteristics of a GaN NW two-terminal device at different stages of processing. The *I*–*V* curves of the as-deposited NW devices were rectifying (non-linear) and asymmetric (not clear in Fig. 2 due to scale resolution). However, the current increased with the deposition of TiO<sub>2</sub> nanoclusters. This can be attributed to decreased surface depletion of the GaN NW due to passivation of surface states, and/or the high temperature deposition (325 °C) of the titania nanoclusters initiating ohmic contact formation. The devices annealed at 700 °C for 30 s showed significant changes in their *I*–*V* characteristics with a majority of the devices exhibiting linear *I*–*V* curves. This is consistent with the fact that low resistance ohmic contacts to the nitrides require annealing at 700–800 °C.

### C. Sensing Behavior of GaN/TiO<sub>2</sub> NWNC Hybrid Sensors

The photocurrent through the bare GaN NW devices did not change when exposed to different chemicals mixed in air, even for concentrations as high as 3 %. In contrast, the TiO<sub>2</sub>-coated hybrid devices responded even to the pulses of 20 sccm airflow in the presence of UV excitation. The response of the TiO<sub>2</sub> NC-coated GaN nanowire hybrid sensors

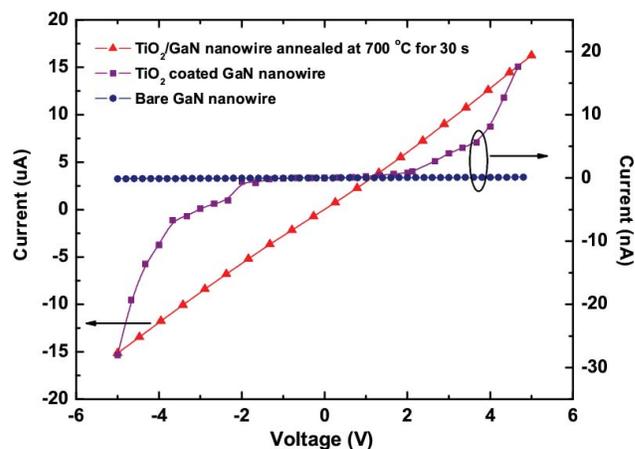


Fig. 2. *I*–*V* characteristics of a GaN NW two-terminal device at different stages of processing.

to different concentrations of benzene, toluene, ethylbenzene, chlorobenzene, and xylene in air was illustrated in our earlier paper<sup>12</sup> and will not be discussed here. The GaN/TiO<sub>2</sub> hybrids showed no response when exposed to other chemicals such as alcohols, ketones, amides, alkanes, nitro/halo-alkanes, and esters.

The response of the TiO<sub>2</sub> coated hybrid devices when exposed to a concentration of 100 ppb of the aromatics and nitro-aromatics in air can be seen in Fig. 3(a). Also, the UV excitation photocurrent for these sensors increased with respect to air when exposed to toluene vapors, whereas for every other aromatic compound the photocurrent decreased relative to air. The response is observed to increase with the increase in the number of nitro-groups attached to the aromatic compound (see Fig. 3(a)). The response of the hybrid device to different concentrations of trinitrotoluene (TNT) in air from 8 ppm down to as low as 500 ppt is shown in Fig. 3(b). Furthermore, the plots shown in Fig. 3 represent the sensing characteristics of the hybrid device after several uses, which clearly demonstrate their reusability and reliability. The response time is defined as the time taken by the change in sensor current to reach 90% of the response  $|I_f - I_0|$ , when exposed to the analyte. The  $I_f$  is the steady-state sensor current level in the presence of the analyte, and  $I_0$  is the current level without exposure to the analyte, which in our case is in the presence of air. The recovery time is the time required for the sensor current to recover to 30% of the response  $|I_f - I_0|$  after the gas flow is turned off. The response and recovery times of the nano-devices to different concentrations of TNT are ~ 30 s. The response and recovery times for the rest of the analytes varied from ~ 60 s to ~ 75 s.

We have defined the sensitivity as  $(R_{\text{gas}} - R_{\text{air}})/R_{\text{air}}$ , where  $R_{\text{gas}}$ ,  $R_{\text{air}}$  are the resistances of the sensor in the presence of air/chemical mixture and in the presence of the air, respectively. The sensitivity plot of a hybrid device for the different aromatics and nitro-aromatics tested is shown in Fig. 4. The sensitivity  $((R_{\text{gas}} - R_{\text{air}})/R_{\text{air}})$  for 1 ppm of TNT is ~ 10%. The devices exhibit a very highly sensitive and selective response to TNT when compared to interfering compounds like toluene. Each of the nitro-aromatic and

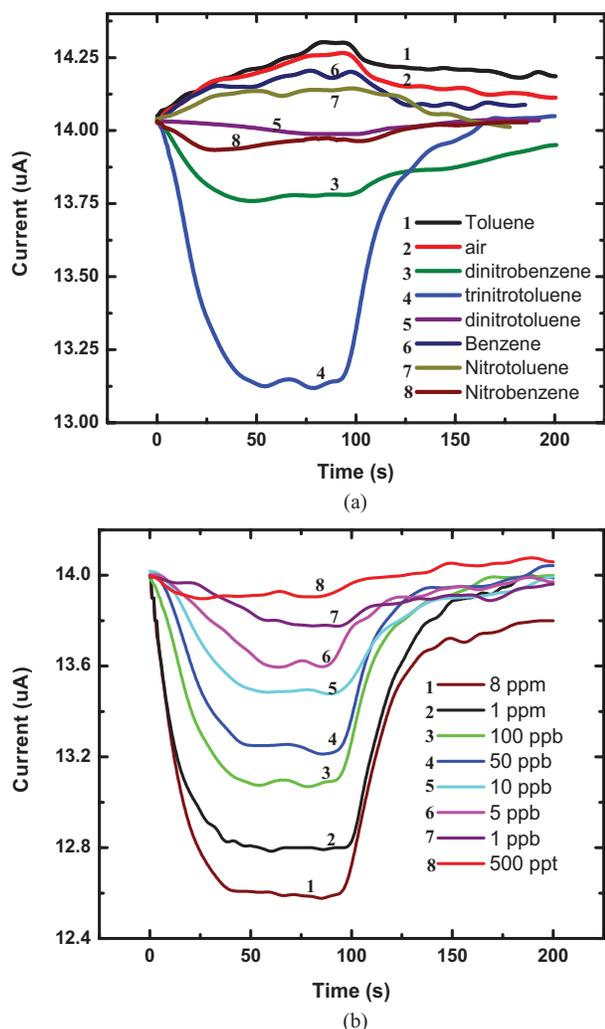


Fig. 3. (a) Response of a single nanowire-nanocluster hybrid sensor to 100 ppb of benzene, toluene, nitrobenzene, nitrotoluene, dinitrobenzene, dinitrotoluene, and trinitrotoluene (TNT) in the presence of UV excitation. (b) Response of the device to different concentrations of TNT under UV excitation.

aromatic compounds can be detected with high selectivity and reliability after several uses, indicating the ability to remove false-positive alarms. The zero level on the vertical axis of Fig. 4 represents the air exposure reference response. Toluene shows a decrease in response (an increase in current) with respect to the air (see also Fig. 3a), whereas; the TNT shows a remarkable increase in response (decrease in current) when compared to that of air. The plots of Fig. 4 demonstrate the sensor's ability to measure wide range of concentrations of the indicated chemicals. The sensitivity of two different devices processed in separate experimental runs, to the different aromatic compounds is shown in Fig. 5. It can be seen that the results are highly reproducible.

#### D. Sensing Mechanism

It is well-established that the oxygen vacancy defects ( $\text{Ti}^{3+}$  sites) on the surface of  $\text{TiO}_2$  are the "active sites" for the adsorption of species like oxygen, water, and organic molecules [14], [15]. In the presence of UV excitation with an energy above the bandgap energy of anatase  $\text{TiO}_2$  (3.2 eV)

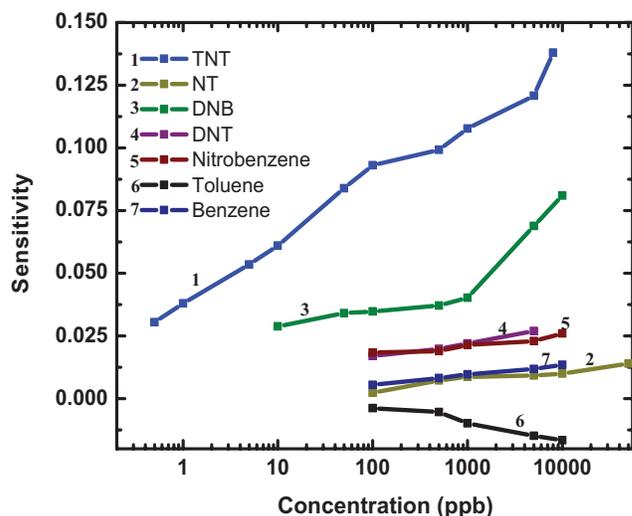


Fig. 4. Sensitivity plot of a GaN- $\text{TiO}_2$  nanowire-nanocluster hybrid device for benzene, toluene, nitrotoluene, nitrobenzene, dinitrotoluene (DNT), dinitrobenzene (DNB), and TNT.

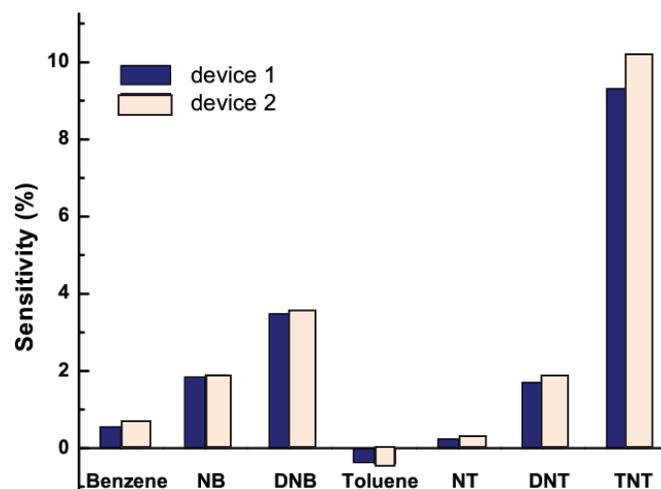


Fig. 5. Sensitivity of two different nanowire-nanocluster hybrid sensors to 100 ppb of the different aromatic compounds.

and GaN (3.4 eV), electron-hole pairs are generated both in the GaN NW and in the  $\text{TiO}_2$  cluster. Photogenerated holes in the nanowire tend to diffuse towards the surface due to the surface band bending. This effect of separation of photogenerated charge carriers results in a longer lifetime of photogenerated electrons, which in turn enhances the photoresponse of the nanowire devices in general. Since the nitro-aromatic compounds are highly electronegative, they tend to attract electrons from other molecules through charge transfer. This charge transfer between the adsorbed species on the  $\text{TiO}_2$  nanocluster and the nitro-groups in the nitro-aromatic compounds increases the width of the depletion region in the nanowire device, reducing the device current and consequently increasing the device resistance.

As seen from Table I, the electron affinity increases with the increase in nitro-groups attached to the aromatic compound i.e., the tendency of the compound to attract/accept an electron is much greater. As explained in our earlier paper [12], toluene showed a different behavior when compared with the rest

TABLE I  
ELECTRON AFFINITIES OF THE CHEMICALS

Chemicals	Electron Affinity (eV)
Benzene	-1.12
Toluene	-1.11
Nitrotoluene	0.94 ± 0.03
Nitrobenzene	1.0 ± 0.03
Dinitrotoluene	1.4
Dinitrobenzene	2.10
Trinitrotoluene	2.6-0.1

of the compounds in the class. In this work, it is observed that except for toluene and nitrotoluene, the sensitivity trend observed can be correlated well with their electron affinities.

#### IV. CONCLUSION

This study highlights the potential of nanowire-nanocluster hybrids for developing next-generation nano-sensors which have the capability of detecting explosive compounds quickly and reliably. The GaN/TiO<sub>2</sub> nanowire nanocluster hybrid devices detected trace amounts of aromatic and nitro-aromatic compounds in air at room temperature with very low response and recovery times (~ 30 s). The nitro-aromatic explosives like TNT can be selectively detected even for concentrations as low as 500 ppt. The deployment of such nano-enabled detection systems will help reducing the terrorist attacks and improving the homeland security.

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