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**6. AUTHOR(S)**
Bruce Kim

E-Mail: bkim@ccny.cuny.edu

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RESEARCH FOUNDATION OF THE
CITY UNIVERSITY OF NEW YORK
160 CONVENT AVE
NEW YORK NY 10031-9101

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SUITE 4229
RESEARCH TRIANGLE PARK NC 27709

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**14. ABSTRACT**
We have purchased the EasyTube 101 CVD system and Raman Spectrometer to synthesize and characterize Zinc Oxide nanowires which will be used in DoD related projects. Through the Apprenticeship Program, we worked wih high school students who worked with undergraduate and graduate students to perform synthesis and characterization of ZnO nanowire. We performed several optimizations using different substrates and temperatures for explosive vapor sensor development. The acquired equipment will be highly valuable for DoD research on CCNY campus.

**15. SUBJECT TERMS**
Zinc Oxide nanowires, CVD, Raman, synthesis, functionalization

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**Standard Form 298 (Rev. 8-98)**
**Prescribed by ANSI Std. Z39.18**

AWARD NUMBER: W911NF-16-1-0493

TITLE: Acquisition of CVD for Synthesis of Explosive Nanosensors

PRINCIPAL INVESTIGATOR: Bruce Kim

CONTRACTING ORGANIZATION:
RESEARCH FOUNDATION OF THE CITY UNIVERSITY OF NEW YORK
160 CONVENT AVE
NEW YORK NY 10031-9101

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US ARMY ACC-APG-RTP W911NF
800 PARK OFFICE DRIVE
SUITE 4229
RESEARCH TRIANGLE PARK NC 27709

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**Final Project Report**

**Acquisition of CVD for Synthesis of Explosive Nanosensors**

## 1. INTRODUCTION

The purpose of this equipment project is to acquire the following commercial instruments that would serve to proliferate nanosensor research at CCNY campus: (i) a customizable EasyTube 101® Chemical Vapor Deposition (CVD) system (First Nano, New York): This would be used for the growth of a spectrum of nanomaterials. It is a multi-functional automatic system with the capability to synthesize semiconducting nanowires, graphene, and carbon nanotubes (CNTs), to name a few, (ii) Horiba Nanolog® Fluorescence system- Since the proposed nanosensor research would utilize dual-modalities (optical and electrical), this system would help to quantify and analyze fluorescence characteristics of the nanosensor and would permit determination of optical response of the sensor in synergy with electrical measurements.

The acquired instruments provided a new thrust to nanotechnology research in alignment with DoD objectives at CCNY. In addition, these sophisticated systems helped inter-departmental collaboration that is otherwise limited due to absence of facilities for achieving mutual research objectives. Most importantly, we have engaged minority, under-represented and female students in research endeavors that are directly aligned with the needs of DoD. The Army apprenticeship program for high school and undergraduate students (HSAP/URAP) was awarded as a piggyback award to this project during the summer of 2018. The main focus of the HSAP/URAP was educating STEM students in nanotechnology. We worked with the High School for Math, Science and Engineering (HSMSE) adjacent to CCNY campus and STEM students at City College of New York (CCNY) through the STEM Career Development Institute (CDI) on the CCNY campus. They learned about the zinc oxide (ZnO) synthesis process and the functional chemistry needed to fabricate zinc oxide nanowires for developing nanosensor devices in explosive vapor sensing applications. We trained them with a nano-kit for hands-on training in fabricating and characterizing nanomaterials. Summary of purchased equipment is listed in Table 1.

Table 1. Summary of Equipment.

| Equipment Description | Unit Price |
|---|---|
| FirstNano EasyTube®101 CVD system | $163,044 |
| Horiba Nanolog® fluorescence system | $59,300 |
| **Total** | $222,344 |

## 2. BODY

Considerable research efforts have been dedicated to metal oxide nanosystems and CNTs (carbon nanotubes) due to their excellent physical, chemical, and optical properties. For example, ZnO is a wide band-gap (3.37 eV) semiconductor, which has been particularly interesting to the research community due to its large exciton binding energy (60 meV), which facilitates room temperature lasing action based on exciton recombination. Due to such excellent optical properties, ZnO nanostructures have versatile applications in short wavelength based optoelectronic devices, such as LEDs, laser diodes, nanocatalysis, OLED based displays, and opto-electronic chemical sensors in particular [1-8]. Similarly, CNTs, functionalized with protein receptors have been shown to approach molecular level detection of explosive molecules. Aforementioned applications require these nanostructures in different morphologies, such as nanowires, nanorods, and nanobelts, and must be subjected to post-synthesis procedures to achieve desired functionality. High aspect-ratio morphologies are desirable due to their large surface-to-volume ratio, which facilitates enhanced surface interactions. Therefore, it is rational to expect surface-related effects to be crucial for any conductometric or dual-modality devices based on these nanostructures. These surface-related effects, hence, mandate rigorous investigation as well. Functionalization of these nanostructures with additional organic or inorganic compounds should, therefore, allow for tailoring the surface for its non-linear signal thereby enabling the design of efficient nanosensors.

<u>Proposed Specific goals</u>

i. Synthesis and characterization of appropriate nanomaterials in different morphologies and topologies. Evaluation of pertinent candidates for nanosensor fabrication.

ii. Surface engineering of nanomaterial surfaces with appropriate fluorescent receptors to achieve optical detection. The chemical functionalization process would comprise identification and evaluation of various fluorescent receptors based on their functionalities.

iii. A dual-modality device will be fabricated and tested with the help of electrical characterization systems. Sensor operation under an opto-electronic stimulus will be validated for a particular nitroaromatic gas. Nitroaromatics are the main class of materials used in explosives and are high in the category of analytes of interest.

It is our belief that such systems would provide the much-needed capability to scope out dangerous areas of hazardous chemical activity without threatening the lives of soldiers and personnel on field. Hence, the requested instrumentation would not only serve to advance the research capabilities at CCNY but would provide a new direction to research as pertinent to DoD objectives. Furthermore, it would allow us to encourage, train, and mentor students from different backgrounds, and communities in areas that are important for national security so that they can become a vital asset to our defense laboratories engaged in cutting-edge research.

## Actual Work Performed with New CVD Equipment

### a) Synthesis analysis

High quality and consistent ZnO nanowire growth was achieved as a result of continuous optimization of process parameters, such as temperature, flow rate of carrier gas, placement of substrates with respect to precursor boat, and time of growth. These parameters are automated using the EasyTube 101 system, however, changing parameters are done by changing recipe programs on the CVD system. These parameters impacted the morphology and growth of ZnO nanowires to a varying degree. Hence, it was imperative that synthesis results were studied to determine a suitable recipe for nanowire growth. Although, almost all aforementioned factors were important, we focused specifically on temperature for ZnO nanowire growth using the automated CVD. It was vital to determine the optimum temperature for consistent ZnO nanowire growth, provided all other parameters have been kept constant at values. Therefore, four different temperatures were tested for ZnO nanowire synthesis and it was found that consistent nanowire growth occurred at 950°C, as can be seen in figure 1. Figure 1(a) shows the gold-coated Si substrate after 30 minutes of growth time at 900°C. Negligible growth is observed, which can be attributed to the insufficient thermodynamic activation of the growth nucleus. The carbothermal reaction, however, is spontaneous as significant amount of Zn and O can be seen deposited on the substrate in the energy-dispersive X-ray (EDAX) spectrum shown in inset of figure 1(a). Figure 1(b), (c) show some ZnO nanowire growth, however, the absence of consistent morphologies and localized growth indicated an unstable supersaturated growth nucleus that hindered nanowire growth. However, when the temperature was raised to 950°C a uniform ZnO nanowire growth was observed with a higher degree of vertical alignment to the substrate, as shown in figure 1(d). This is a characteristic that is required in device fabrication.

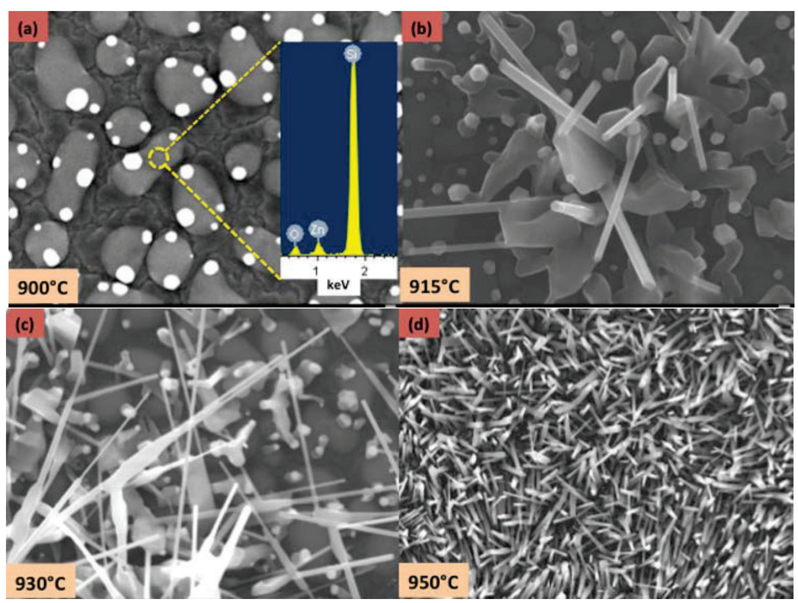


Figure 1: ZnO nanowire growth at (a) 900°C, (b) 915°C, (c) 930°C, and (d) 950°C. A uniform ZnO nanowire growth was observed at 950°C with a high degree of vertical alignment to the substrate as required for device fabrication.

b) **SEM, TEM, XRD, EDAX, and Photoluminescence analysis**

To effectively and consistently synthesize ZnO nanowires on templated-Si substrates, growth of ZnO nanowires was performed on ZnO (0001) and sapphire (11-20) substrates. The growth on these substrates was performed to validate the synthesis process developed for large band-gap substrates, such as insulators, to aid in amperometric device fabrication. The device design envisioned, for this research, was a conventional resistive sensor with an additional optical sensing mechanism to ensure high sensitivity and selectivity. Hence, a hierarchical and interconnected ZnO nanowire growth on insulating substrates was necessitated that possessed a vertically oriented, open-structure to facilitate analyte diffusion. In addition, it would promote carrier transport along ZnO nanowire backbone. The morphology of pristine ZnO nanostructures was determined through field emission-scanning electron microscopy (FE-SEM) on JEOL-7000, and the crystal structure was ascertained through high resolution-transmission electron microscopy (HR-TEM) on FEI-Tecnai. Furthermore, the chemical composition of as-synthesized ZnO nanowires was studied through energy dispersive X-ray spectroscopy (EDAX). For determination of orientational preference and complementary information on crystal structure, X-ray diffraction (XRD) of pristine ZnO nanowires was performed on Bruker AXS. Since low-defect ZnO nanowires, with minimal defect-related emission, were critical to nanosensor fabrication, the optical properties of pristine nanowires were characterized by photoluminescence spectroscopy.

Figure 2 shows the SEM images of pristine ZnO nanowires synthesized on Si (100), ZnO (0001), and sapphire (11-20) substrates. Figures 2(a) and 2(b) show the top-view of ZnO nanowires as-synthesized on Si (100) substrates. Nanowires with diameters ranging from 60-80 nm, and length up to 2-4 µm were observed. The random growth directions are attributed to the high degree of lattice mismatch and different crystal structure between Si and ZnO (~18%). However, as mentioned earlier, vertically oriented and interconnected structure is required for nanosensor device application. Therefore, ZnO nanowires were grown on ZnO (0001) substrates, as shown in figure 2(c), (d). It can be observed that ZnO nanowires grown on ZnO substrates have a very high degree of vertical orientation as well as an interconnected structure. While the vertical orientation can be attributed to the absence of lattice mismatch, the interconnected hierarchical structure can be posited to high concentration of Zn vapors in the chamber, and unstable, supersaturated catalyst droplets leading to horizontal growth, as shown in Figure 2 (c), (d). Although this is the desirable morphology, ZnO nanowires on ZnO substrates cannot be used for fabricating resistive devices due to their semiconducting nature. Therefore, sapphire (11-20) substrates were utilized due to their low lattice mismatch with ZnO that can result in almost epitaxial growth, and its insulating nature facilitating device fabrication. Figure 2(e), (f) illustrate ZnO nanowire morphology on sapphire (11-20) substrates. It can be observed that the nanowires are vertically oriented while maintaining a closely interconnected yet porous structure that is critical to device fabrication. In addition, the dimensions of nanowires fabricated on different substrates were found to be substrate independent, and therefore the morphology of ZnO nanowires on sapphire substrates were determined to be appropriate for nanosensor design.

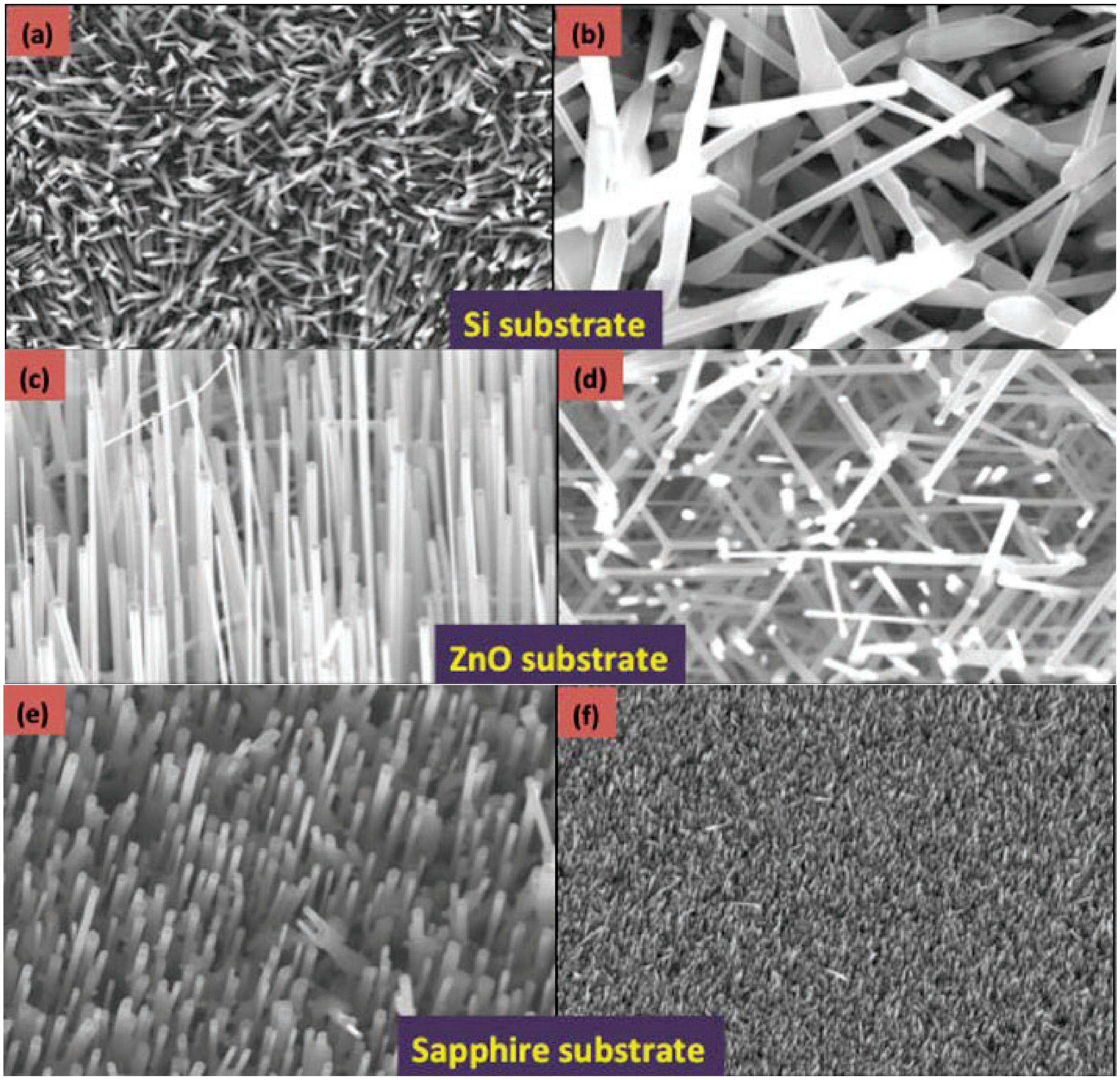


**Figure 2.** ZnO nanowires on **(a)**, **(b)** Si (100) substrate in top-view; **(c)**, **(d)** ZnO (0001) substrate in inclined and top-view respectively, and **(e)**, **(f)** sapphire (11-20) substrate in inclined view.

To ascertain the crystal structure of synthesized ZnO nanowires, TEM studies were conducted. Figure 3(a) shows the low-magnification TEM image of a single ZnO nanowire that is fully consistent with the SEM observations. The selected area electron diffraction pattern (SAED) in the inset of Figure 3(a) confirms a very high crystallinity (single-crystal nature) of the synthesized nanowires where a distinct dot pattern is observed. Furthermore, figure 3(b) shows the high-resolution TEM image of a selected area on ZnO nanowire, which conforms to the empirical observations of ZnO nanowires' tendency towards *c*-axis (<0001>) growth direction via a VLS process and typical interplanar spacing of 0.52 nm. The inset in Figure 3(b) shows a schematic of wurtzite-type crystal structure of ZnO nanowires that typically results from VLS growth process using a new CVD.

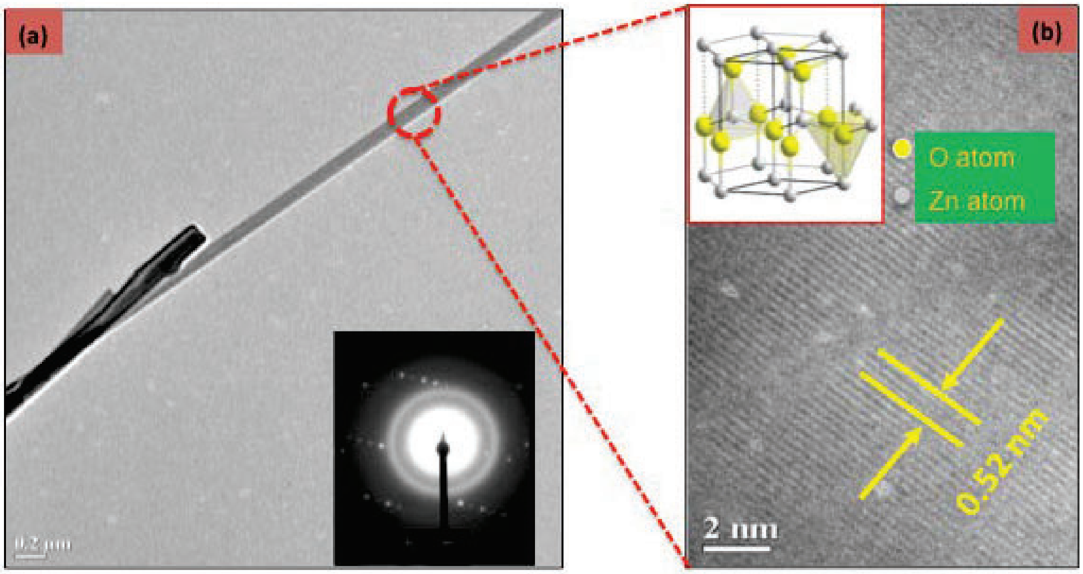


**Figure 3. (a)** Low-magnification TEM of a single ZnO nanowire; (inset) selected area electron diffraction (SAED) pattern of ZnO nanowire elucidating its single crystal nature, **(b)** HR-TEM image of a selected area on ZnO nanowire. The lattice spacing of 0.52 nm along [0001] direction confirms crystal structure of ZnO nanowire as wurtzite; (inset) schematic of wurtzite-type ZnO.

In order to ascertain the chemical composition of ZnO nanowires resulting from VLS process, EDAX studies were performed. As shown in Figure 4, ZnO nanowires as-synthesized on Si (100) substrates consist of peaks pertaining to elemental Zn and O in addition to substrate peak of Si. This confirmed that no contaminant phases were present in the pristine ZnO nanowires.

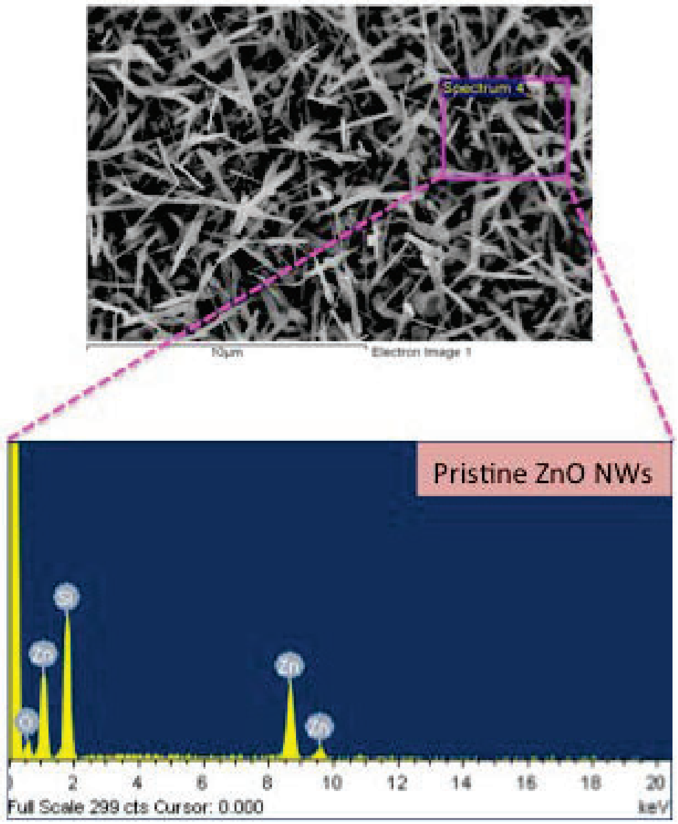


**Figure 4.** EDAX analysis of pristine ZnO nanowires (on Si substrate) as synthesized through the VLS process. The measurement area is $5\mu m^2$.

Furthermore, the atomic percentages of Zn and O were found to be in agreement with the stoichiometric ratio of the molecule, which indicated that ZnO was the only constituent present in the synthesized nanowires.

To determine optical characteristics and qualitatively estimate the defect concentration of synthesized ZnO nanowires, photoluminescence (PL) from the samples was measured. It was measured by suspending ZnO nanowires, scraped from the substrates, into pure acetone and drop casting the solution on to a quartz slide to be inserted in a conventional fluorimeter. It is to be noted that the measurements were taken by replacing the conventional quartz cuvette, which goes into the fluorimeter sample holder, by a quartz slide with nanowires. The quartz slide was repeatedly drop-casted with nanowire solution until a good reading was obtained by keeping the slide at 45 degrees to the incident monochromatic excitation light (325 nm). Keeping the slide at 45 degrees to the incident light prevented flooding of the detectors with reflected light and allowed for a good signal-to-noise.

## ZnO Nanowire Functionalization and Raman Characterization using Horiba

The functionalization of PBA compound on ZnO nanowires was carried out by two different approaches. The rationale behind for using two distinct coating methods was to ensure proper signal during surface characterization studies. First, conventional dip-coating method was utilized. PBA, purified through recrystallization in ethyl acetate was utilized for coating experiments. Silicon substrates with ZnO nanowires were dipped in 30 mM solution of PBA prepared in acetone, and were kept at 50°C under dark conditions. In an alternative approach for functionalization, ZnO nanowires were scraped from substrates into a concentrated solution of PBA (1 M). It is to be noted that for device fabrication, dip-coating method was used and the coating concentration of 30 mM PBA in acetone was optimized through repeated testing of fluorescence characteristics of PBA-coated ZnO nanowires.

For surface characterization, Raman spectroscopy will be performed on PBA-functionalized ZnO nanowire samples. The samples will be introduced in a Raman spectrometer (HR-Horiba). The measurements will be performed with 532 nm laser excitation source.

**Working with high school students on the Army Apprenticeship Program (HSAP/URAP)**

We had three high school students from New York City. Two male and one female students were involved doing research on nanotechnology. This opportunity was perfect due to additional award from the Army Apprenticeship Program during summer 2018. The award was modified to piggy back the award to this project. Students came every day from June 1, 2018 to August 30, 2018 to the lab and worked on the equipment. They learned characterization equipment from the new ASRC building where the nanofabricaton facility is located.

## 3. KEY RESEARCH ACCOMPLISHMENTS

- Synthesis of ZnO nanowires using Easytube 101 CVD system
- Nanowires were synthesized on Si, ZnO and Sapphire substrates
- Nanowires were synthesized in four different temperatures for optimization for explosive sensor devices.
- Raman Spectrometer by Horiba was bought, but it is not completed for lab set-up.
- Nanowires were functionalized with PBA compound for surface characterizations with Horiba.
- Three high school students were hired during summer 2018 for training on the CVD system. They learned about synthesis of nanowires and characterizations of nanowires using SEM and TEM. Raman spectroscopy was not done due to uncompleted set-up of Horiba and lack of time during summer time.

## 4. REPORTABLE OUTCOMES

We have published following papers based on the outcomes of the equipment.

1. B. Kim, S.B. Cho, "Zinc Oxide nanowire sensor packaging," IMAPS 2018, Pasadena, CA, October 2018.
2. B. Kim, S.B. Cho, "3D TSV Inductors for Secure IoT," IMAPS Device Conference, Scottsdale, AZ, March 2018.
3. B. Kim, A. Gupta, "Device Synthesis Topology for Zinc Oxide Nanowire Sensors," IEEE Nanotechnology Conference, Philadelphia, July 2017.

## 5. CONCLUSION

We have purchased the EasyTube 101 CVD system and Raman Spectrometer by Horiba to synthesize and characterize Zinc Oxide nanowires which will be used in DoD related projects. High school students worked with undergraduate and graduate students to perform synthesis and characterization of ZnO nanowire. We performed several optimizations using different substrates and temperatures for explosive vapor sensor development.

## 6. REFERENCES

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# REPORT OF INVENTIONS AND SUBCONTRACTS

*(Pursuant to "Patent Rights" Contract Clause) (See Instructions on back)*

*Form Approved OMB No. 9000-0095 Expires Jan 31, 2008*



**PLEASE DO NOT RETURN YOUR COMPLETED FORM TO THE ABOVE ORGANIZATION. RETURN COMPLETED FORM TO THE CONTRACTING OFFICER.**

| | |
|---|---|
| 1.a. NAME OF CONTRACTOR/SUBCONTRACTOR | The City College of New York |
| c. CONTRACT NUMBER | Same as 2(c) |
| b. ADDRESS *(Include ZIP Code)* | 160 Convent Avenue, New York, NY 10031 |
| d. AWARD DATE *(YYYYMMDD)* | 20160815 |
| 2.a. NAME OF GOVERNMENT PRIME CONTRACTOR | Same as 1(a) |
| c. CONTRACT NUMBER | W911NF-16-1-0493 |
| b. ADDRESS *(Include ZIP Code)* | Same as 1(b) |
| d. AWARD DATE *(YYYYMMDD)* | 20160815 |
| 3. TYPE OF REPORT *(X one)* | a. INTERIM [ ] b. FINAL [X] |
| 4. REPORTING PERIOD *(YYYYMMDD)* | a. FROM 20160815 b. TO 20180814 |

## SECTION I - SUBJECT INVENTIONS

5. "SUBJECT INVENTIONS" REQUIRED TO BE REPORTED BY CONTRACTOR/SUBCONTRACTOR *(If "None," so state)*

| NAME(S) OF INVENTOR(S) *(Last, First, Middle Initial)* a. | TITLE OF INVENTION(S) b. | DISCLOSURE NUMBER, PATENT APPLICATION SERIAL NUMBER OR PATENT NUMBER c. | ELECTION TO FILE PATENT APPLICATIONS *(X)* d. (1) UNITED STATES (a) YES | (1) UNITED STATES (b) NO | (2) FOREIGN (a) YES | (2) FOREIGN (b) NO | CONFIRMATORY INSTRUMENT OR ASSIGNMENT FORWARDED TO CONTRACTING OFFICER *(X)* e. (a) YES | e. (b) NO |
|---|---|---|---|---|---|---|---|---|
| NONE | NONE | NONE | | X | | | | |

| f. EMPLOYER OF INVENTOR(S) NOT EMPLOYED BY CONTRACTOR/SUBCONTRACTOR | |
|---|---|
| (1) (a) NAME OF INVENTOR *(Last, First, Middle Initial)* | (2) (a) NAME OF INVENTOR *(Last, First, Middle Initial)* |
| (b) NAME OF EMPLOYER | (b) NAME OF EMPLOYER |
| (c) ADDRESS OF EMPLOYER *(Include ZIP Code)* | (c) ADDRESS OF EMPLOYER *(Include ZIP Code)* |

| g. ELECTED FOREIGN COUNTRIES IN WHICH A PATENT APPLICATION WILL BE FILED | |
|---|---|
| (1) TITLE OF INVENTION | (2) FOREIGN COUNTRIES OF PATENT APPLICATION |

## SECTION II - SUBCONTRACTS *(Containing a "Patent Rights" clause)*

6. SUBCONTRACTS AWARDED BY CONTRACTOR/SUBCONTRACTOR *(If "None," so state)*

| NAME OF SUBCONTRACTOR(S) a. | ADDRESS *(Include ZIP Code)* b. | SUBCONTRACT NUMBER(S) c. | FAR "PATENT RIGHTS" d. (1) CLAUSE NUMBER | (2) DATE *(YYYYMM)* | DESCRIPTION OF WORK TO BE PERFORMED UNDER SUBCONTRACT(S) e. | SUBCONTRACT DATES *(YYYYMMDD)* f. (1) AWARD | (2) ESTIMATED COMPLETION |
|---|---|---|---|---|---|---|---|
| None | | | | | | | |

## SECTION III - CERTIFICATION

7. CERTIFICATION OF REPORT BY CONTRACTOR/SUBCONTRACTOR *(Not required if: (X as appropriate))* [ ] SMALL BUSINESS or [X] NONPROFIT ORGANIZATION

**I certify that the reporting party has procedures for prompt identification and timely disclosure of "Subject Inventions," that such procedures have been followed and that all "Subject Inventions" have been reported.**

| a. NAME OF AUTHORIZED CONTRACTOR/SUBCONTRACTOR OFFICIAL *(Last, First, Middle Initial)* | b. TITLE | c. SIGNATURE | d. DATE SIGNED |
|---|---|---|---|
| Douglas Adams | Director, Technology Commercialization Office CUNY | Doug Adams | 20190207 |

8. PROHIBITION ON USING FUNDS UNDER GRANTS AND COOPERATIVE AGREEMENTS WITH ENTITIES THAT REQUIRE CERTAIN INTERNAL CONFIDENTIALITY AGREEMENTS.

A. The recipient may not require its employees, contractors, or subrecipients seeking to report fraud, waste, or abuse to sign or comply with internal confidentiality agreements or statements prohibiting or otherwise restricting them from lawfully reporting that waste, fraud, or abuse to a designated investigative or law enforcement representative of a Federal department or agency authorized to receive such information.

B. The recipient must notify its employees, contractors, or subrecipients that the prohibitions and restrictions of any internal confidentiality agreements inconsistent with paragraph (a) of this award provision are no longer in effect.

C. The prohibition in paragraph (a) of this award provision does not contravene requirements applicable to Standard Form 312, Form 4414, or any other form issued by a Federal department or agency governing the nondisclosure of classified information.

D. If the Government determines that the recipient is not in compliance with this award provision, it:

1. Will prohibit the recipient's use of funds under this award, in accordance with section 743 of Division E of the Consolidated and Further Continuing Resolution Appropriations Act, 2015, (Pub. L. 113-235) or any successor provision of law; and

2. May pursue other remedies available for the recipient's material failure to comply with award terms and conditions.

EXHIBIT A – Budget

EXHIBIT B – Reporting Requirements for Instrumentation Grants – A final report shall be submitted within 90 days following the end of the specified performance period, or any authorized extension thereto, listing all items of equipment actually acquired by name, manufacturer where possible, cost, and a description of any special circumstances regarding the acquisition of the equipment. The report will also include a concise summary of the research projects on which equipment has been or will be used, including support of (a) the research work described in the proposal and (b) other research work of interest to DoD.