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6. AUTHORS T. M. Crawford, G. Koley, X. Li, T. Vogt, and R. A. Webb			5d. PROJECT NUMBER		
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14. ABSTRACT This project has supported research to develop multimodal microcantilever gas sensing, to investigate the mechanical properties of Zn and Sn oxide nanobelts for mechanical detection, nanoscale assembly of magnetic nanomaterials into user defined shapes, noise properties of spintronic multilayer systems, and finally, conductivity changes in self-assembled layers of DNA for sensing applications. The project has led to 3 issued U.S. patents, 3 provisional patent applications, 20 publications, 25 invited talks, as well as numerous conference talks and					
15. SUBJECT TERMS Self Assembly, Nanopatterning, Nanomanufacturing, Microcantilever Sensor, multimodal detection, Atomic Force Microscopy, SnO2 nanowire sensor, nanoindentation, nanobelts					
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Report Title

Final Report: Sensing biological, chemical, and radiation hazards in harsh environment

ABSTRACT

This project has supported research to develop multimodal microcantilever gas sensing, to investigate the mechanical properties of Zn and Sn oxide nanobelts for mechanical detection, nanoscale assembly of magnetic nanomaterials into user defined shapes, noise properties of spintronic multilayer systems, and finally, conductivity changes in self-assembled layers of DNA for sensing applications. The project has led to 3 issued U.S. patents, 3 provisional patent applications, 20 publications, 25 invited talks, as well as numerous conference talks and proceedings. 3 Postdoctoral fellows, 6 PhD students, 3 temporary researchers, and 4 undergraduates (3 of whom were women science majors) were supported by this project.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

ReceivedPaper

- 05/07/2013 1.00 L. Ye, B. Terry, O. T. Mefford, C. Rinaldi, T. M. Crawford. All-nanoparticle concave diffraction grating fabricated by self-assembly onto magnetically-recorded templates, *Optics Express*, (01 2013): 0. doi: 10.1364/OE.21.001066
- 05/07/2013 2.00 Y. Chen, B. Zhong, S. Garzon, T. M. Crawford, R. A. Webb. Low frequency noise characteristics of submicron magnetic tunnel junctions, *Journal of Applied Physics*, (2011): 0. doi: 10.1063/1.3562039
- 05/07/2013 3.00 Samir Garzon, Richard A. Webb, Mark Covington, Shehzaad Kaka, Thomas M. Crawford. Effect of resistance feedback on spin torque-induced switching of nanomagnets, *Journal of Magnetism and Magnetic Materials*, (10 2009): 0. doi: 10.1016/j.jmmm.2009.05.044
- 05/07/2013 4.00 Samir Garzon, Yaroslav Bazaliy, Richard Webb, Mark Covington, Shehzaad Kaka, Thomas Crawford. Macrospin model to explain the absence of preswitching oscillations in magnetic tunnel junctions: Fieldlike spin-transfer torque, *Physical Review B*, (03 2009): 0. doi: 10.1103/PhysRevB.79.100402
- 05/07/2013 5.00 J. R. Henderson, T. M. Crawford. Repeatability of magnetic-field driven self-assembly of magnetic nanoparticles, *Journal of Applied Physics*, (2011): 0. doi: 10.1063/1.3556770
- 05/07/2013 7.00 Ehtesham B. Quddus, Alina Wilson, Jie Liu, Zhihua Cai, Deepak Veerreddy, Xinyong Tao, Xiaodong Li, Richard A. Webb, Goutam Koley. Structural and elastic properties of InN nanowires, *physica status solidi (a)*, (04 2012): 0. doi: 10.1002/pssa.201127530
- 05/07/2013 8.00 Xiaodong Li, Ioannis Chasiotis, Takayuki Kitamura. In Situ Scanning Probe Microscopy Nanomechanical Testing, *MRS Bulletin*, (01 2011): 0. doi: 10.1557/mrs2010.568
- 05/07/2013 9.00 Lihong Bao, Zhi-Hui Xu, Rui Li, Xiaodong Li. Catalyst-Free Synthesis and Structural and Mechanical Characterization of Single Crystalline Ca, *Nano Letters*, (01 2010): 0. doi: 10.1021/nl9034187
- 05/07/2013 10.00 Md. W. K. Nomani, V. Shields, G. Tompa, N. Sbrockey, M. G. Spencer, R. A. Webb, G. Koley. Correlated conductivity and work function changes in epitaxial graphene, *Applied Physics Letters*, (2012): 0. doi: 10.1063/1.3691628
- 05/07/2013 11.00 Md. W.K. Nomani, D. Kersey, J. James, D. Diwan, T. Vogt, Richard A. Webb, G. Koley. Highly sensitive and multidimensional detection of NO₂ using In₂O₃ thin films, *Sensors and Actuators B: Chemical*, (12 2011): 0. doi: 10.1016/j.snb.2011.07.044
- 05/07/2013 12.00 Goutam Koley, Zhihua Cai, Ehtesham Bin Quddus, Jie Liu, Muhammad Qazi, Richard A Webb. Growth direction modulation and diameter-dependent mobility in InN nanowires, *Nanotechnology*, (07 2011): 0. doi: 10.1088/0957-4484/22/29/295701
- 05/07/2013 13.00 Muhammad Qazi, Nicholas DeRoller, Abdul Talukdar, Goutam Koley. III-V Nitride based piezoresistive microcantilever for sensing applications, *Applied Physics Letters*, (2011): 0. doi: 10.1063/1.3657467
- 05/07/2013 14.00 Md.W.K. Nomani, Razib Shishir, Muhammad Qazi, Devendra Diwan, V.B. Shields, M.G. Spencer, Gary S. Tompa, Nick M. Sbrockey, Goutam Koley. Highly sensitive and selective detection of NO₂ using epitaxial graphene on 6H-SiC, *Sensors and Actuators B: Chemical*, (9 2010): 0. doi: 10.1016/j.snb.2010.06.069
- 05/07/2013 15.00 Muhammad Qazi, Mohammad W. K. Nomani, M. V. S. Chandrashekar, Virgil B. Shields, Michael G. Spencer, Goutam Koley. Molecular Adsorption Behavior of Epitaxial Graphene Grown on 6H-SiC Faces, *Applied Physics Express*, (7 2010): 0. doi: 10.1143/APEX.3.075101

05/07/2013 16.00 Muhammad Qazi, Jie Liu, M. V. S. Chandrashekhar, Goutam Koley. Surface electronic property of SiC correlated with NO₂ adsorption, Journal of Applied Physics, (2009): 0. doi: 10.1063/1.3251404

05/07/2013 17.00 Jie Liu, Zhihua Cai, Goutam Koley. Charge transport and trapping in InN nanowires investigated by scanning probe microscopy, Journal of Applied Physics, (2009): 0. doi: 10.1063/1.3273380

TOTAL: 16

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. B. Terry, J. Henderson, A. Netz, and T. M. Crawford, Nanoparticle diffraction grating fabricated with pattern transfer nanomanufacturing. Magnetism and Magnetic Materials Conference, Scottsdale, AZ, November 2011. (Contributed Oral)
2. L. Ye, S. Garzon, M. Covington, R. A. Webb, and T. M. Crawford, Asymmetric delay dependence in ultrafast nanomagnet dynamics excited by oppositely polarized picosecond spin torque impulses. Magnetism and Magnetic Materials Conference, Scottsdale, AZ, November 2011. (Contributed Oral)
3. J. Henderson and T. M. Crawford, Repeatability of magnetic-field driven self-assembly of magnetic nanoparticles. Magnetism and Magnetic Materials Conference, Atlanta, GA, November 2010. (Contributed Oral)
4. S. Shi, J. Henderson, and T. M. Crawford, Programmable magnetic nanoparticle assembly and pattern transfer for nanomanufacturing. Joint Magnetism and Magnetic Materials/International Magnetism Conference. Washington, DC. January 2010 (Contributed Oral).
5. T. R. Ray, J. S. Snipes C. J. Murphy, T. M. Crawford, and S. C. Baxter, Force handles for alignment of metallic nanoparticles. Materials Research Society Fall Meeting, Boston, MA. November 2009. (Contributed Poster)
6. S. Garzon, Y. Bazaliy, R. A. Webb, M. Covington, S. Kaka, and T. M. Crawford. Macrospin model of incubation delay due to field-like spin transfer torque. APS March Meeting., Pittsburgh, PA, March 2009. (Contributed Oral)
7. L. Ye, S. Garzon, R. A. Webb, M. Covington, S. Kaka, and T. M. Crawford. Effective damping measurements with ultrafast spin torque pulses. Conference on Magnetism and Magnetic Materials, Austin, TX, November, 2008. (Contributed Oral).
8. B. Knaus, S. Garzon, and T. M. Crawford. Alkanethiol capping induced changes in the magnetoresistance of Au/Co bilayers. Conference on Magnetism and Magnetic Materials, Austin, TX, November 2008. (Contributed Poster)
9. L. Ye, S. Garzon, R. A. Webb, M. Covington, S. Kaka, and T. M. Crawford. Coherent control of spin momentum transfer-driven switching in magnetic nanopillars APS March Meeting, New Orleans, LA March, 2008. (Contributed Oral)
10. B. Knaus, S. Garzon, and T. M. Crawford. Alkanethiol capping-induced changes in the magnetoresistance of Au/Co bilayers. APS March Meeting. New Orleans, LA. March 2008. (Contributed Oral)
11. Xiaodong Li, "Flexible Nanostructured Composite Electrodes for High Performance Supercapacitors," TMS 2013, 142nd Annual Meeting & Exhibition, San Antonio, TX, March 3-7, 2013 (invited talk).
12. Xiaodong Li, "Flexible Nanomaterials-Enabled High-Performance Supercapacitor Electrodes," MS&T'2012, Material Science & Technology 2012 Conference & Exhibition, Pittsburgh, PA, October 7-11, 2012 (invited talk).
13. Xiaodong Li, "Boron and Boride Nanowires and Their Composites," MS&T'2012, Material Science & Technology 2012 Conference & Exhibition, Pittsburgh, PA, October 7-11, 2012 (invited talk).
14. Xiaodong Li, "Surface and Environmental Effects on the Mechanical Behavior and Function of Nanostructures," MS&T'2012, Material Science & Technology 2012 Conference & Exhibition, Pittsburgh, PA, October 7-11, 2012 (invited talk).
15. Xiaodong Li, "Nature-inspired Composite Design and Manufacturing," Innovations in Biomedical Materials 2012, Raleigh, NC, September 10-13, 2012 (invited talk).
16. Xiaodong Li, "Environmental Effects on the Mechanical Behavior and Function Performance of Nanostructures," Plasticity 2012, San Juan, Puerto Rico, January 3-8, 2012 (invited talk).
17. Xiaodong Li, "Environmental Effects on the Mechanical Behavior and Function Performance of Nanostructures," TMS 2011, 140th Annual Meeting & Exhibition, San Diego, California, February 27 - March 3, 2011 (invited talk).
18. Xiaodong Li, Yingchao Yang, Jianfeng Zang, Zhi-Hui Xu, and Richard A. Webb, "Environmental Effects on the Mechanical Behavior and Function Performance of Nanostructures," 2011 ASME Annual Conference, International Mechanical Engineering Congress & Exposition (IMECE), Denver, Colorado, November 11- 17, 2011.
19. Xiaodong Li, "In situ Atomic Force Microscopy Nanomechanical Testing and Nanofabrication," 2010 MRS Fall Meeting, Boston, MA, November 29 - December 3, 2010 (invited talk).
20. Zhi-Hui Xu, Xiaodong Li, Michael A. Sutton, and Ning Li, "Drift and Spatial Distortion Elimination in Atomic Force Microscopy Images by the Digital Image Correlation Technique," 2009 SEM Fall Symposium and Workshop - Advanced Image-Based Measurement Methods: Recent Developments and Applications in Engineering and Medicine, Columbia, SC, October 5-7, 2009 (invited talk).
21. Zhi-Hui Xu, Xiaodong Li, Michael A. Sutton, and Ning Li, "Drift and Spatial Distortion Elimination in Atomic Force Microscopy Images by the Digital Image Correlation Technique," 2009 MRS Fall Meeting, Boston, MA, November 30 - December 4, 2009.

22. Abdul Talukdar, and Goutam Koley, "Highly Sensitive III-V Nitride Based Piezoresistive Microcantilever using Embedded AlGa_N/Ga_N HFET", poster presentation at the Hilton Head Workshop, Hilton Head, SC, June, 2012. (100%)
23. Md. W. K. Nomani, A. Singh, V. Shields, M. Spencer, G. Tompa, N. Sbrockey, and G. Koley, "Work Function and Conductivity changes due to Molecular adsorption in Epitaxial Graphene on 6H-SiC," invited oral presentation at the IEEE Nano Conference, Portland, Oregon, August, 2011. (80%)
24. Muhammad Qazi, Md. Nomani, and Goutam Koley "Piezoresistive Microcantilever with Embedded AlGa_N/Ga_N HFET for Sensing Applications," oral presentation at the EMC 2011 conference in Santa Barbara, June 22, 2011. (100%)
25. Mohammad Waliullah Nomani, Amol Singh, Virgil Shields, Mike Spencer, Gary Tompa, Nick Sbrockey, and Goutam Koley, "Correlated Conductivity and Work Function in Epitaxial Graphene," oral presentation at the EMC 2011 conference in Santa Barbara, June 23, 2011. (80%)
26. Muhammad Qazi, Md. W. K. Nomani, MVS Chandrashekhar, Goutam Koley, V. B. Shields, and M. G. Spencer, "Molecular adsorption behavior of epitaxial graphene grown on 6H-SiC faces," oral presentation at the Spring MRS conference in San Francisco, April, 2011. (85%)
27. Muhammad Qazi, Nicholas DeRoller, Jie Liu, and Goutam Koley, "AlGa_N/Ga_N based microcantilever for potentiometric molecular sensing," oral presentation at the Spring MRS conference in San Francisco, April, 2011. (100%)
28. Nicholas Deroller, Jie Liu, Muhammad Qazi, and Goutam Koley," Characterization of an AlGa_N/Ga_N Electrostatically Actuated Cantilever Using the Finite Element Method" oral presentation at the COMSOL Boston conference, October, 2010.
29. Goutam Koley, Muhammad Qazi, MVS Chandrashekhar, Md. W. Nomani, and Virgil Shields, and M. G. Spencer," NO₂ sensitivity of SiC and epitaxial graphene on SiC," oral presentation at the Spring MRS conference, San Francisco, April 6, 2010.
30. Goutam Koley, Zhihua Cai, and Ehtesham Bin Quddus, "Structural and Electrical Properties of High Quality Narrow Diameter InN Nanowires," oral presentation at the Spring MRS conference, San Francisco, April 6, 2010.
31. Muhammad Qazi, Md. W. Nomani, MVS Chandrashekhar, and Goutam Koley, "NO₂ Sensitivity of Wide Area SiC and Epitaxial Graphene on SiC Substrates," oral presentation at the ISDRS Conference, Maryland, Dec 9, 2009. 100%)
32. Jie Liu, Zhihua Cai, and Goutam Koley, "Charge transport in InN nanowires investigated by scanning probe microscopy," oral presentation at the ISDRS Conference, Maryland, Dec 11, 2009. (100%)

Number of Presentations: 32.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u>	<u>Paper</u>
05/07/2013 18.00	A Talukdar, G Koley. HIGHLY SENSITIVE III-V NITRIDE BASED PIEZORESISTIVE MICROCANTILEVER USING EMBEDDED ALGAN/GAN HFET, Hilton Head Solid State Sensors, Actuators, and Microsystems Workshop. 2012/06/03 00:00:00, . . . ,
05/07/2013 19.00	M W K Nomani, M G Spencer, G. Koley. Highly Sensitive and Selective NO ₂ Sensing using Epitaxial Graphene on 6H-SiC Goutam, IEEE Sensors Conference. 2010/11/01 00:00:00, . . . ,
05/07/2013 20.00	M Nomani, A Singh, V Shields, M Spencer, G Tompa, N Sbrockey, G Koley. Investigation of work function and conductivity changes due to molecular doping in epitaxial Graphene grown on both the faces of 6H-SiC, IEEE Nano Conference. 2011/08/15 00:00:00, . . . ,
05/07/2013 21.00	M Nomani, M Qazi, MVS Chandrasekhar, G Koley. NO ₂ sensitivity of wide area SiC and epitaxial graphene on SiC substrates, ISDRS. 2009/12/09 00:00:00, . . . ,
TOTAL:	4

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

1. "Energy Storage from Cotton Textiles," Xiaodong Li, Provisional US Patent Application 4/19/2012
 2. J. Henderson and T. M. Crawford, Methodology for nanoparticle coating on a magnetically recorded surface (Provisional Patent Application filed).
 3. T. M. Crawford and D. J. Tedeschi Radiation Sensor/Dosimeter employing synthetic DNA and electromagnetic readout – (Provisional Patent Application filed).
-

Patents Awarded

1. "Multi-dimensional Integrated Detection and Analysis System" Goutam Koley and T. Vogt, US Patent # 8,236,569
issued on 8/7/2012

2. Method of Sensing Chemical and Bio-molecular Analytes and Sensing System Using a Microcantilever" Goutam Koley, M. Qazi, and T. Vogt
US Patent# 8,252,598 issued on 8/28/2012

3. T. M. Crawford and S. Garzon, Nanoscale Spintronic Chemical Sensor. University of South Carolina. U.S. # 8,372,344 (2013).

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Jie Liu	0.65	
Yuqing Mao	0.13	
MD Quddus	1.00	
Amol Singh	0.25	
Longfei Ye	0.50	
Yihao Zhu	0.13	
FTE Equivalent:	2.66	
Total Number:	6	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Lihong Bao	0.75
Irina Roof	0.25
Razib Shishir	0.25
FTE Equivalent:	1.25
Total Number:	3

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Thomas M. Crawford	0.08	No
Goutam Koley	0.08	No
Xiaodong Li	0.08	No
FTE Equivalent:	0.24	
Total Number:	3	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Olivia Keyes	0.25	Physics
Bradley Terry	0.37	Chemistry and Biochemistry
Amy Hill	0.06	Chemistry and Biochemistry
Kelsey Hincke	0.06	Physics
FTE Equivalent:	0.74	
Total Number:	4	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:	2.00
The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....	2.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....	2.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):.....	2.00
Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:.....	0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense	0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:	0.00

Names of Personnel receiving masters degrees

<u>NAME</u>
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>	
Muhammad Nomani	
Muhammad Qazi	
Jie Liu	
Total Number:	3

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Kareem Alasady	0.06
Zihua Cai	0.06
Jianfeng Zang	0.50
FTE Equivalent:	0.62
Total Number:	3

Sub Contractors (DD882)

1 a. Marshall University Research Corporation

1 b. Marshall University Research Corporatic

401 11th Street

Huntington WV 25701

Sub Contractor Numbers (c):

Patent Clause Number (d-1):

Patent Date (d-2):

Work Description (e):

Sub Contract Award Date (f-1):

Sub Contract Est Completion Date(f-2):

1 a. Marshall University Research Corporation

1 b. 1050 4th Avenue

Huntington WV 25755

Sub Contractor Numbers (c):

Patent Clause Number (d-1):

Patent Date (d-2):

Work Description (e):

Sub Contract Award Date (f-1):

Sub Contract Est Completion Date(f-2):

1 a. North Carolina State University

1 b. Research Administration

2701 Sullivan Drive, Suite 240

Raleigh NC 276957514

Sub Contractor Numbers (c):

Patent Clause Number (d-1):

Patent Date (d-2):

Work Description (e):

Sub Contract Award Date (f-1):

Sub Contract Est Completion Date(f-2):

1 a. North Carolina State University

1 b. Research Administration & Sponsored P

Box 7514

Raleigh NC 276957214

Sub Contractor Numbers (c):

Patent Clause Number (d-1):

Patent Date (d-2):

Work Description (e):

Sub Contract Award Date (f-1):

Sub Contract Est Completion Date(f-2):

Inventions (DD882)

5 Energy Storage From Cotton Textiles

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: Xiaodong Li

5f-1a:

5f-c:

5 Method of Sensing Chemical and Bio-molecular Analytes and Sensing System Using a Microcantilever

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: Goutam Koley

5f-1a:

5f-c:

5a: Tom Vogt

5f-1a:

5f-c:

5 Methodology for nanoparticle coating on a magnetically-recorded surface

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: J. Henderson

5f-1a:

5f-c:

5a: T. M. Crawford

5f-1a:

5f-c:

5 Multi-dimensional Integrated Detection and Analysis System

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: Goutam Koley

5f-1a:

5f-c:

5a: Mohammed Qazi

5f-1a:

5f-c:

5a: Tom Vogt

5f-1a:

5f-c:

5 Nanoscale Spintronic Chemical Sensor

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: S. Garzon

5f-1a:

5f-c:

5a: T. M. Crawford

5f-1a:

5f-c:

5 Radiation Sensor/Dosimeter Employing Synthetic DNA and Electromagnetic Readout

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: T. M. Crawford

5f-1a:

5f-c:

5a: D. J. Tedeschi

5f-1a:

5f-c:

Scientific Progress

Crawford

This project partially supported efforts to employ magnetic recording media as nanotemplates for assembling magnetic nanoparticles into designed structures. We have successfully built prototype 3D composites from magnetic nanoparticles with interleaved structures, with strong potential for yielding THz metamaterials. In addition, we have built optical diffraction gratings on polymer substrates entirely out of magnetic nanoparticles and demonstrated optical spectroscopy with the gratings. These gratings could be capable of both mechanical and chemical detection of optical spectrum changes in analytes adsorbed on the nanoparticle surfaces. Thus these materials could support multi-functional sensing, e.g. responding to simultaneous mechanical and chemical stimuli. This technique could be a game-changer for THz sensing because it is very inexpensive to build complex structures from nanomaterials over centimeter² areas

This project supported work studying the dynamical properties and noise of nanomagnet devices whose magnetization was subjected to a spin transfer torque by an electric current. First, we demonstrated that the macrospin model can explain the absence of preswitching oscillations in magnetic tunnel junctions via a field-like spin transfer torque. We also showed that for magnetic tunnel junctions undergoing large changes in resistance as the magnetization changes, that the dynamically changing voltage can impact the device dynamics, as can capacitive shunting. We further studied low frequency noise in sub-micron magnetic tunnel junctions, observing Lorentzian-like noise spectra with significant deviations from $1/f$ behavior. These magnetic results are critical for employing nanomagnets for sensing changes in chemisorbed synthetic DNA as part of a nanoscale spintronic chemical sensor. The concept for this sensor led to an issued patent as part of this project.

Finally, this project also supported efforts to support ongoing ARO projects through collaborations with Tom Pearl at NC State and Mike Norton at Marshall. Crawford acquired a fluorescence microscopy capability and photoelastic modulator which were used to study DNA systems on surfaces in support of Pearl and Norton. Similarly, resistance measurements on gold films with self-assembled DNA structures were made in support of this project.

Li

(1) We developed several in situ atomic force microscopy (AFM) nanomechanical testing methods spanning (1) probing the mechanical properties of individual one dimensional (1D) nanostructures, (2) mapping local, nanoscale strain fields, fracture and wear damage of nanostructured heterogeneous materials, and (3) measuring the interfacial strength of nanostructures. The novel AFM nanomechanical testing methods are expected to lead to further advancements in nanoscale mechanical testing and instrumentation towards the exploration and the fundamental understanding of mechanical property size effects in nanomaterials.

(2) We successfully synthesized single crystalline $\text{Ca}_2\text{B}_2\text{O}_5 \cdot \text{H}_2\text{O}$ (CBOH) nanobelts for the first time using a facile catalyst-free hydrothermal method at low temperature. After being annealed at 400 °C for 10 h, the CBOH nanobelts transformed into curve shaped $\text{Ca}_2\text{B}_2\text{O}_5$ (CBO) nanogrooves with ordered stacking fault arrays along the c direction. The single crystalline CBOH nanobelts and the stacking faulted CBO nanogrooves provided a unique platform for investigating the effects of hydrated H_2O and stacking faults on the mechanical properties of nanomaterials. Nanoindentation tests were performed directly on individual CBOH nanobelts and CBO nanogrooves to probe their mechanical properties. It was found that the CBOH nanobelts, with the presence of hydrated H_2O , achieved 28.7% increase in elastic modulus, whereas the stacking faulted CBO nanogrooves, with the absence of hydrated H_2O , exhibited 99% loss in elastic modulus, compared to bulk CBO.

(3) ZnO nanostructures, one spotlight of functional and structural nanobuilding blocks in nanoelectronics and nanooptoelectronic devices, have been intensively studied in the past decade. The thermal stability of such nanostructures needs to be carefully considered because of their extremely high surface areas. We observed the entire melting process of individual ZnO nanobelts in situ by atomic force microscopy (AFM) and transmission electron microscopy (TEM). We also demonstrated how size affects the melting point of ZnO nanobelts. The finding of melting point depression in ZnO nanobelts provides an additional design guideline for constructing robust ZnO nanodevices and opens up unprecedented opportunities for developing temperature sensors.

(4) A simple, low-cost technique that combines hydrogen passivation (HP) with ultrasonication has been developed to separate graphene flakes into monolayered sheets with an excellent dispersion in both absolute ethanol solution and epoxy resin, overcoming the major roadblock in the application of graphene. The yield is based on the quantity of hydrogen introduced into the solution and the power of ultrasonication. The fabricated graphene/epoxy composites exhibit simultaneously increase in elastic modulus, fracture strength and toughness. The elastic modulus, fracture strength, and fracture energy values of the 1.0 wt.% graphene/epoxy composites fabricated by the coupled HP and ultrasonication are 12.28 ± 0.84 GPa, 121.34 ± 5.48 MPa, and 0.030 ± 0.004 J, respectively, demonstrating respective 103.3%, 47.9%, and 111.2% increase compared to those of the matrix material. The coupled HP and ultrasonication method should find more applications where the structural uniqueness and exceptional properties of graphene need to be fully utilized.

(5) By coupling scanning transmission electron microscopy (STEM) and digital image correlation (DIC) techniques,

atomic-scale imaging correlation successfully unveiled local strain fields induced by lattice distortion in the surface layers of SnO₂ nanowires. With the help of DFT, a clearer picture of the atomistic surface configuration of the nanowires was unraveled, showing a reduced surface with subsurface oxygen vacancies. The vacancies were found to be responsible for the large and vigorously alternating strains identified by DIC near the surface. Such configuration better explains the size effect on the mechanical properties measured by nanoindentation, unveiling a mixed effect from surface energy and expanded bond length due to vacancy formation. Such configuration also better explains the superior gas sensing capabilities of SnO₂ nanowire networks. The atomic-scale imaging correlation technique should find more applications in other nanostructures, providing an in-depth understanding of the correlation between the surface atomistic structure and nano-properties.

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This project resulted in the development of novel potentiometric and multimodal detection methodology for analytes, and fabrication of Silicon (Si) and Gallium Nitride (GaN) based piezoresistive microcantilever sensors that can be used to detect chemical and biomolecules based on either their static deflection or change in resonance amplitude. Microcantilevers, which resemble tiny diving boards, are highly sensitive to a variety of physical parameters, such as temperature, electronic charge, electrostatic potential, stress, and mass loading, which can be used to develop extremely sensitive physical, chemical, and biological sensors. A major impediment in developing these sensors, either in miniaturized form or in an array, is the problem of detecting nanoscale changes in deflection (both static and dynamic), which are almost exclusively measured by optical means, which cannot be easily miniaturized. This project has successfully addressed this problem by fabricating both Si and GaN microcantilevers with ultrasensitive AlGaIn/GaN field effect transistors integrated at the base of these cantilevers. We have shown that due to special piezoelectric properties of III-V Nitrides, these transistors can very efficiently transduce the static or dynamic deflections of the cantilever, leading to strain gauge factors in the range of few thousands, and offering performance very close to that of optical transduction systems. In addition, novel potentiometric and multimodal detection methodology has been developed in this project to perform highly sensitive and selective detection of common gaseous analytes.

In this project, novel techniques for designing microcantilever sensors and predicting their performance were developed using COMSOL Multiphysics based finite element simulations. Fabrication methodologies involving state-of-the-art photolithographic techniques were used to fabricate the microcantilever sensors and package them in the form of a chip. The sensor chips were then used to measure static deflection and resonant amplitude of the microcantilever, leading to the detection of electrostatic signals with very high sensitivity. The developed detection methodology led to the detection of H₂ down to a few ppm and NO₂ down to ppb ranges. In addition, the multimodal detection technique led to the determination of unique two-dimensional signatures of common analytes leading to their selective detection. The overall project led to 9 journal publications, 2 approved patents, 15 conference presentations, and 8 invited talks.

The project activities also led to very significant technological and educational broader impacts. In terms of technological broader impact, the development of the piezoresistive microcantilever sensor has opened up the possibility to detect a whole range of chemical, biological, and nuclear analytes. The project also resulted in the training of one postdoctoral researcher and three doctoral students. The training that the students obtained from this project enabled them to find employment in reputed companies such as Intel, Applied Materials, and Advanced Micro Devices. The intellectual properties generated from the research led to one start-up company (SENS4, LLC), which was successful in obtaining small business innovation research funding, and created employment for two persons. The project also led to the creation of research infrastructure (in terms of specialized equipment, test beds, and software), knowledge base, and expertise in the PI's lab, which is expected to lead to further application of this very promising technology in a large variety of fields.

Technology Transfer