



Defense Threat Reduction Agency  
8725 John J. Kingman Road, MS  
6201 Fort Belvoir, VA 22060-6201



DTRA-TR-21-17

# TECHNICAL REPORT

## IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

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HDTRA1-15-1-0036

Prepared by: Michael  
Alles

Vanderbilt University  
Nashville TN 37203

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**HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES**

**FINAL REPORT**  
**Grant #: HDTRA1-15-1-0036**

**Title:** IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

**PI Name:** Michael Alles<sup>1</sup>  
**Co-PIs:** K.I. Bolotin<sup>1\*</sup>, A. Zettl<sup>2</sup>, B. Homeijer<sup>3</sup>

**Organizations/Institutions:**  
Vanderbilt University, <sup>1</sup>EECS, <sup>1\*</sup>Physics, Nashville, TN  
<sup>2</sup>University of California, Berkeley, CA  
<sup>3</sup>Sandia National Laboratories – Albuquerque, NM

## **HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES**

### **Executive Summary:**

This 3-year research project applied experimental measurements and theoretical calculations to quantify mechanisms and manifestations of radiation effects on constituent MEM/NEM (M&NEM) materials and structures, including radiation-induced defects and resulting modification of mechanical properties of thin-film and 2D materials and devices. 2D materials are of particular interest due to the unique properties afforded by low atomic dimension in (at least) one direction. The same low-dimensional property also introduces questions as to mechanisms and models for interaction with radiation and how defects impact the mechanical properties.

Previous research has shown that the radiation-induced electrical response of un-passivated 2D materials is dominated by atmospheric contamination. When integrated into passivated electronic device structures (or measured in vacuum), the responses were found to be dominated by the expected responses of the adjacent surrounding thicker bulk materials. These are largely electrostatic effects, and similar to those seen in conventional devices, for example thin film silicon-on-insulator or multi-gate MOS electronic device structures. For M&NEMs, radiation-induced trapped charge in insulators can induce electrostatic forces that alter the electro-mechanical operation, in a sense throwing off the calibration; the counterpart of throwing off the threshold voltage of a transistor in electrical counterpart terms. As part of this research we applied experimental (10-keV X-rays and 14.3 MeV oxygen ions) and simulation-based analyses of bimorph electro-thermal MEMS actuators, using, and measuring corresponding changes in mechanical displacement using an optical microscope, to characterize how radiation-induced charging in Al/SiO<sub>2</sub> bimorph electro-thermal micro scanners results in changes in the vertical position and resistance of the structures.

For application of 2D materials for MEM/NEMs, it is more likely to require suspended membranes or stacks. Here, the nature of the interaction of radiation, especially particles, with the mono-layer suspended material, and the resulting defects and mechanical properties, is of particular interest. Underlying the study on how radiation-induced defect modify material properties, is the presupposition that one understands the material properties and relationships between defects and properties, and in fact has the ability to characterize these. In addition to a new theoretical modeling approach for radiation damage in 2D materials, this research project supported the advancement of both the capabilities to characterize 2D material properties, and the fundamental understanding of defect formation and properties of 2D materials through experiments and theoretical calculations. Unique experimental capabilities were developed for 2D materials characterization and for radiation testing, enhanced by interactions and common needs with other DTRA grants. Interactions with other grants also lead to joint publication with other groups.

The research results are detailed in the numerous presentations and publications listed in this report, as well as in three annual review reports from the program.

## HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

### Goal:

The goal of the research was to advance the understanding of the effects of radiation on material properties relevant to micro and nano scale electro-mechanical applications, particularly thin-film (ultimately 2D) materials. The research accomplished this goal through a series of materials characterization, radiation exposure experiments, and theoretical calculations.

### Accomplishments:

#### 1) *Major activities:*

- a) Radiation study of conventional (not 2D materials) MEMs functional devices
- b) Advancement of experimental characterization capability for 2D material properties
- c) Experimental study of radiation-induced defect formation in specific 2D materials
- d) Theoretical modeling of the interactions of particle radiation with 2D materials

#### 2) *Specific Objectives:*

The specific objectives were to *advance the understanding of:*

- a) the impact of trapped radiation-induced charge (total-ionizing-dose effects) on operation of MEMs devices which contains layers of adjacent thin films
- b) 2D materials structural and mechanical properties
- c) radiation-induced defect formation in 2D materials
- d) changes in mechanical properties of 2D materials due to radiation-induced defects

#### 3) *Significant Results*

The detailed results of this program have been reported in three annual review presentations and reports, and detailed in numerous publications as listed as references at the end of this report.

Key scientific findings are summarized as follows:

- Charge trapping at the interfaces of PZT and electrodes used in a Piezoelectric Micro-machined Ultrasonic Transducer was found to change the resonant frequency as a function of x-ray exposure dose and bias (including polarity dependence in the direction of the shift) during exposure. The observed effects are due to bias-dependent transport and trapping of radiation-induced charge at oxygen-vacancy **defects near the electrodes** that alters the stress and strain of the PZT.

## HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

- Radiation-induced charging in Al/SiO<sub>2</sub> bimorph electrothermal microscanners was shown to result in changes in the vertical position and resistance of the structures. The radiation-induced changes in the vertical position and resistance and post-irradiation recovery or super-recovery of these quantities are sensitive to DC biases applied during and/or after irradiation. Simple electrostatic and computational models are qualitatively consistent with observed trends in device response. Despite the observed changes in z-position and resistance caused by irradiation or bias-induced charging effects, the microscanners remained functional and controllable through the full series of tests performed in this study. This suggests that a suitable, feedback and control system could be applied to compensate for radiation or dielectric charging induced changes in resistance and z-position; however the radiation sensitivity of this control system would also have to be considered.
- Characterization methods were developed and applied to measure mechanical properties of 2D materials, finding that intrinsic crumpling of 2D graphene results in an increase in stiffness
- Defects induced in graphene by high-energy particle irradiation were found to be self-passivated in multi-layer graphene
- The structure of defects induced in hexagonal Boron Nitride subject to electron-irradiation at elevated temperature was experimentally found to be a function on the temperature
- A new use of 2D material to increase the sensitivity of measurements using MEMS was demonstrated and presents some new opportunities for the study of radiation effects
- Monte Carlo modeling, and experimental data, were combined to provide the first estimate of the defect levels vs. magnitude of mechanical property changes in 2D graphene. As mechanical structures scale from the  $\sim \mu\text{m}$  thickness regime to atomic dimensions (2D), the distinction of bulk vs. surface (or interface effects) becomes unclear, and can be dominated by adjoining materials if present (vs. suspended materials). To advance the understanding of the impact of exposure to particles (protons, electrons, ions) on fundamental mechanical properties of 2D materials, we developed and applied computational Monte Carlo models, as well as experimental techniques. The common approach to simulate ion interactions with bulk materials using TRIM does not work with 2D materials.

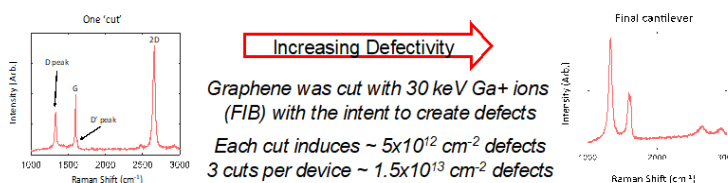
# HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

A summary of the findings on the 2D materials is represented in the figures below.

Direct experimental characterization of the impact of radiation on the mechanical properties of 2D materials and structures presents several challenges.

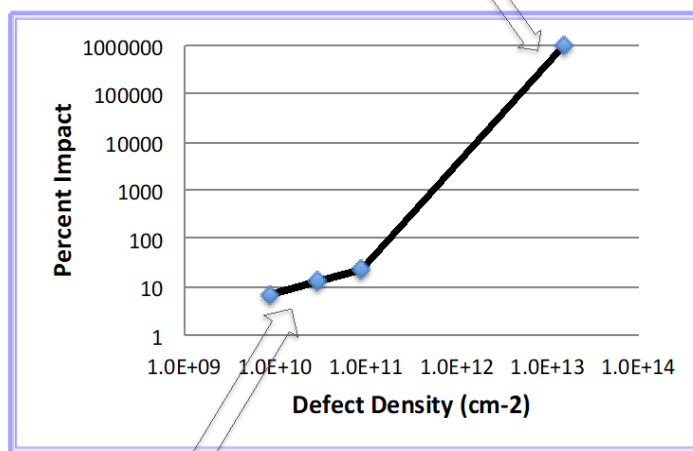
Integration of characterization methods for the 2D materials with radiation sources of practical interest for in-situ characterization is not straightforward. Use of TEM imaging is effective in examining the structural nature of defects, but even with the electrons serving as both the radiation source as well as the imaging method, this does not directly measure mechanical properties. By linking defect creation due to radiation exposure, with mechanical changes due to defects, we can surmise the change in mechanical properties due to the radiation exposure as shown in the series of figures. In the figure shown, there is a simple linear connection across 2 orders of magnitude in defect

density. At the end of the 3 year period, we had developed an experimental path to help to fill in the gap. Unique sample holders were designed, and materials samples were prepared. Potential radiation source options were identified. The idea was to use the new Monte Carlo simulations to help identify the radiation dose, based on the source parameters, required to create defect densities in the gap region. The plan included a collaboration with Case Western Reserve University for non-destructive optical characterization of the material properties pre- and post-radiation exposure and defect characterization by U.C. Berkley. The program was ended without option year awards so this experiment remains an opportunity for future work. Figures on the samples, holders, and experimental characterization approach are shown below.



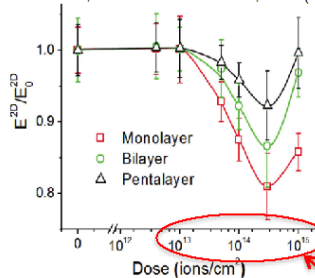
Mechanical response with static bending deflections from interferometry and from resonances  $\sim 1$  MHz observed in the SEM:

**Bending stiffness ( $\kappa$ ) increased by > 1000X due to defects**  
 Pristine graphene  $\kappa = 1$  eV vs. Defective graphene  $\kappa > 1$  KeV



Experimental: 3MeV He<sup>2+</sup>Irradiation & Elastic Modulus of Graphene

K. Liu et al., Advanced Materials 27, 6841 (2015)

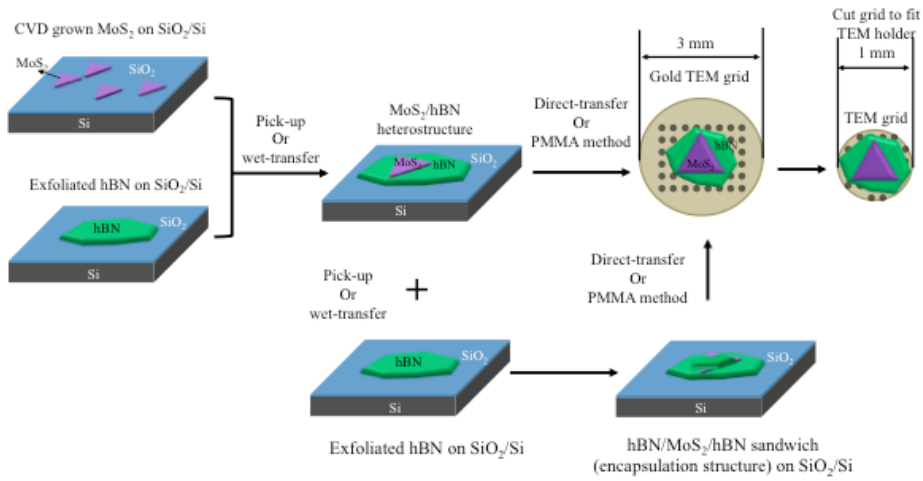


Monte-Carlo Simulations (this project)

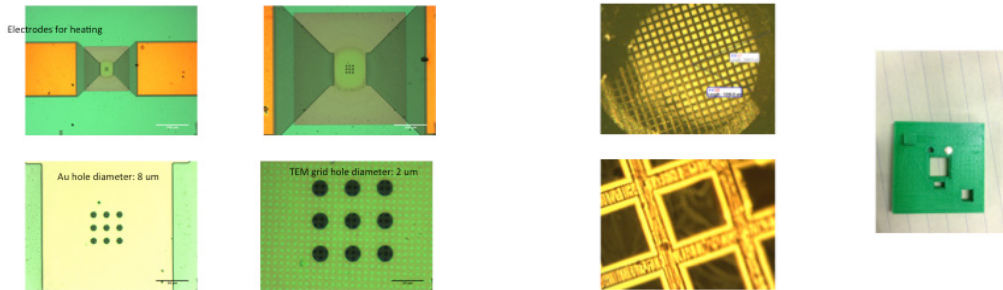
Ion	Energy	MC/DY Kinchin-Peace	New MC/DY elastic collision and molecular simulation	Exp. DY
C	500 keV	0.033	0.02	0.032
He	3 MeV	$3.6 \times 10^{-4}$	$1.63 \times 10^{-4}$	$3.54 \times 10^{-4}$
He	3 MeV	$3.5 \times 10^{-4}$	$1.59 \times 10^{-4}$	$2.3 \times 10^{-4}$
Ag	100 MeV	$3.5 \times 10^{-3}$	$2.2 \times 10^{-3}$	$2.3 \times 10^{-3}$
H	2 MeV	$8.1 \times 10^{-5}$	$1.5 \times 10^{-5}$	$4.32 \times 10^{-5}$

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## 2D SAMPLE PREP and PICTURES OF SAMPLES (U.C. Berkley)

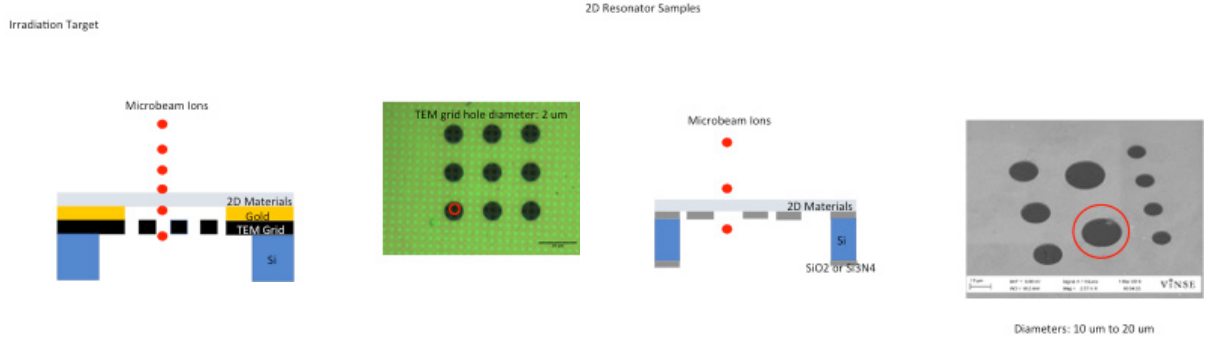


Holder design for the TEM foil

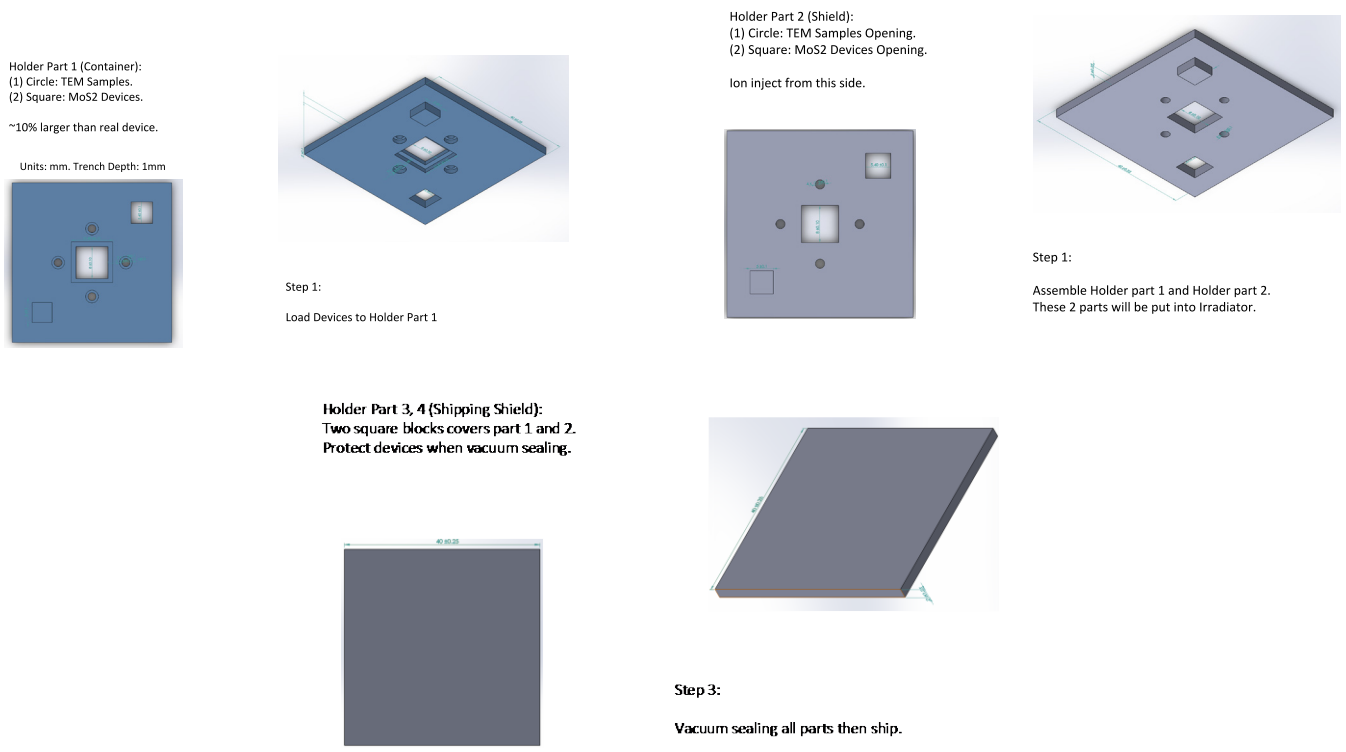


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## CONCEPTUAL REPRESENTATION FOR IRRADIATION (VU/Sandia)

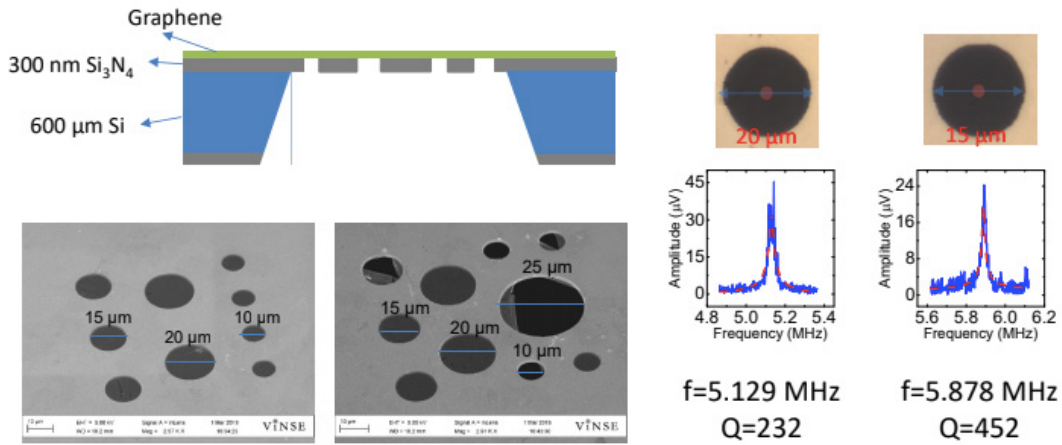


## SAMPLE HOLDERS FOR 2D MATERIALS IRRADIATION (VU/Sandia)



**HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES**

**EXAMPLE RESONANCE CHARACTERIZATION OF 2D SAMPLE BY CWRU (non-irradiated)**



**4) Key Outcomes and Other Achievements:**

The program resulted in 17 publications and 15 conference presentations as listed below. The program supported 3 Ph.D. graduate students. Findings advanced the understanding of the effects of radiation on MEMS, and in particular the impact of radiation exposure on the defectivity and mechanical properties of 2D materials, including development and demonstration of a Monte-Carlo modeling approach for simulation of the interaction of ions with 2D materials.

## Publications

1. R.J.T. Nichol, N.V. Lavrik, I. Vlassiuk, B. R. Srijanto, and K.I. Bolotin, "Hidden area and mechanical nonlinearities in freestanding graphene", *Phys. Rev. Lett.*, 118, 266101, 10.1103/PhysRevLett.118.266101, (2017)
2. Ryan J.T. Nicholl, "Non-Hookean mechanics of crystalline membranes," Ph.D. Dissertation, Vanderbilt University, <https://etd.library.vanderbilt.edu/available/etd-07182017-013046/>, published July 2017
3. S.M. Gilbert, G. Dunn, A. Azizi, T. Pham, B. Shevitski, E. Dimitrov, S. Liu, S. Aloni, and A. Zettl, "Fabrication of Atomically Precise Nanopores in Hexagonal Boron Nitride," *Nature Scientific Reports*, 10.1038/s41598-017-12684-x, (2017)
4. S. Matt Gilbert, Stanley Liu, Gabe Schumm, and Alex Zettl, "Nanopatterning Hexagonal Boron Nitride with Helium Ion Milling: Towards Atomically-Thin, Nanostructured Insulators," *MRS Bulletin*, 10.1557/adv.2018.117, (2018)
5. Wenjun Liao, Michael L. Alles, En Xia Zhang, Daniel M. Fleetwood, Robert A. Reed, Robert A. Weller, and Ronald D. Schrimpf, "Monte Carlo Simulation of Displacement Damage in Graphene," in preparation for submission to IEEE TNS (RADECS).
6. Wenjun Liao, "Radiation Effects on Microelectromechanical Systems," Ph.D. Dissertation, Vanderbilt University, August (2018)
7. Rajan Singh, Ryan J.T. Nicholl, Kirill Bolotin, Saikat Ghosh, "Motion transduction with thermo-mechanically squeezed graphene resonator modes," *Nano Lett.* 2018, 18, 11, 6719–6724, October (2018)
8. T. Pham, A.L. Gibb, A. Li, S.M. Gilbert, C. Song, S.G. Louie, and A. Zettl, "Formation and Dynamics of Electron-Irradiation-Induced Defects in Hexagonal Boron Nitride at Elevated Temperatures," *Nano Letters* 16, 7142 (2017)
9. W. Liao, E. X. Zhang, M. L. Alles, C. X. Zhang, H. Gong, K. Ni, A. L. Sternberg, H. Xie, D. M. Fleetwood, R. A. Reed, and R. D. Schrimpf, "Total-Ionizing-Dose Effects on Piezoelectric Micromachined Ultrasonic Transducers," *IEEE Trans. Nucl. Sci.*, vol. 64, no. 1 (2017)
10. Charles N Arutt *et al.*, 2017 *Semicond. Sci. Technol.* 32 013005 (Describes 5 DTRA grants in this topic) **[Invited]**
11. R.J.T. Nicholl, N.V. Lavrik, I. Vlassiuk, B. R. Srijanto, and K.I. Bolotin, "Hidden area and mechanical nonlinearities in freestanding graphene", in press, *Phys. Rev. Lett.* (2017)
12. S.M. Gilbert, G. Dunn, A. Azizi, T. Pham, B. Shevitski, E. Dimitrov, S. Liu, S. Aloni, and A. Zettl. Fabrication of Atomically Precise Nanopores in Hexagonal Boron Nitride, APS March Meeting 2018, abstract id.B18.012 (2018)
13. Ryan J.T. Nicholl, Hiram J. Conley, Nickolay V. Lavrik, Ivan Vlassiuk, Yevgeniy S. Puzyrev, Vijayashree Parsi Sreenivas, Sokrates T. Pantelides & Kirill I. Bolotin, "The effect of intrinsic crumpling on the mechanics of free-standing graphene", *Nature Communications*, 6:8789 (2015); selected for DOE headquarters highlight and Outstanding Student Publication award at Vanderbilt (2016)

## HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES

14. M. Alles, K. Bolotin, A. Zettl, B. Homeijer, J. Davidson, R. Schrimpf, R. Reed, D. Fleetwood, "Radiation Effects in M&NEMS," GOMACTech Proceedings (2016)
15. K. Liu, C.-L. Hsin, D. Fu, J. Suh, S. Tongay, M. Chen, Y. Sun, A. Yan, J. Park, K.M. Yu, W. Guo, A. Zettl, H. Zheng, D.C. Chrzan, and J. Wu., "Self-passivation of defects: effects of high-energy particle irradiation on the elastic modulus of multilayer graphene," *Adv. Mat.* 27, 43 pp. 6841-6847 doi: 10.1002/adma.201501752 (2015)
16. W. Liao, M. L. Alles, E. X. Zhang, D. M. Fleetwood, R. A. Reed, R. A. Weller, and R. D. Schrimpf, Ronald D. Schrimpf, "Monte Carlo Simulation of Displacement Damage in Graphene," *IEEE Trans. Nucl. Sci.*, Feb (2019), doi: 10.1109/TNS.2019.2899579
17. W. Liao, *Radiation Effects on Microelectromechanical Systems*, Ph.D. Dissertation, Vanderbilt University, 2018.

### Presentations

1. Wenjun Liao, Michael L. Alles, En Xia Zhang, Daniel M. Fleetwood, Robert A. Reed, Robert A. Weller, and Ronald D. Schrimpf, "Monte Carlo Simulation of Displacement Damage in Graphene, 2018 RADECS.
2. R. Singh, R. Nicholl, S. Chakraborty, K. Bolotin, and S. Ghosh, "Motion transduction with suspended Graphene resonators", APS March Meeting, Los Angeles, CA, March 7, 2018.
3. K. Bolotin, "Bending, pulling and stretching crumpled two-dimensional materials", IWPENM international conference in Kirchberg, March 2018.
4. W. Liao, E. X. Zhang, M. L. Alles, C. X. Zhang, H. Gong, K. Ni, A. L. Sternberg, H. Xie, D. M. Fleetwood, R. A. Reed, and R. D. Schrimpf, "Total-Ionization-Dose Effects on Microscanner with Al/SiO<sub>2</sub> Electrothermal Bimorph Actuators, IEEE NSREC, July 2017.
5. M.L. Alles, "Update on Radiation Effects in N&MEMs," Radiation Effects in Microelectronics Program Review, Vanderbilt University, May 2017
6. W. Liao *et al.* Total-Ionization-Dose Effects on Microscanner with Al/SiO<sub>2</sub> Electrothermal Bimorph Actuators, NSREC 2017
7. K. Bolotin, "Bending, cutting and pulling two-dimensional materials," CMD-26 Conference, Gronigen September 2016 **[Invited]**
8. E. X. Zhang, C. Liang, C. X. Zhang, D. M. Fleetwood, R. D. Schrimpf, M. L. Alles, R. A. Reed, K. Bolotin, and S. T. Pantelides, "Radiation effects in 2D materials and devices," University of Memphis, Physics Dept. Colloquium, Oct. 7, 2016 **[Invited]**
9. K. Bolotin, "Bending, cutting and pulling two-dimensional materials", NACRE GDR Conference Berlin, November 2016 **[Invited]**
10. M. Alles, "Radiation Effects in 2D Materials and Nano Electrical Mechanical Devices," AVS63, Nashville, TN, November 6-11, 2016 **[Invited]**
11. R Nicholl et. al, "Probing Mechanics of Crumpled Two-Dimensional Membranes and Cantilevers", APS March Meeting (2016)
12. Contribution P13: "Slipping and friction at the interface between two-dimensional materials" APS March Meeting (2016)

**HDTRA1-15-1-0036: IMPACT OF RADIATION DAMAGE ON MECHANICAL AND ELECTRICAL PROPERTIES OF MEM/NEM STRUCTURES**

13. T. Pham, A. L. Gibb, S. M. Gilbert, C. Song and A. Zettl. Formation and Dynamics of Electron-irradiation-induced Defects in Hexagonal Boron Nitride at Elevated Temperatures. Poster Presentation, MRS Spring, Phoenix, Arizona (2016)
14. M. Alles, K. Bolotin, A. Zettl, B. Homeijer, J. Davidson, R. Schrimpf, R. Reed, D. Fleetwood, "Radiation Effects in M&NEMS," GOMACTech (2016)
15. Total Ionization Dose Effect on Piezoelectric Micromachined Ultrasonic Transducers, Wenjun Liao, En Xia Zhang, Michael L. Alles, Cher Xuan Zhang, Huiqi Gong, Kai Ni, Andrew Sternberg, Daniel M. Fleetwood, Robert A. Reed and Ronald D. Schrimpf, 2016 NSREC

## **Training and Professional Development**

Ryan J.T. Nicholl, Wenjun Liao, and Tham Pham completed Ph.D. degrees under this DTRA program.

Professor Dan Fleetwood (Vanderbilt) taught: EECE 304. Radiation Effects and Reliability of Microelectronics. Prof. Dan Fleetwood, VU

The students, post-docs, and professors on the team attended numerous international conferences, which not only supported dissemination of project results, but also served to advance their personal profession development.

## **Dissemination of Results**

The results have been disseminated to communities of interest through the numerous publications and presentations as listed previously in this report. These presentations and publications include venues beyond the typical radiation effects community.