



AFRL-AFOSR-VA-TR-2019-0287

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**DURIP16 Atomic Force Microscopy and Surface Spectroscopy Instrumentation to Enable  
Nanoscale Tribology and Materials Research**

**Robert Carpick  
TRUSTEES OF THE UNIVERSITY OF PENNSYLVANIA**

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**08/29/2019  
Final Report**

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**Air Force Research Laboratory  
AF Office Of Scientific Research (AFOSR)/ RTB2  
Arlington, Virginia 22203  
Air Force Materiel Command**

**REPORT DOCUMENTATION PAGE**

Form Approved  
OMB No. 0704-0188

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<b>1. REPORT DATE (DD-MM-YYYY)</b> 08/26/2019	<b>2. REPORT TYPE</b> FINAL	<b>3. DATES COVERED (From - To)</b> 9/30/16-9/30/17 (5/31/19)
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<b>4. TITLE AND SUBTITLE</b> DURIP-16: Atomic Force Microscopy and Surface Spectroscopy Instrumentation to Enable Nanoscale Tribology and Materials Research	<b>5a. CONTRACT NUMBER</b> NA
	<b>5b. GRANT NUMBER</b> FA9550-16-1-0525
	<b>5c. PROGRAM ELEMENT NUMBER</b>

<b>6. AUTHOR(S)</b> R CARPICK, Ph.D.	<b>5d. PROJECT NUMBER</b>
	<b>5e. TASK NUMBER</b>
	<b>5f. WORK UNIT NUMBER</b>

<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> Trustees of the University of Pennsylvania 220 S. 33rd Street Philadelphia PA 19104	<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> FINAL
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<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> USAF, AFRL DUNS 143574726 AF OFFICE OF SCIENTIFIC RESEARCH 875 NORTH RANDOLPH STREET, RM 3112 ARLINGTON VA 22203-1954 ERNA PETRICH 703-696-6563 erna.petrich@us.af.mil	<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b> AOARD
	<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b>

**12. DISTRIBUTION/AVAILABILITY STATEMENT**  
DISTRIBUTION A: Distribution approved for public release.

**13. SUPPLEMENTARY NOTES**

**14. ABSTRACT**  
The purpose of this project was to perform a major upgrade of a multifunctional equipment system to enable new research for nanoscale tribology specifically, and more broadly, nanoscale materials characterization. The new capabilities are greatly benefiting a project supported by AFOSR. Furthermore, the instrumentation will benefit research whose impact is relevant to multiple aspects of DoD's mission. The instrumentation that was upgraded consists of two major systems: an ultrahigh vacuum atomic force microscope (AFM) and a surface spectroscopy system. These two powerful research tools are uniquely suited to complement each other to enable comprehensive studies of surfaces and interfaces, including...

**15. SUBJECT TERMS**

<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b>	<b>19a. NAME OF RESPONSIBLE PERSON</b> JILLIAN R CAWLEY, Sr Assoc Director
<b>a. REPORT</b>	<b>b. ABSTRACT</b>	<b>c. THIS PAGE</b>			<b>19b. TELEPHONE NUMBER (Include area code)</b> 215-573-6716
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# **Atomic Force Microscopy and Surface Spectroscopy Instrumentation to Enable Nanoscale Tribology and Materials Research**

**PI: Prof. Robert W. Carpick**

Dept. of Mechanical Engineering and Applied Mechanics  
University of Pennsylvania

## **Final Report**

### **Abstract: PUBLICLY RELEASABLE**

The purpose of this project was to perform a major upgrade of a multifunctional equipment system to enable new research for nanoscale tribology specifically, and more broadly, nanoscale materials characterization. The new capabilities are greatly benefiting a project supported by AFOSR. Furthermore, the instrumentation will benefit research whose impact is relevant to multiple aspects of DoD's mission. The instrumentation that was upgraded consists of two major systems: an ultrahigh vacuum atomic force microscope (AFM) and a surface spectroscopy system. These two powerful research tools are uniquely suited to complement each other to enable comprehensive studies of surfaces and interfaces, including studies of nanoscale tribology. More broadly, they enable probing of the composition and structure of novel materials using the surface science tools, and the structure, topography, and mechanical properties using AFM. The original system, which was ca. 8 years old at the time of the award (with some components >15 years old) has enabled significant research progress. However, the performance, throughput, and functionality were all seriously limited by its problematic configuration. Furthermore, new developments in control electronics and instrument components now offer significant benefits. The instrumentation described in the proposal was acquired, delivered, installed, tested, and is performing at the expected level and providing research quality results. Specifically, both the AFM system, and the surface science system are working and in regular use.

## **Final Report**

### **Atomic Force Microscopy and Surface Spectroscopy Instrumentation to Enable Nanoscale Tribology and Materials Research**

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The instrumentation described in the proposal was acquired, delivered, installed, tested, and is performing at the expected level and providing research quality results. Specifically, both the atomic force microscope (AFM) system, and the surface science system are working and in regular use. Below are examples of data obtained with the AFM system and the surface science system.

The new capabilities are greatly benefiting a new project supported by AFOSR through the Asian Office of Aerospace Research and Development (AOARD) within the US-Taiwan Nanoscience Program. It is also benefiting a Phase II STTR grant funded by the U.S. Army Combat Capabilities Development Command (CCDC) on improving the efficiency and reliability of ground vehicles. Furthermore, the instrumentation will benefit research pursuits whose impact is relevant to multiple aspects of DoD's mission and beyond, including work funded by other federal agencies and industry.

Presently, we have multiple publications that are in preparation or submitted/under review which have benefited from and will acknowledge this grant.

The instrumentation has now allowed multiple graduate students, undergraduate students, and postdoctoral researchers to gain training and experience with both state-of-the-art atomic force microscopy and surface science techniques. In addition to enhancing the resources for DoD funded research, the system users have helped contribute to the trained workforce with knowledge that can help solve many issues of interest to the DoD. This includes two Ph.D. students who were both military veterans, who successfully completed and defended their Ph.D.'s and now have full-time employment as researchers.

This instrumentation enhances the capabilities of our existing instrumentation, allowing for more sophisticated and precise surface characterization under ultrahigh vacuum conditions. Nanoscale friction and wear will be investigated with greater sensitivity to both topological and frictional features with AFM and chemical bonding with surface science. These resources will create a place for cutting edge research into the fundamental tribological interactions at the nanoscale.

See images on next page.

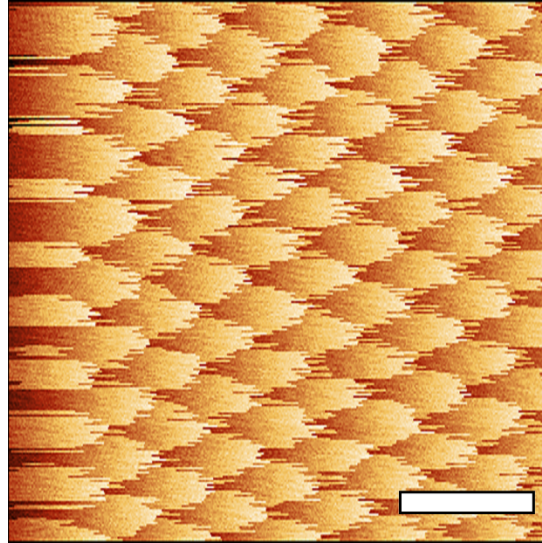


Fig. 1. Experimental AFM lateral force image of a 2H MoS<sub>2</sub> bulk sample acquired in ultrahigh vacuum using the instrumentation that was upgraded by the DURIP funding. The scale bar is 0.5 nm. The periodic pattern is produced by stick-slip frictional motion that matches the spacing and symmetry of the MoS<sub>2</sub> surface lattice. The high signal-to-noise and the overall clarity of the image demonstrates the excellent performance of the equipment. Sample grown by CVD and provided by Prof. A.T. Johnson, UPenn.

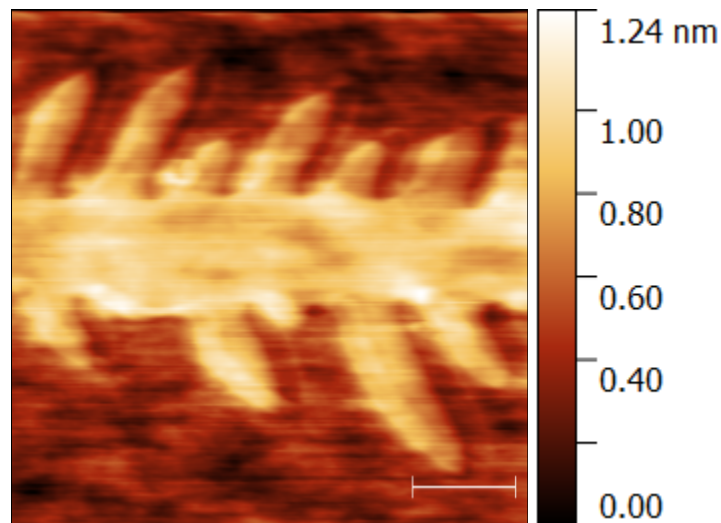


Fig. 2. Experimental AFM topographic force image of a 2H MoS<sub>2</sub> dendrite, which is present on top of a MoS<sub>2</sub> monolayer supported on a silicon substrate. Image acquired in ultrahigh vacuum. The scale bar is 100 nm. The image shows extremely high lateral and vertical resolution, demonstrating the excellent performance of the UHV system. Sample grown by CVD and provided by Prof. A.T. Johnson, UPenn.

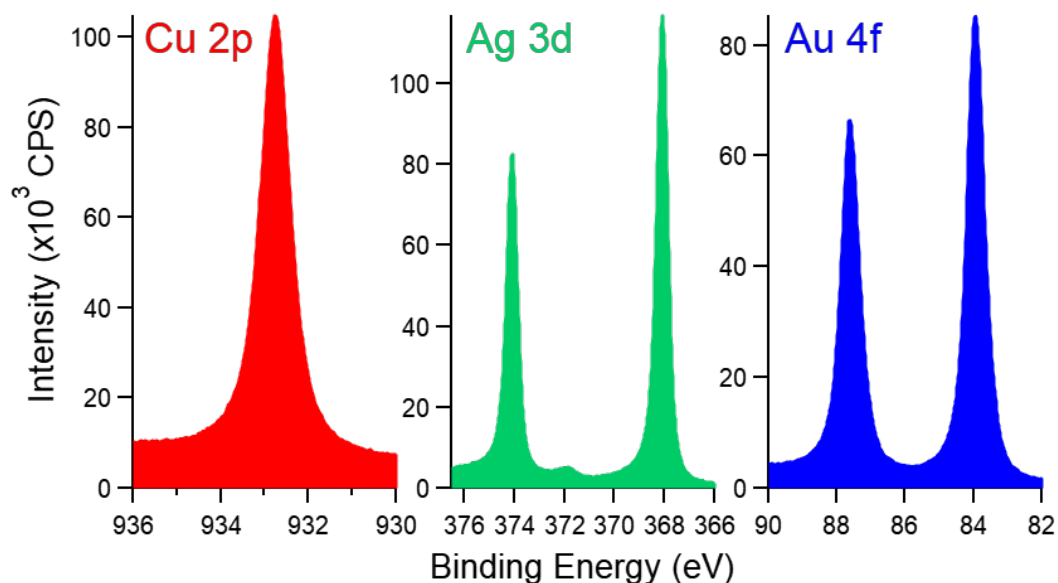


Fig. 3. High resolution XPS spectra acquired after the system upgrade. This is an example of a 3-point calibration data procedure that is performed to check intensities, peak positions, and peak widths measured to verify instrument performance. The samples measured are ultra-pure metal foils which have been sputter cleaned immediately before data acquisition to ensure the metals are clean and in their lowest oxidation state at the surface. The measured binding energies are within 0.15 eV of their reference values, confirming good linearity of the spectrometer across the full range of accessible binding energies. All full width at half maximum (FWHM) values are exceptionally low, near their theoretical limits, ensuring that resolution for more complex samples will be optimal. Furthermore, intensity values after the upgrade are the highest we have ever measured, a characteristic which is paramount for maintaining the high spatial resolution enabled by the upgrade. The overall intensity is subdivided in spatially resolved spectroscopy, so a high intensity improves both the resolution and the required acquisition time during such measurements. We note that a custom-built UHV tribometer, supported by other funding, is in final testing before pairing with the spatially-resolved XPS to allow for direct, *in situ* comparison of the surface chemistry for worn and unworn regions of samples of interest.