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Advancing Layered Two-Dimensional Semiconductors

JEREMY ROBINSON
CORY CRESS
JOSE FONSECA
JAMES CULBERTSON
EVAN GLASER

Power and Advanced Materials Branch
Electronics Science and Technology Division

THOMAS REINECKE

Senior Scientist for Nanoelectronics
Electronics Science and Technology Division

GLENN JERNIGAN
ANDREW YEATS

Quantum and Optoelectronics Branch
Electronics Science and Technology Division

BLAKE SIMPKINS
DANIEL RATCHFORD

Quantum and Optoelectronics Branch
Electronics Science and Technology Division

TODD BRINTLINGER

Materials and Sensors Branch
Materials Science Division

MAXIM ZALALUTDINOV

Physical Acoustics Branch

Acoustics Division

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Jeremy Robinson, Thomas Reinecke, James Culbertson, Cory Cress, Andrew Yeats, Glenn Jernigan, Jose Fonseca, Evan Glaser, Blake Simpkins, Daniel Ratchford, Todd Brintlinger, Maxim Zalalutdinov

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Semiconductors form the basis of modern electronics and their success is a result of decades of research in material development, which ranges from high-quality crystalline samples to doping/alloying to engineering the semiconductor/metal interface. In this program, we aim to springboard the development of a relatively new class of two-dimensional (2D) semiconductors, the transition metal dichalcogenides, in an effort to exploit their atomically thin form factors that are ideal for Navy systems, including radiation-hardened electronics, optoelectronics, and sensors. Their 2D structure, coupled with a direct bandgap in the visible to near infrared, makes them highly suitable for ultralow-power electronics with low leakage and extreme electrostatic control. The program efforts rely on detailed spectroscopic characterization and theory to unravel the interplay between surface and defect states, and metal/semiconductor interfaces, which are essential for building advanced electronic systems for naval applications.

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19a. NAME OF RESPONSIBLE PERSON	19b. PHONE NUMBER *(Include area code)*
Jeremy Robinson	(202) 404-4517

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EXECUTIVE SUMMARY

There is a current need within the Navy and the DoD for high-performance devices for sensors, satellite electronics, and ultracompact RF components, in addition to further-reaching next-generation technologies employing quantum-based sciences. Advances beyond traditional electronic components require research and development of both basic materials systems and component design. For example, semiconductors form the basis of modern electronics, and their success is a result of decades of research in material development, which ranges from high-quality crystalline growth to defect passivation to doping/alloying. In this research program, we sought to springboard the development of a new class of two-dimensional (2D) semiconductors, the transition metal dichalcogenides (TMDs), in an effort to exploit their atomically thin form factors that are ideal for Navy systems, including radiation-hardened electronics, optoelectronics, and sensors. Exploiting these features requires research into the metal/atomically thin semiconductor interfaces, defect properties and passivation, and spatially controlled doping, among others. A wide range of NRL capabilities were employed to form research-grade 2D semiconductor samples and device structures, as well as their subsequent characterization. These efforts relied on detailed spectroscopic assessment and theoretical modeling to unravel the interplay between surface and defect states, and metal/semiconductor interfaces, which are essential for building advanced electronic systems for naval applications. This research provides a framework for using and implementing 2D semiconductors in the following Naval Research and Development Framework areas: information, cyberspace, and spectrum superiority, advanced RF electronics and materials, high-performance, solid-state electronic components, subsystems, and devices for military RF systems, and platform design and survivability (solid-state electronics).

This report has research contributions from NRL scientists Dr. Jeremy Robinson (6883), Dr. Thomas Reinecke (6803), Dr. James Culbertson (6883), Dr. Cory Cress (6883), Dr. Andrew Yeats (6819), Dr. Glenn Jernigan (6812), Dr. Jose Fonseca (6883), Dr. Evan Glaser (6882), Dr. Blake Simpkins (6178), Dr. Daniel Ratchford (6178), Dr. Todd Brintlinger (6366), and Dr. Maxim Zalalutdinov (7133).

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ADVANCING LAYERED TWO-DIMENSIONAL SEMICONDUCTORS

1 INTRODUCTION

1.1 Objective

The Naval Research Laboratory (NRL) conducted this research to create, understand, and manipulate large-area, two-dimensional (2D) semiconductors. The technical approach explored the formation and modification of light-emitting processes in 2D semiconductors, understanding the interface interactions between 2D semiconductors and metals, and tuning the activation barriers for semiconductor-to-metal phase changes. Their layered structure, coupled with a direct bandgap in the visible-to-near-infrared portion of the electromagnetic spectrum, has particular suitability for digital electronics in the extreme size-scaling limit, especially for use in harsh radiation environments, and for optoelectronics.

1.2 Motivation

2D layered materials have emerged as an important platform to explore basic science questions and to build new technologies that exploit their uniquely thin form factors. Fundamentally, 2D materials offer a range of properties that far surpass conventional materials at similar length scales thanks to the weak van der Waals interactions that bind individual layers. Technologically, the road map for 2D materials includes electronic devices ranging from highly selective and sensitive sensors, to high-frequency/logic transistors, to optoelectronic and phase-change elements. Of particular interest to the Navy is the potential for these materials to enable superior performance of electronic and optoelectronic systems in the areas of low-dimensional electronic materials for advanced components, quantum information and sensor science and technology, and radiation-hard or -immune sensing and computing.

While the best-known 2D material is graphene, whose carbon backbone offers high stability and excellent properties as an atomically thin conductor, conventional and future electronics require well-controlled materials with a bandgap. Filling this niche within 2D materials are the transition-metal dichalcogenides (TMDs), which can be formed as monolayers (~6 Å thick) and represent an ultimate thinness limit for semiconductors. Their 2D structure coupled with a direct bandgap in the visible to near infrared makes them highly suitable for ultralow-power electronics with low leakage (due to nonzero bandgap) and extreme electrostatic control (due to thinness). These features open possibilities to develop and integrate novel optoelectronic devices based on 2D TMDs to improve performance, to reduce power, and to improve radiation tolerance. This, in turn, drives the need to create and understand new growth and doping techniques that work in harmony with the chemistries and “all-surface” nature of these materials.

TMD semiconductors are in their early stages for material development as compared to ubiquitous silicon, which has six decades of research in growth, defects, passivation, and doping. While the field of 2D materials has matured significantly in the past decade, we are entering a second phase of basic research in which large-area material growth and development are critical for transitioning newly discovered properties and device concepts to Navy-relevant technologies. The ability to modify the local carrier

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concentration in conventional semiconductors forms the basis of all solid-state electronic and optoelectronic device technologies, and it is critical to achieve this same level of control in 2D semiconductors. For example, high-quality Ohmic contacts and controlled doping are important challenges to overcome. Recent studies also reinforce the importance of 2D semiconductor defects, influencing variables such as carrier scattering, energy barriers between phases, or single quantum emitters.

2 APPROACH

This program combined materials synthesis/fabrication, spectroscopy, and theory to quantify and assess the properties of atomically thin semiconductors, with a focus on interactions with metals under readily accessible laboratory conditions.

The ever-maturing experimental techniques for manipulating, transferring and characterizing 2D materials were applied to studying the 2D semiconductors examined here. The primary techniques brought to bear in this program include Au-assisted exfoliation and transfer of large-area monolayer films, semiconductor lithography and fabrication, “dry” (e.g., implantation) and “wet” modification chemistries, transport, spectroscopies, and theoretical calculations using density functional theory (DFT). We utilized expertise from multiple branches to carry out the experimental and theoretical techniques.

Detailed characterization and analysis of material structure and bonding were central to the program. We implemented numerous spectroscopies and designed novel experiments to study not only the base materials, but also materials that underwent a variety of postprocessing treatments and interface interactions. A combination of spectroscopies including Raman, photoluminescence (PL), X-ray (XPS) and photoemission (PEEM), and electron diffraction were used to elucidate chemical and electronic structures of our material systems. The XPS core-level spectra shows the local chemical states of the metal or chalcogen atoms, any band-bending that may occur from electron transfer between the film and the substrate, and the elemental ratios within the film. Raman spectroscopy was used to quantify the layer number and the built-in strain. PL was used to identify the presence of radiative and nonradiative defect states, while time-resolved studies (“g2” measurements) informed on the presence of single-photon-emitting centers within our 2D semiconductors.

From the perspective of future electronic materials, one argument for implementing 2D TMDs is the fact that their electronic properties such as mobility, while not competitive with bulk silicon, exceed those of silicon in the extreme size-scaling limit. Specifically, as silicon devices are scaled downward their favorable electronic properties drop below those of 2D TMDs. Basic electronic properties were examined by fabricating test structures and analyzing: (i) contact resistance, (ii) carrier type, concentration, and Hall mobility, and (iii) I-V and gated-transfer-characteristics of FET structures to determine the basic electrical device properties and ambient stability

Theoretical work was implemented to determine the optimal conditions and parameters for materials formation, to predict new materials systems, and to guide experimental work. These techniques included first-principles density function theory for ground-state structures, for electronic properties, and for vibrational properties, as well as ab initio molecular dynamics techniques. All of these theoretical techniques are well-developed within the team and were applied to the layered structures to elucidate the chemical functionalization and the role of substrates of interest in this program.

Understanding how processing-related defects influence the intrinsic material properties and how they may negatively or positively influence the optical and electrical device performance is critical. Defect correlation studies were accomplished by introducing defects and strain into the 2D semiconductors using

incident particles such as electrons, protons, and ions, and correlating the rate of change of physical, electrical, and optical properties with defect concentration.

3 EXPERIMENTS

The following subsections provide an overview of selected experimental efforts within the research program. The full research output of the program can be found in [1–18].

3.1 Enabling Remote Quantum Emission in 2D Semiconductors via Porous Metallic Networks

In this effort, we demonstrated how two-dimensional crystal (2DC) overlayers influence the recrystallization of relatively thick metal films and the subsequent synergetic benefits this provides for coupling surface plasmon-polaritons (SPPs) to photon emission in 2D semiconductors (Fig. 1). We found that annealing 2DC/Au films on SiO_2 results in a "reverse epitaxial" process by which initially nanocrystalline Au films gain texture, crystallographically orient with the 2D crystal overlayer, and form an oriented porous metallic network (OPEN) structure in which the 2DC can suspend above or coat the inside of the metal pores. Both laser excitation and exciton recombination in the 2DC semiconductor launch propagating SPPs in the OPEN film. Energy in-/outcoupling occurs at metal pore sites, alleviating the need for dielectric spacers between the metal and 2DC layers. At low temperatures, single-photon emitters (SPEs) are present across an OPEN-WSe2 film, and we demonstrate remote SPP-mediated excitation of SPEs at a distance of 17 μm. For more details, see [3].

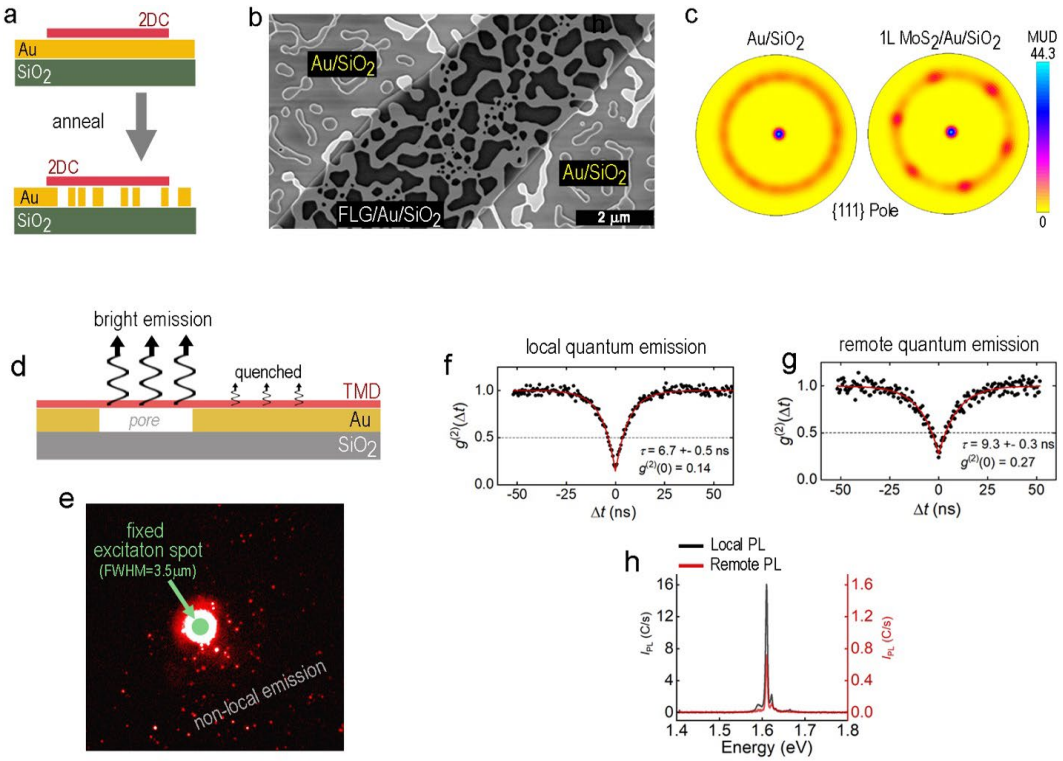

Fig. 1—Process and characterization of the OPEN gold/2D layer structure. (a) Schematic showing the formation of the OPEN structure after annealing a 2DC/Au film on glass. (b) SEM image showing a graphene layer on the porous gold structure. (c) Diffraction showing the gold layer recrystallizes underneath a MoS_2 monolayer to form a quasiepitaxial interface. (d–e) Light emission strongly occurs at the suspended semiconductor over the gold pores and is quenched where the semiconductor directly touches Au. (e) Fluorescence image showing non-local light emission at pore sites due to plasmonic coupling to the porous Au layer. (f–h) Time-correlated measurements ("g2") showing quantum emission at certain suspended semiconductor sites, locally (f) and remote (g). (h) PL spectra of the local and remote emission.

3.2 Electronic Changes in Molybdenum Dichalcogenides on Gold Surfaces

In this effort, we use X-ray and ultraviolet photoelectron spectroscopy on monolayers of transition metal dichalcogenides (TMDs) MoS_2, $MoSe_2$, and $MoTe_2$ on Au surfaces to identify charge transfer processes and changes in structural phase upon annealing up to 500°C (Fig. 2). Although charge transfer does appear to occur as the spacing between the TMD monolayer and the Au surface decreases, we did not find spectroscopic evidence for the 2H- to 1T-phase change in exfoliated TMDs. However, we find the defect density of the TMDs influences the evolution of the X-ray photoelectron spectra during thermal treatment on Au. Both metal-organic chemical vapor deposition grown and helium-ion irradiated exfoliated TMDs annealed on Au surfaces show shifts in their spectra that could be interpreted as a phase change, but are the result of TMD/Au hybridization, grain boundaries, and defects interacting with the Au surface. For more details, see [12].

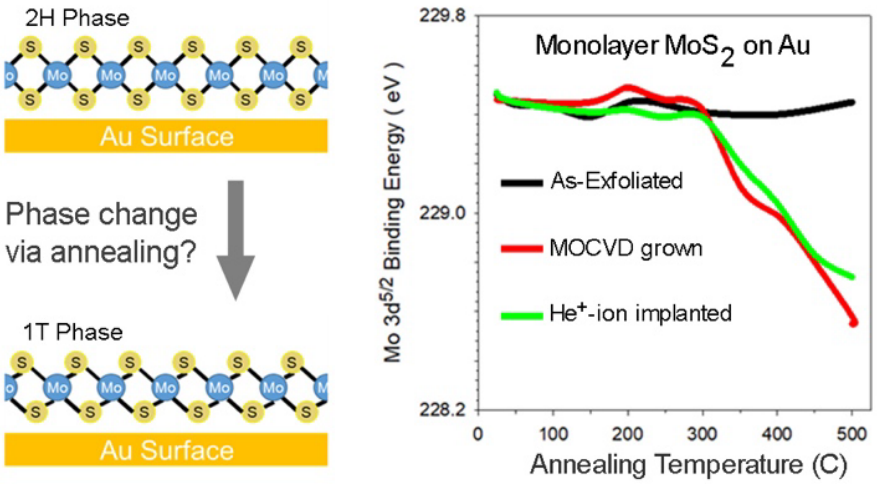

Fig. 2—(Left) Schematic showing possible interactions of TMD semiconductors with an Au surface. (Right) XPS data showing the position of the Mo core level as a function of substrate treatment. When defects are present, the TMD films show large shifts in their bonding state (red and green lines), while defect-free samples (black line) show no change.

3.3 Electronic Structure and Stacking Arrangement of Tungsten Disulfide at the Gold Contact

There is an intensive effort to control the nature of attractive interactions between ultrathin semiconductors and metals and to understand their impact on the electronic properties at the junction. In this effort, we used photoelectron spectroscopy to analyze the interface between WS_2 films and gold, with a focus on the occupied electronic states near the Brillouin zone center (Fig. 3). To delineate the spectra of WS_2 supported on crystalline Au from the suspended WS_2, we employ a microscopy approach and a tailored sample structure in which the WS_2/Au junction forms a semi-epitaxial relationship and is adjacent to suspended WS_2 regions. The photoelectron spectra, as a function of WS_2 thickness, display the expected splitting of the highest occupied states at the Brillouin zone center. In multilayer WS_2, we discovered variations in the electronic states that spatially align with the crystalline grains of underlying Au. Corroborated by density functional theory calculations, we attribute the electronic structure variations to stacking variations within the WS_2 films. We propose that strong interactions exerted by Au grains induce slippage of the interfacing WS_2 layer with respect to the rest of the WS_2 film (Fig. 3). Our findings illustrate that the electronic properties of transition metal dichalcogenides, and more generally, 2D layered materials, are physically altered by the interactions with the interfacing materials, in addition to the electron screening and defects that have been widely considered. For more details, see [15].

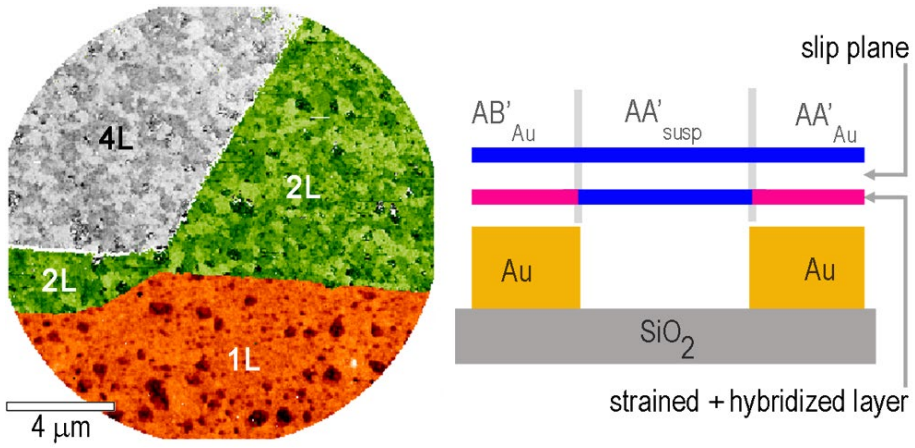

Fig. 3—(Left) Colorized photoelectron emission image showing different film thicknesses (1-layer, 2-layer, 4-layer). The slight color variations within each layer are due to structural changes in the semiconductor layers on the porous Au surface. (Right) Schematic illustrating the slip plane between the Au-interface layer and the second top layer.

3.4 Engineering of Nanoscale Heterogeneous Transition Metal Dichalcogenide-Au Interfaces

Engineering the transition metal dichalcogenide (TMD)-metal interface is critical for the development of two-dimensional semiconductor devices. By directly probing the electronic structures of WS_2-Au and WSe_2-Au interfaces with high spatial resolution, we delineate nanoscale heterogeneities in the composite systems that give rise to local Schottky barrier height modulations (Fig. 4). Photoelectron spectroscopy reveals large variations (>100 meV) in TMD work function and binding energies for the occupied electronic states. Characterization of the composite systems with electron backscatter diffraction and scanning tunneling microscopy leads us to attribute these heterogeneities to differing crystallite orientations in the Au contact, suggesting an inherent role of the metal microstructure in contact formation. We then leverage our understanding to develop straightforward Au processing techniques to form TMD–Au interfaces with reduced heterogeneity. Our findings illustrate the sensitivity of TMDs' electronic properties to metal contact microstructure and the viability of tuning the interface through contact engineering. For more details, see [17].

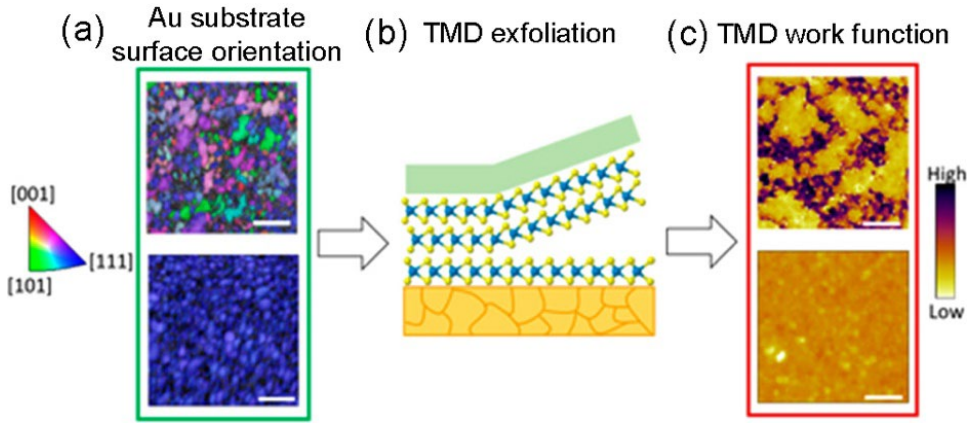

Fig. 4—(a) Colorized diffraction map showing the in-plane crystallographic orientation of an as-deposited Au film (top) and film treated with O_2 plasma (bottom). (b) Schematic showing the exfoliation of TMDs onto a polycrystalline Au surface. (c) Work function map showing large variations across the TMD semiconductor on as-deposited Au (top) versus an Au surface first treated with O_2 plasma (bottom).

3.5 Thermally Induced Reactions of Monolayer WS2 with Au-Ti Substrates

The thermal stability of WS_2 flakes is studied after integration into a device structure with a Si/SiO_2 substrate, a Ti adhesion layer, and an Au-contact layer. WS_2/Au/Ti is commonly used in catalytic and electronic devices, and its thermal stability is important for extended operation and mitigation of breakdown at local hot spots during operation. The thermal stability is severely impacted by rapid diffusion of Ti into and through the polycrystalline Au (Fig. 5). Thin-film samples of Au/Ti, with and without an exfoliated WS_2 monolayer, were annealed sequentially from 275 to 625°C in vacuum. The near-surface chemical information was extracted with X-ray photoelectron spectroscopy for each annealing step. Ti from the sticking layer, which is buried under 25 nm of Au, diffuses into the near-surface region and initiates a wide range of reactions forming Ti oxide, carbide, sulfide, and a Ti-Au alloy, mainly Au_2Ti. WS_2 experiences S loss for T > 350°C and forms Ti-sulfide for T > 600°C. Higher thermal loads spanning the same temperature range lead to coalescence within the nanocrystalline Au layer, limiting grain-boundary diffusion of Ti. The results provide insight on the stability of the WS_2/Au/Ti material system, which impacts its performance in catalytic and electronic applications. For more details, see [11].

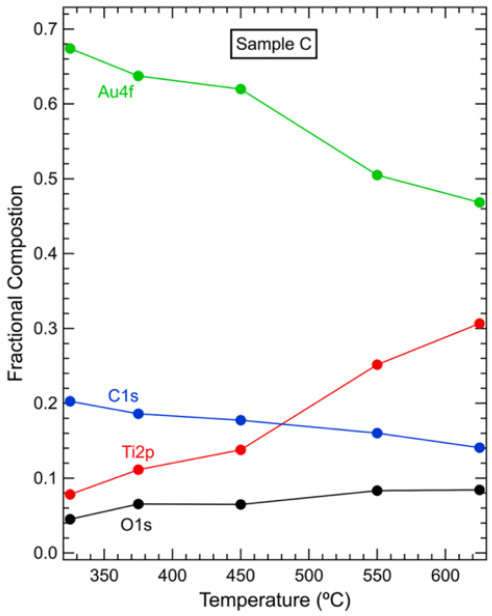

Fig. 5—Plot tracking the chemical changes of a TMD semiconductor on a Ti/Au film on SiO_2/Si substrate. As the sample temperature is increased, the Ti sticking layer beings to diffuse into and through the Au film to react with the TMD semiconductor on the surface.

3.6 Metallicity of 2H-MoS_2 Induced by Au Hybridization

Recent studies have shown that MoS_2 can undergo a structural phase transition from the 2H to 1T′ phase on Au substrates at moderate temperatures. Here, we use ultraviolet and X-ray photoelectron spectroscopy, Raman and photoluminescence spectroscopy, and scanning tunneling microscopy/spectroscopy to probe the impact of annealing exfoliated, monolayer MoS_2 on Au. Our results across multiple length scales indicate that 2H-MoS_2 becomes hybridized with Au upon thermal annealing without inducing the 1T′ structural phase and the bandgap can be modulated to zero width depending on the degree of hybridization (Fig. 6). These results can be used to control the resistance of metal-MoS2 contacts at the atomic scale without introduction of defects or structural phase. For more details, see [4].

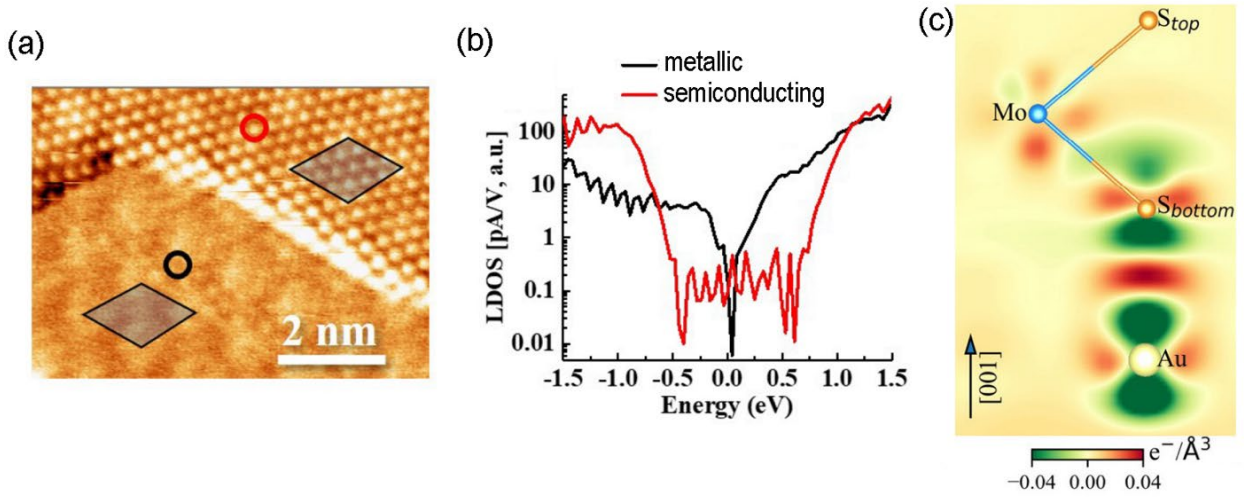

Fig. 6—(a) Scanning tunneling microscopy (STM) image showing the boundary between a "metallic-like" and a "semiconducting-like" region on an MoS_2 monolayer on Au. (b) Tunneling spectroscopy showing the metallic and semiconducting response from the color circles in (a). (c) Electron redistribution plotted along the vertical plane passing through the Au, S, and Mo atoms where the S-Au interaction is the strongest.

4 CONCLUSIONS

Based on the above data, we have quantified and assessed characteristics of the interface between 2D semiconductors and metals, the transitions for semiconductor-to-metal phase changes, and the formation and engineering of light-emitting processes in 2D semiconductors. The understanding of 2D semiconductor device performance and the materials from which they are constructed is informing on the design and construction of more capable electronic testing devices. The added understanding for how the 2D semiconductor/metal interface behaves is helping to strategize future devices that can be implemented by the Navy and add technical value toward next-generation devices and sensors.

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