Structural Analysis of Polymorphic Graphene and other Novel Layered Materials by STM and LEEM/LEED-I(V)

Maxwell Grady[1], Zhongwei Dai[1], Steven Arias[1], Karsten Pohl[1], Taisuke Ohta[2], Bogdan Diaconescu[2]

1. Department of Physics and Material Science, University of New Hampshire Durham, NH
2. Center for Integrated Nanotechnology, Sandia National Laboratory Albuquerque, NM

Abstract

Graphene has aroused tremendous interest as a novel material in the field of organic electronics and organic photovoltaics due to its numerous unique structural and electronic properties. Graphene's optical properties combined with its intrinsic electric conductance make it an ideal candidate for organic photovoltaic devices. Polymorphic graphene also provides a unique structure for the exploration of the processes of molecular self-assembly and the growth of ordered arrays of nano-particles. STM can adequately characterize the structure of the surface layer but provides little information about subsurface reconstructions. Furthermore, layer thickness measurements with STM are difficult, thus, LEEM and LEED-I(V) experiments will be utilized to further characterize the structure and thickness polymorphic graphene and other novel layered materials.

Background

2D-Layered materials such as Graphene, Molybdenum Disulfide, and Black Phosphorus provide exciting avenues for design of novel electronic devices such as organic photovoltaics, OLEDs, and transistors based on layered heterostructures. Understanding the surface structure of these materials at the atomic scale provides information about the interface between materials which is crucial to understanding device performance. Scanning Tunneling Microscopy and Low Energy Electron Microscopy are ideally suited tools for surface structure analysis of 2D materials. STM provides atomic scale resolution images of the top most surface layer topography. LEEM provides an avenue for discerning layer thickness while also providing a method for fast switching between high resolution real space and reciprocal space imaging. LEED-I(V) can also contribute data concerning the precise atomic positions in the near surface area of the sample as a result of low mean free path for low energy electrons.

University of New Hampshire

Materials

The following materials were required to complete the research:

- Homebuilt UHV VT-STM @ UNH
- Elmitec LEEM @ CINT
- Custom PVD graphene growth system
- Ru(0001) single crystal substrate
- Custom software package for analysis of LEEM/LEED data

Methods

The materials were prepared according to the steps outlined below:

1. Ru crystal preparation via annealing in O_2 atmosphere
2. High temperature PVD graphene growth in H_2 atmosphere
3. Preliminary characterization via STM and Conventional LEED **@ UNH**
4. Further analysis using LEEM-I(V) and LEED-I(V) **@ CINT**

STM Results - Polymorphic Graphene

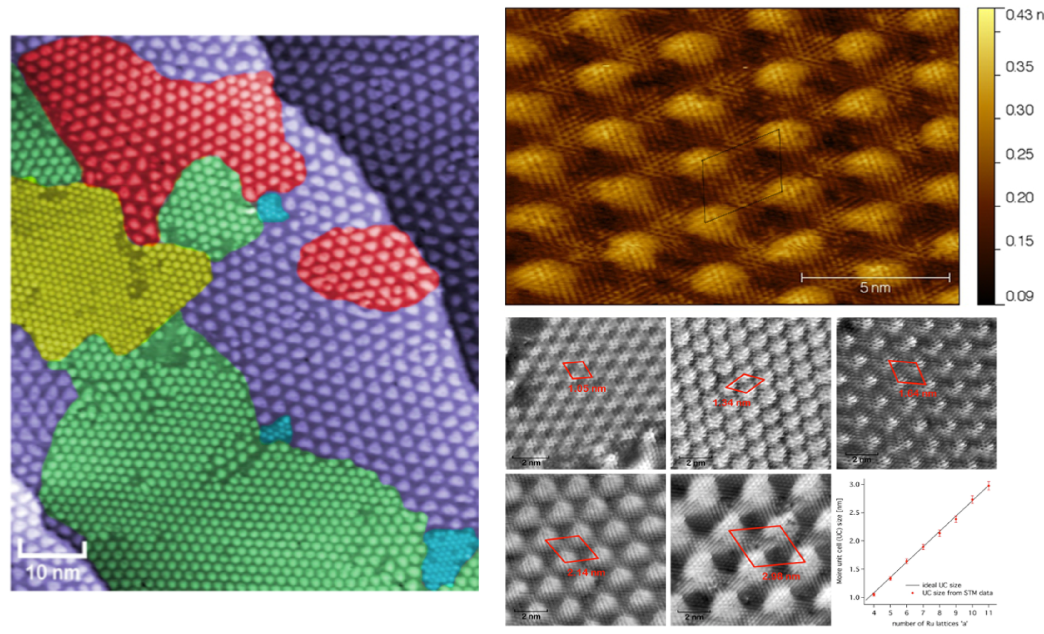

Figure 2: Right: High resolution STM image showing multiple distinct moire domains in the polymorphic graphene surface. Left: Atomically resolved STM images display ability to accurately measure the superstructure size with ultra high lateral resolution.

LEEM/LEED Results - Polymorphic Graphene

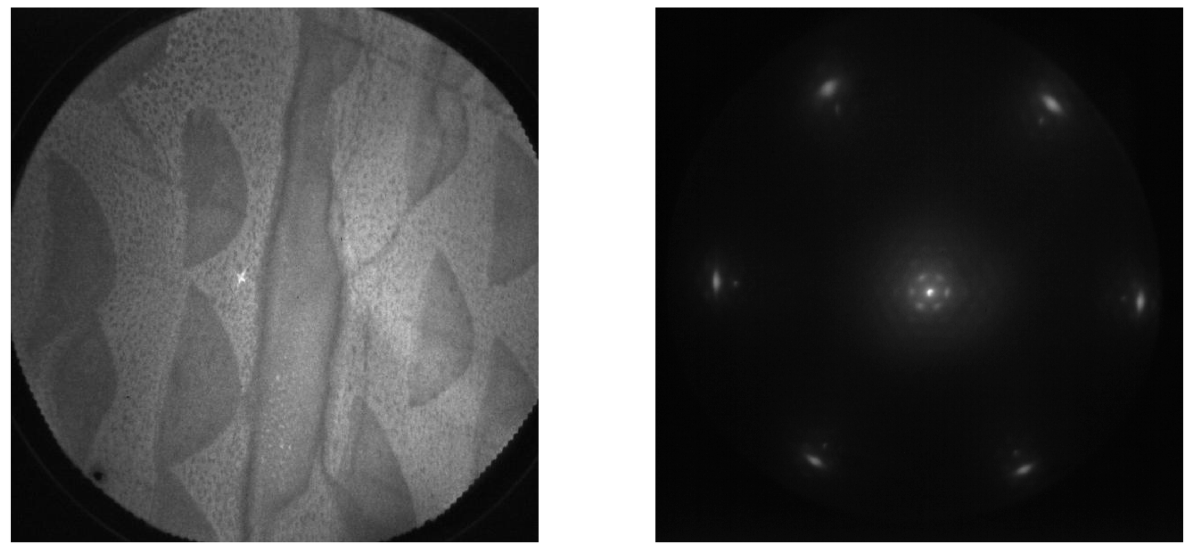

Figure 1: Preliminary LEEM and micro-LEED I(V) results demonstrate viability of growing samples at UNH followed by *ex-situ* characterization by LEEM. (right) 50-μm FOV real space LEEM image showing characteristic lens shaped graphene islands multiple microns in size. (left) micro-LEED pattern collected from sub 5 micron electron beam spot atop graphene island.

Analyzing the surface structure with atomic resolution in 3D requires detailed analysis of LEEM/LEED-I(V) curves combined with computational modeling. LEEM-I(V) curves taken from graphene sheets in the ultra-low energy regime display characteristic intensity oscillations which should correlate with layer thickness, a well known result from thin film interference.

LEEM/LEED-I(V) Results

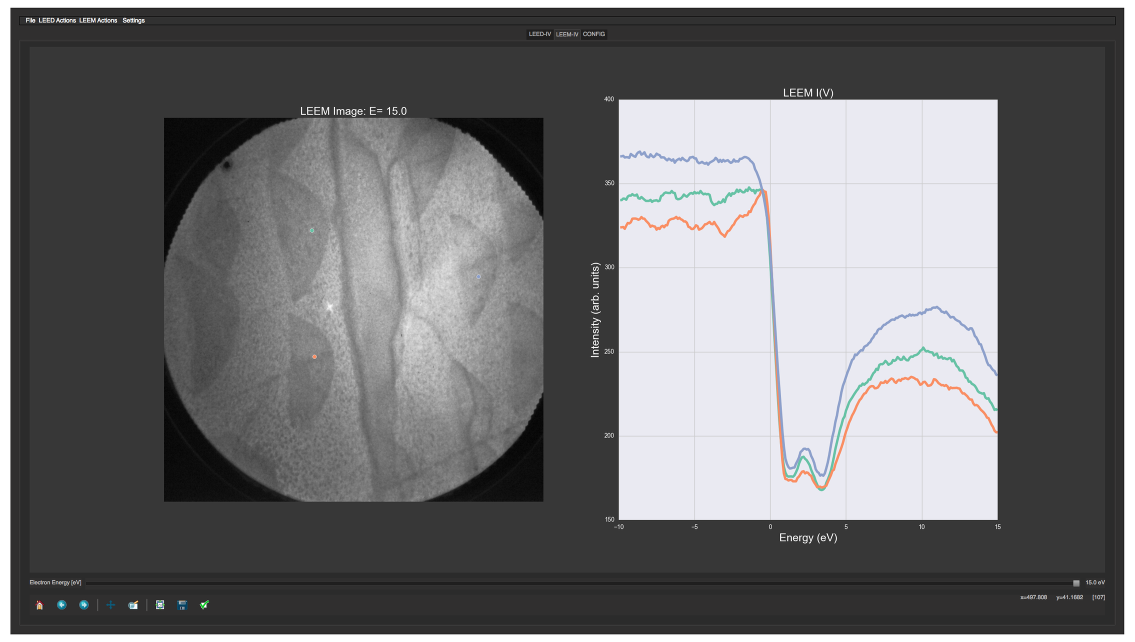

Figure 3: LEEM-I(V) curves extracted from real-space LEEM data collected @ CINT using the custom built PLEASE software package. Under development is a method for automating the procedure to count the number of oscillations in the region near 0eV to discern the layer thickness.

Other 2D Material Results

LEEM and LEED data collected from the polymorphic graphene study have been analyzed using a custom built software package designed for aiding analysis and visualization of LEEM/LEED-I(V) data sets. Further work is underway to use the same software to analyze data from a wide variety of novel materials.

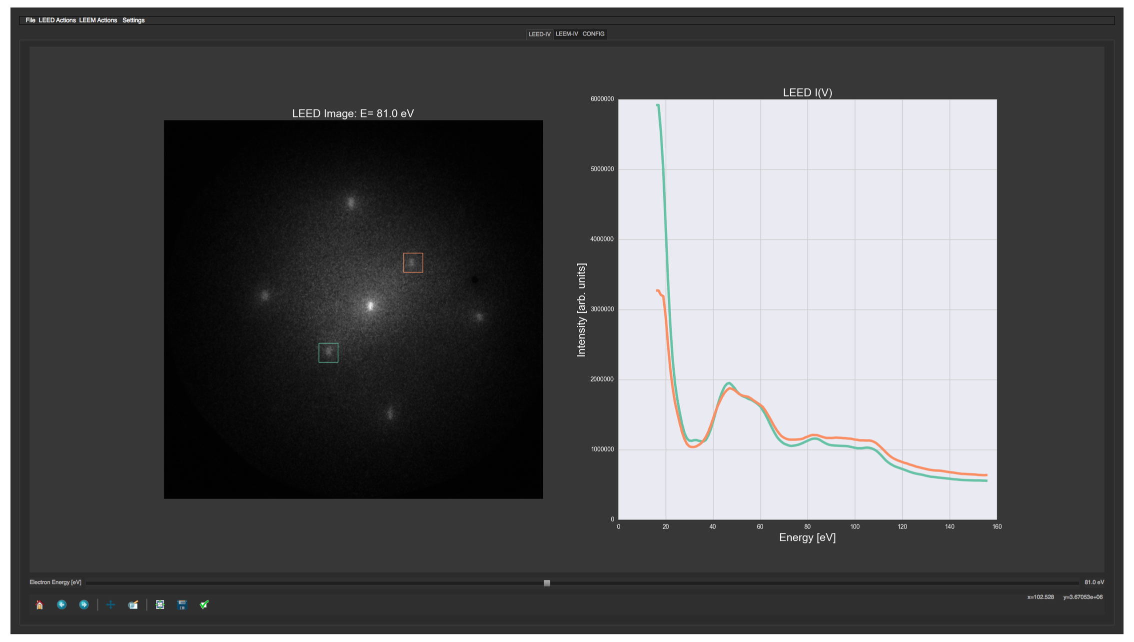

Figure 4: LEED-I(V) data extracted from bulk black phosphorus sample. Future experiments will examine changes in surface structure from bulk to few and monolayer samples as well as examine the possibility to measuring sample thickness with LEEM-I(V).

Conclusion and Future Work

We have shown the ability to grow a graphene system with novel surface structure characteristics. Work will be continued to analyze the structure in more detail using LEEM and LEED-I(V). Additional work is underway concerning analysis of structural properties of many novel materials such as Molybdenum Disulfide and Black Phosphorus. Finally, each new data set analyzed aids in the development, refinement, and optimization of the PLEASE software package.

Acknowledgements

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