XPS Depth Profiling of Epitaxial Graphene Intercalated with FeCl$_3$

Mahdi Ibrahim  
Maynard H. Jackson High School  
Atlanta, GA.

Faculty Advisor: Dr. Kristin Shepperd

Research Group: Prof. Edward Conrad  
School of Physics  
Georgia Institute of Technology

STEPUP 2011
Epitaxial Graphene Intercalated with $\text{FeCl}_3$

Epitaxial graphene is a hexagonal array of carbon atoms extending over two dimensions.

It is the same material that graphite is made up of, except that graphite is multi layered with different orientation of the stacking.

Graphene has several desirable physical and electronic properties that make it attractive provided the issue of conduction of electrons is taken care of.
Graphene

http://bigthink.com/ideas/24381

http://cnx.org/content/m29187/latest/graphene.jpg

Image from P. First, GT
Production of Graphene

• Exfoliation (scotch tape method)

Chemical Vapor Deposition (CVD)

Epitaxial growth
Why Epitaxial Graphene

- **Epitaxial Versus Exfoliated**

- **Exfoliated graphene:**
  - layers of the material removed with tape from a block of graphite
  - It is not suitable for industrial production
  - It is more of flakes that are not continuous and uniform over the surface/sheet

- **Epitaxial graphene is preferred by electronics company because**
  - It can be produced as a large wafer with large range uniformity
  - existing industrial technology can be used easily
Epitaxial Graphene

Blank silicon carbide (SiC)

Si atoms sublimate. Leaving C-rich surface

Graphene bonds form at 1500°C
Physical and Electronic Properties of Graphene

- Ballistic transport of electrons
- High mobility, high thermal conductivity, high frequency
- Optically transparent.
- A possible material for transparent conducting electrode.
- Sheet resistance is high
- **Dope graphene to increase carrier density and lower sheet resistance**
Band gap and Band Energy

- Band gap for metals (conductors), semiconductors, and insulators
  - Metal
  - Semiconductor
  - Insulator

- Band gap for grapheme (cone on the right)
Graphene Doping Types

a) n type (electron donor)  b) un-doped graphene  c) p type (electron acceptor)
Doping of Graphene

- Vapor transport (2 zone furnace)
- Iron (III) chloride, anhydrous
- Staging controlled by varying temperature of intercalate

**FeCl\textsubscript{3}-MEG stable in ambient conditions.** FeCl\textsubscript{3} is an electron acceptor; results in **hole doping (p-type doping)** of graphene
Staging of FeCl$_3$-MEG

MEG

Stage 1

Stage 2

Stage 3
Theory (XPS Instrument)

Photo-Emitted Electrons (< 1.5 kV) escape only from the very top surface (70 - 110Å) of the sample.

Focused Beam of X-rays (1.5 kV)

SiO<sub>2</sub> / Si<sup>+</sup> Sample

Samples are usually solid because XPS requires ultra-high vacuum (<10<sup>-6</sup> torr)

Electron Collection Lens

Electron Take-Off-Angle

Electron Energy Analyzer (0-1.5 kV) (measures kinetic energy of electrons)

Electron Detector (counts the electrons)

Si (2p) XPS signals from a Silicon Wafer
HOW Does XPS Work

- A mono-energetic x-ray beam of known wavelength and energy (hv) hits the surface.
- The x-ray photons knocks of electrons from the sample surface.
- Cylindrical Mirror Analyzer (CMA) measures the KE of emitted e-s.
- The spectrum plotted by the computer from the analyzer signal.
- The binding energies can be determined from the peak positions and the elements present in the sample can be identified.

\[
E_{\text{binding}} = E_{\text{kinetic}} - hv - \phi
\]

where \( E_{\text{Binding}} \) = Binding Energy
\( E_{\text{Photon}} \) = energy of the X-ray photon used
\( \phi \) = work function of the spectrometer

- Depth profiling with Ar+ at 1000 eV energy
XPS is used to measure:

- elemental composition of the surface (top 1–10 nm usually) of pure substance
- chemical or electronic state of each element in the surface
- uniformity of elemental composition across the top surface (or line profiling or mapping)
- uniformity of elemental composition as a function of ion beam etching (or depth profiling)
Method

- We analyze the FeCl$_3$-EG, stage 1, 10 layers samples with XPS
- Chemical make-up as a function of depth, is the sample uniform at the EG-SiC interface
- Is the FeCl$_3$ present at the SiC interface
Results and Discussions

FeCl₃ Stage 1 C₁s Depth profile

Intensity

284.6 eV

283.3 eV

Binding Energy (BE)
C1s, Cl2p and Fe2p XPS spectra

- C 1s BE at 284.6 eV; fit to single Lorentzian; **no direct chemical bonding to FeCl₃**
- Cl 2p binding energy at 199.5 eV and Fe 2p binding energy at 710.4 eV; consistent with FeCl₃ not fragments of iron chloride
Cl 2p XPS at SiC interface

- Small amount of Cl at the SiC interface
- Inconclusive if Cl is present at SiC interface from intercalation directly or due to sputtering and settling of Cl back into etch pit.
Results ...

- XPS results show FeCl₃ is weakly interacting with MEG; NO DIRECT CHEMICAL BONDING
- C 1s
  - BE = 284.6 eV, and also fit with single function –
- Fe 2p and Cl 2p
  - BE consistent with FeCl₃ (not FeCl₂, etc.)
    - FeCl₃ stays in tact after intercalation
- C 1s as a function of etch depth
  - Shift in BE as you approach the SiC interface
    - (283.2eV – SiC)
- SiC interface, Cl 2p present
- Inconclusive if Cl is present at SiC interface from intercalation directly or due to sputtering and settling of Cl back into etch pit.
Conclusion

Future work:
Do XPS spatial map to determine FeCl$_3$ uniformity.
Lesson Plan

☐ Resistivity of Graphene
☐ Factors affecting resistivity
☐ Relationship between resistivity and resistance
☐ How the doping of graphene by FeCl$_3$ changes the resistivity of graphene
☐ Conductivity in relations to the band gap between valence band and conductions bands of material
What I take to my home school

- Research methods and Ideas
- Research equipment
- Application of pure science
- Great working environment (hard work, cooperation, ...)
Acknowledgment and Thanks

- Dr. Leyla Conrad - STEPUP Director
- Dr. Kristin Shepperd – Faculty Advisor
- Prof. Edward Conrad – Research Group Leader
- All members of Prof. E. Conrad’s research team