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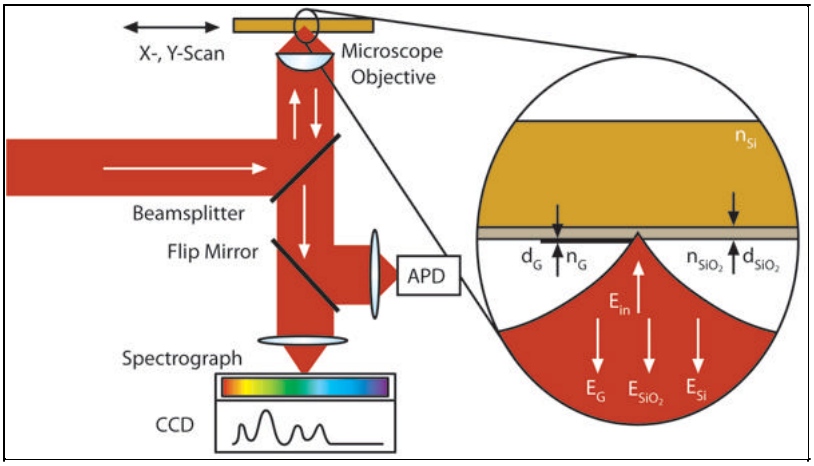
Rapidly Counting Graphene Layers, One by One

by Hank Hogan

Carbon someday could rival silicon in forming the basis for miniature electronics. Two-dimensional sheets of carbon called graphene are promising candidates because they are stable and they move electrons rapidly. Furthermore, graphene-based devices can be scaled down to nanometers.

Graphene typically is produced by cleaving graphite, which results in a few monolayer flakes among a welter of much thicker samples. From such piles, investigators would like to rapidly find the single-layer flakes, which are the best for research. However, this task has been difficult to accomplish. Now a team has discovered why standard Rayleigh imaging, which is based on simple light scattering, could do the trick.



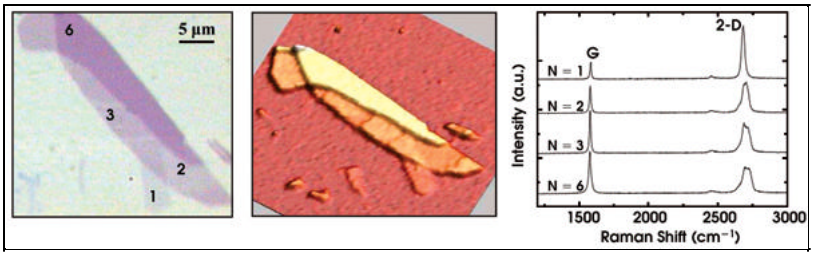
In this setup, an incoming beam focuses on a sample of graphene. The back-scattered signal from the sample comprises Rayleigh scattering, which is detected by an avalanche photodiode (APD), and Raman scattering, which is detected by a spectrometer and a CCD. The inset shows the optical field interacting with graphene on silicon covered by silicon dioxide. Images reprinted with permission of the American Chemical Society.

"The main goal is a quick technique to identify and count graphene layers. This is badly needed in the field," said Andrea C. Ferrari, a lecturer at Cambridge University in the UK and a member of the research team. Others involved in the research were from Ludwig Maximilians University in Munich, from the University of Ioannina in Greece and from the University of Manchester, also in the UK.

The researchers combined Rayleigh imaging — used to count graphene layers — with Raman scattering — used to provide structural information. With the two techniques, they demonstrated quick and easy sample measurement and characterization.

In a homebuilt setup, they used an inverted microscope with either a HeNe laser at 633 nm or a collimated white-light beam as a light source. They generated coherent white-light pulses by pumping a photonic crystal fiber with the output of a Ti:sapphire laser operating at 760 nm.

They focused the excitation light on a sample, which consisted of graphene flakes on a silicon wafer coated with SiO_2. They sent the backscattered light into an avalanche photodiode for Rayleigh measurements. Alternatively, they flipped a mirror and sent the light through a notch filter and into a spectrometer for Raman measurements, using a system from Renishaw plc of Wotton-under-Edge, UK.



In these images, one to six layers of graphene are visible. The left image is an optical micrograph, while in the center is a contrast-reversed 3-D confocal map of Rayleigh scattering from the sample. On the right are Raman spectra as a function of layer number.

The spatial resolution was 800 nm, with the Rayleigh images taking a few milliseconds of acquisition time per pixel. The Raman imaging, in contrast, required a few minutes.

The researchers compared the Rayleigh and Raman results with those obtained with an atomic force microscope from Veeco Instruments Inc. of Woodbury, N.Y. They found that the Raman spectra correlated with the number of layers, and they were able to distinguish between parts of a sample composed of one, two, three and six layers.

They also found the same to be true for Rayleigh imaging done with a 633-nm source. The contrast increased linearly with the thickness, an expected result.

The group knew before beginning research that thin layers of graphene are visible to the unaided eye when on an SiO_2-coated silicon wafer. For example, a single layer of graphene can be seen when it is on a 300-nm-thick layer of SiO_2. The challenge was to understand why this happened and to generalize it to any substrate.

In analyzing the situation, the researchers looked at the multiple reflections and at the presence of SiO_2, concluding that the graphene samples can be considered as the superimposition of single sheets, with the electrons in each layer acting independent of the others. The result is a change in contrast that tracks the number of sheets, up to six layers thick.

Consequently, Rayleigh scattering can determine graphene thickness quickly, and Raman imaging can be used for characterization. The group now plans to put the measurement tools to use. “The ultimate goal is to advance graphene research to become a viable industrial technology,” Ferrari said.

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