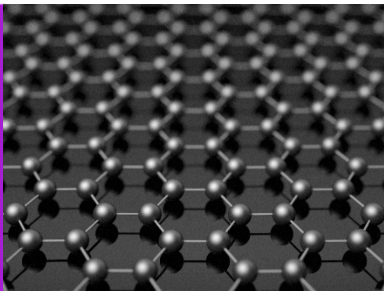
Large Area Characterization of Graphene using Spectroscopic Reflectometry

Filmetrics® F-Series Thin Film Systems



Introduction

Graphene, a monolayer of graphite, is of great interest due to its wide range of applications in fields such as healthcare, semiconductors, and aerospace. Its unique properties are leading to new areas of research examining other atomically thin materials, collectively referred to as "2D Materials" [1-6].

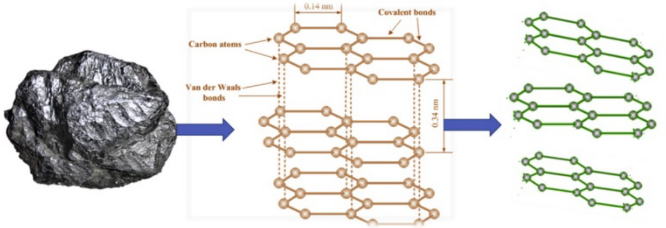


Figure 1. Image of graphite (left); graphite structure (center), and graphene structure (right)[7].

One of the major limitations of graphene transitioning from academic research to industrial applications is the difficulty in characterization. Due to being extremely thin, one monolayer is only 3.35Å, and requires methods such as atomic force microscopy (AFM), scanning transmission electron microscopy (STEM) or Raman spectroscopy to properly characterize. While these methods can be very effective, they are not without their own limitations. Error in AFM thickness measurement can be exacerbated by sample moisture absorption, and both AFM and STEM cannot characterize the large areas that would be required for high volume production. Raman spectroscopy is much faster than either AFM or STEM, but measurement time remains significantly longer than is feasible for high volume manufacturing. Further, the method for determining graphene thickness via Raman spectroscopy involves comparing the spectral peak intensity ratios, which can be difficult, as the change in ratios is very small after 5 layers of graphene and can be affected by multiple additional factors, including the quality of the graphene sample. Finally, all three of these methods can be prohibitively expensive.

Spectral Reflectance of Graphene

A low-cost method for large area analysis that shows great promise is spectral reflectance, which has been demonstrated to accurately characterize monolayer and multi-layer graphene stacks on a microscopic scale[8]. Graphene has typically been characterized by examining its optical contrast with the underlying substrate. However, this process requires that (a) the optical constants of the system are known, and (b) the underlying substrate is of the appropriate thickness to obtain sufficient contrast.

Measurement Setup

In this example, spectral reflectance was used to both examine the optical contrast as well as the spectral shift that results from the 2D material being placed on the substrate. Further, with the Filmetrics suite of thin film tools, the optical constants of graphene were measured by placing the graphene onto a transparent (fused silica) substrate and measuring it using the Filmetrics F10-RT-UVX system. The collected reflection and transmission spectra, shown in Figure 2, were then used to determine the optical constants of the material, shown in Figure 3, allowing for thickness characterization across arbitrary substrates.

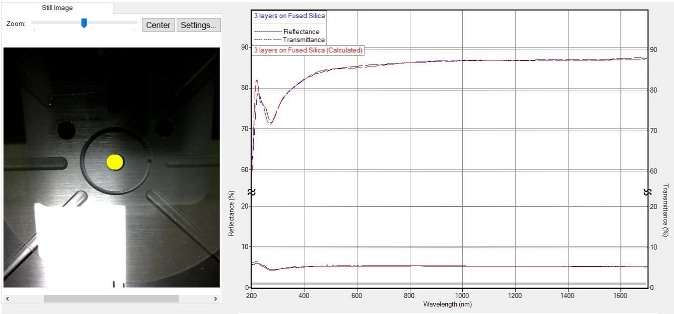


Figure 2. Transmission (upper trace) and reflection (lower trace) data for tri-layer graphene on a fused silica substrate. The layer thickness measured 1.2nm.

Using this data, monolayer and bilayer graphene on SiO_2 on Si wafers were measured. As can been seen from the spectral comparison in Figure 4, there is a significant wavelength and peak intensity shift, depending on the number of layers of graphene. For 300nm of SiO_2, a large enough shift can be seen that graphene on these wafers can be observed directly. The advantage of spectral reflectance is that it can be used as a characterization technique on any arbitrary substrate. In addition, it can be used to quickly characterize graphene over large areas, as shown by the wafer maps in Figure 5, generated by the F50 thin film mapping tool.

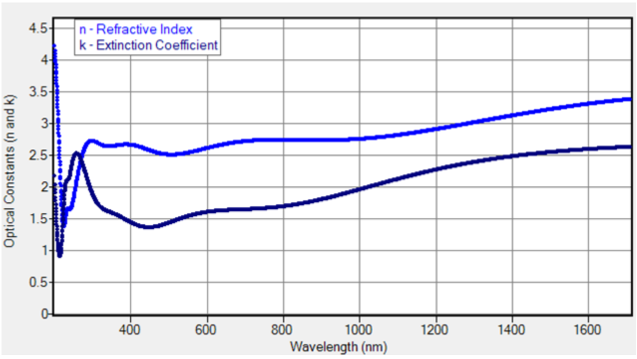


Figure 3. The refractive index (n) and extinction coefficient (k) of graphene were modelled using the reflection and transmission data from graphene on fused silica.

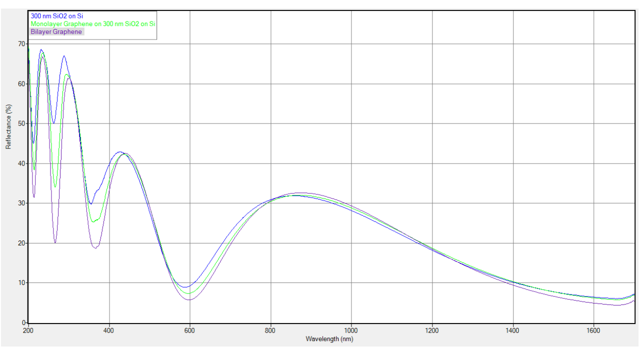


Figure 4. Spectral reflectance spectral comparison of a 300nm SiO_2 wafer (blue), a monolayer of graphene on 300nm SiO_2 (green), and bilayer graphene on 300nm SiO_2 (purple).

Conclusions

The Filmetrics F10-RT measurement technique can be used for both the determination of optical constants and large area characterization of 2D materials. Future work will look at examining additional 2D materials as well as different orientations of multilayer stacks of graphene.

About the Filmetrics F10-RT-UVX

The Filmetrics F10-RT Thin-Film Analyzer requires just a mouse-click to simultaneously capture both reflectance and transmittance spectra by eliminating the need for hardware configuration changes between measurements. The F10's true normal incidence design is critical for accurate characterization of display films, and the video camera with image capture makes measurement location easy and recordable. The industry-standard FILMeasure software brings the power of Filmetrics analysis to provide accurate measurement for many different types of applications.

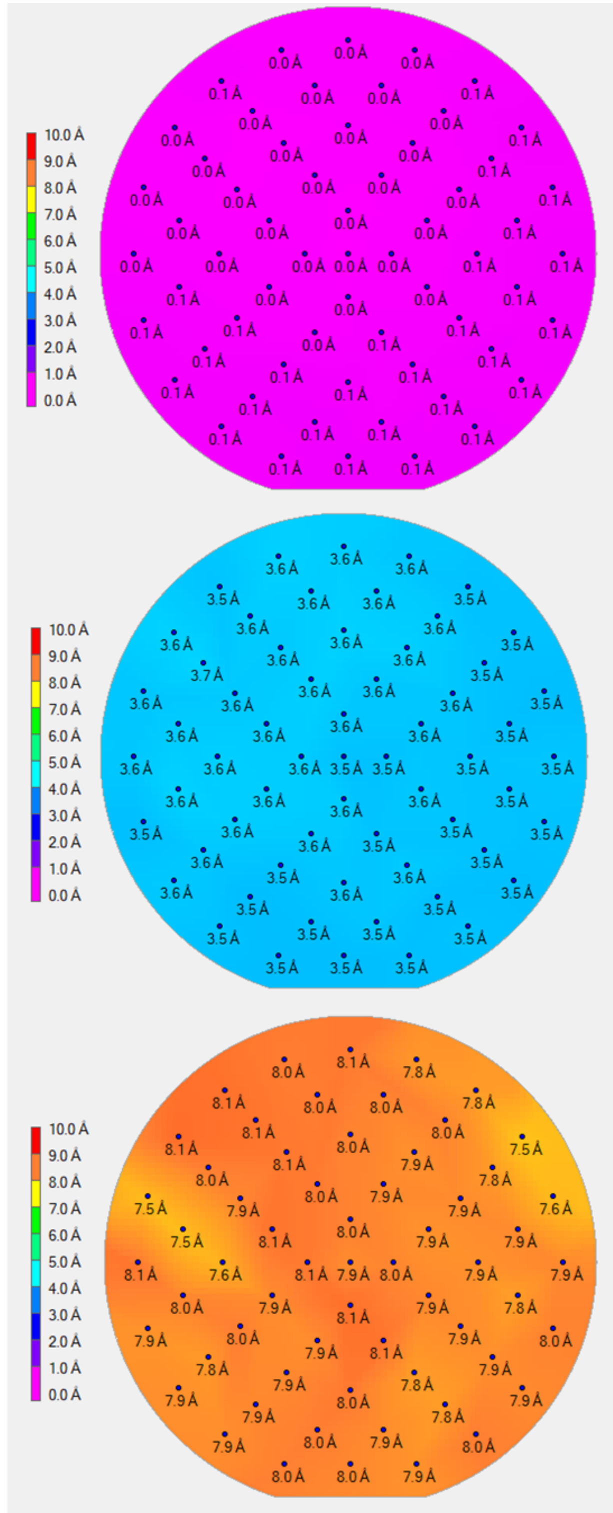


Figure 5. Wafer maps of graphene thickness of 100mm Si wafers with only 300nm SiO_2, (left), a monolayer of graphene (center), and a bilayer of graphene (right), As can clearly be seen, there is a distinct difference in thickness measurement between each wafer, with largely uniform thickness values across each wafer.

KLA INSTRUMENTS

About the Filmetrics F50-UVX

The Filmetrics F50 is a thin film mapping tool that can be used to measure samples up to 450mm in diameter. The motorized R-theta stage moves automatically to the measurement point and can provide thickness measurements as fast as two points per second. The F50 comes with predefined map patterns and has the option for users to generate their own pattern, allowing users to set up and run measurements in minutes.

Please contact us to schedule thickness measurement on your thin film samples, or 3D surface topography using the Filmetrics Profilm3D® optical profiler, which quickly and easily generates surface measurements of roughness, step height, or other features of interest.

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