

UV-Vis Reflectance and Raman micro-spectroscopy of single and multiple layers graphene

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Introduction

Graphene possesses extraordinary electric and optical properties, which have attracted enormous interests in electronic and photonic devices fabrication [1]. However, the production of defect-free single-layer graphene still remains a challenge. Very often, defects and more-than-monolayer of graphene are produced, which significantly affect the performance of these devices. Hence, understanding the optical properties of the graphene layer(s) is critical in verifying the minute differences in graphene' structures and morphology. In particular, a quick characterization tool that is able to differentiate single and few-layer graphene is crucial in optimizing and/or improving graphene-based devices' performance.

Raman spectroscopy and reflectance spectroscopy are two widely adopted methods to determine the quality of the graphene layers. In this application note, we combine UV-Vis reflectance and Raman micro-spectroscopy to understand the characteristic of single and multiple layers of graphene within a single microscopic region of interest.

Method and set up

The entire integrated system consists of a uSight-2000 and uSight-X module mounted onto a Nikon Ci-S microscope equipped with a motorized stage, reflected and transmitted halogen light source. The uSight-2000 offers UV-Vis-NIR measurement functionalities whereas the uSight-X offers laser-induced spectroscopy functionalities.



Figure 1. uSight-2000 and uSight-X module mounted onto a Nikon Ci-S microscope

The uSight-2000 comes with a precise LED indicator that helps to pinpoint the measurement spot. Other than the LED location indicator, the operating software also comes with a soft indicator. The uSight-2000 in-built spectrometer covers a spectral range from 200 nm to 1100 nm at 1.5 nm resolution. The uSight-X is configured with in-built 532 nm frequency stabilized laser. Signal collected with the uSight-X is guided to an external spectrometer. Such configuration offers flexibility in the choice of spectrometers.

Raman and reflectance measurements were with 100 x 0.9 N.A. objective lens (Figure 1). The optical collection path of both uSight systems are aligned to collect from the same region of interest. Integration time for the Raman measurement is 10 s per spectrum and laser power attenuated below the damage threshold for the graphene (< 4 mW). A monolayer of graphene (6 x 6 mm²) was transferred from Cu foil to a 300-nm SiO₂ , substrate according to the procedure documented in previous App note[2].

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Results and discussion

The optical property of graphene is very sensitive to the number of graphene layers. In particular, the Raman spectroscopy is a hugely popular tool to determine the quality of graphene. For instance, the Raman's G'/G band peak ratio decreases with increase in graphene layer due to the hindering of G' band's triple resonance. On the other hand, the change in number of layer also affects the reflectance (as well as absorption).

The optical image of the transferred single-layer graphene on SiO_2 shows different reflective colors in certain regions (Figure 2A). Some areas appear darker due to the folding of monolayer graphene to few-layer graphene during the transfer process. We identify four different regions of interest, i.e. (i), (ii), (iii) and (iv), where the reflectance and Raman measurements were performed.

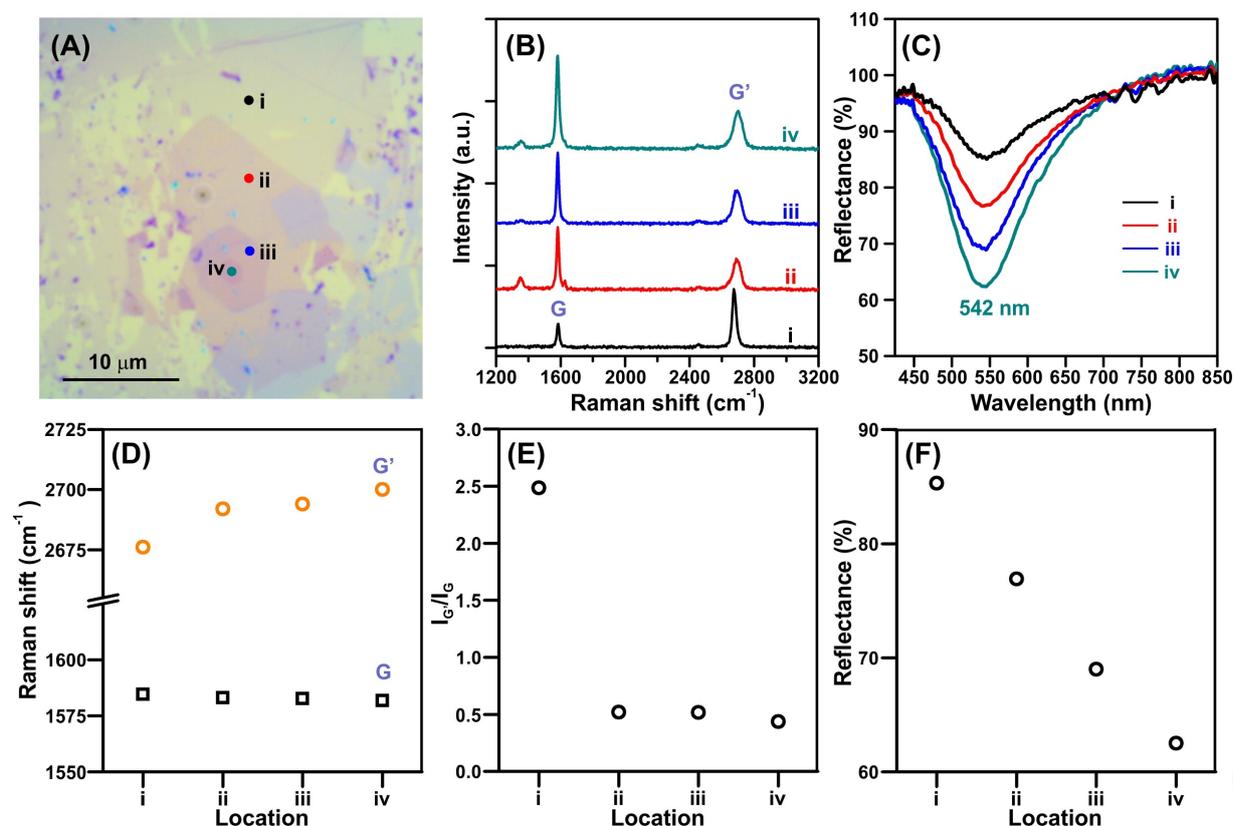


Figure 2. (A) Optical image of single layer graphene transfer to 300nm SiO₂ where regions of various defects are visible. (B-C) Raman and Reflectance measured from the various locations indicated in (A). (D) Raman shift, (E) peak intensity ratio ($I_{G'}/I_G$) of 2D and G-band and (F) the Reflectance obtained from the locations (i – iv) in A.

The Raman spectra demonstrate distinct spectral differences in these four regions of interest (Figure 2B). In particular, the peak position of G' shifts more than 25 cm^{-1} to higher wavenumber from location (i) to (iv), while the G band remains relatively constant (Figure 2D). There is also a clear difference in G'/G band peak ratio. The peak ratio in region (i) is 2.5, whereas other regions are ~ 0.5 (Figure 2E). In addition, region (ii, iii, and iv) show a broadening of G' band's peak width as compared to region (i). Collectively, our results reveal that region (i) contains monolayer graphene (Figure 2B-i). Our finding is further supported by literature which indicates that a typical monolayer graphene spectrum exhibits a sharp and intense G' -band relative to the G -band^[3]. The peak width data of G' -band (Table 1) from region (ii – iv) suggests the number of graphene layers at these regions are at least 4 layers^[4].

Similarly, the reflectance measurements can clearly reveal the morphological changes in these graphene regions. The reflectance from location (i) to (iv) are evidently reduced across the visible wavelength (450 to 700 nm) (Figure 2C). A linear reduction in reflectance from 85% to 63% can be tabulated when measuring the intensity at 545 nm from region i to iv (Figure 2F), which can be attributed to an increase in absorption of visible light with increasing number of graphene layer.

Table 1 Summary of Raman and Reflectance measurement.

Region	Raman							Reflectance	
	G			G'			$I_{G'}/I_G$	Peak (nm)	Reflectance (%)
	Peak (cm^{-1})	Width (cm^{-1})	Intensity (cts)	Peak (cm^{-1})	Width (cm^{-1})	Intensity (cts)			
(i)	1584.5	16.8	1492	2676	29.2	3709	2.5	546.0	85.3
(ii)	1583.0	16.6	3898	2692	61.1	2027	0.5	543.6	76.9
(iii)	1582.6	16.6	4433	2694	65.5	2285	0.5	542.1	69.0
(iv)	1581.8	21.1	5886	2700	72.0	2564	0.4	542.1	62.5

The new uSight micro-spectroscopy series from Technospex. Pte. Ltd. provide the flexibility to combine UV-Vis-NIR micro-spectroscopy and laser spectroscopy in one platform that enable various optical measurements including reflectance, transmission, absorption, photoluminescence and Raman spectroscopy with mm spatial resolution.

Reference

- [1] Nature Photon 2010, 4, 611.
- [2] Application of uRaman on Graphene.
- [3] Phys Rep 2009, 473, 51.
- [4] Small 2010, 6, 195.

