

Determine lateral size and thickness of graphene using quantitative optical mapping

Li-Shang Lin¹, Wei Bin-Tay², Zabeada Aslam², A.V.K. Westwood² and R. Brydson²

¹ Research Center of Applied Science (RCAS), Academia Sinica, Taipei, Taiwan

² School of Chemical & Process Engineering, University of Leeds, Leeds LS29JT, UK

E-mail: asimo945@gmail.com

Abstract

Graphene, a two-dimensional honeycomb sp^2 carbon lattice has received enormous attention because of the potential for various applications such as the electrodes of photovoltaic devices and batteries, next generation flexible electronics and even antibacterial coatings [1]–[3]. Interest in the application of graphene is mainly due to its unique physical and chemical properties, flexibility, and tuneability of the properties in graphene-based materials [3], [4]. However, while promising applications of graphene are being discussed, the term ‘graphene’ is often misused, and the difficulties in large-scale production of true two-dimensional graphene have further limited its applications [5], [6]. A three-dimensional graphite needs to be delaminated completely to benefit from full potential of the two-dimensional graphene: only single-layer graphene (SLG) and bilayer graphene (BLG) have the unique zero-bandgap electronic configuration, for few-layer graphene (FLG, 3 to <10 layers), the conduction and valence bands start to overlap, thicker graphene structures thus should be considered as thin film graphite instead of graphene [7]–[9]. Methods such as top-down solution-processed exfoliation was developed to overcome the obstacles for large-scale graphene production, but these approaches do not yet produce completely delaminated and homogeneous graphene. To monitor and optimise the graphene production process, the development of a fast, standardised and reliable characterisation protocol for large-scale solution-processed graphene is therefore desirable [9], [10].

Among the many characteristics of graphene flakes, the nano-structural features including the lateral dimension and the thicknesses of graphene are the most important factors that affect the various properties of graphene. However, though many of the analytical techniques have continuously been improved, methods to obtain and quantify these graphene nano-structural features are still limited. This is owing to the difficulties of visualising the ultra-thin nano-flakes and the fact that many of the properties of graphene are still unknown to be used to identify the

material [11], [12]. Today, detail graphene characterisation relies on directly examination using modern high-resolution transmission electron microscopy (HR-TEM), which the delicate technique is limited by its output and challenging for quantitative data interpretation. Even though the atomic force microscopy (AFM) can alternatively obtain the topographical features of graphene flakes in a comparably faster manner, the thickness determination can be still ambiguous, due to the possible C-H contaminant and the existence of a buffered layer between the graphene and substrate [13], [14].

In this study, a characterisation method based on quantitative optical mapping was proposed to determine the distributions of lateral dimension and thickness of graphene features of graphene. For the accurate assessment, the topographical feature of graphene was initially characterised by the most precise method based on direct imaging from transmission electron microscopy (TEM), the results were being used as benchmarks. An alternative image mapping technique was developed via optical microscopy to obtain the lateral dimension and thickness distribution rapidly. A Fabry-Perot interferometer was used as a substrate to make graphene visible under reflected light microscopy and the distribution of graphene thickness and lateral flake sizes can be obtained by analysing the optical microscopic images. We have shown a very good correlation between the lateral dimension distributions obtained TEM and optical microscopy imaging with an error of only 1.8% and 0.4% for the mean value of the primary flake and aggregated flake sizes respectively.

In summary, the methodology demonstrated offered the possibility to examine the topographical properties of graphene rapidly while avoiding time-consuming method such as AFM and TEM. This is valuable for quantifying the topographical properties of graphene and can be used to optimise the graphene synthesis process.

Reference:

- [1] K. S. Novoselov *et al.*, “A roadmap for graphene,” *Nature*, vol. 490, no. 7419, pp. 192–200, Oct. 2012.
- [2] B. Pollard, “Growing Graphene via Chemical Vapor Deposition,” *Ph. D. Thesis*, pp. 1–47, 2011.
- [3] M. Hofmann, W.-Y. Y. Chiang, T. D Nguyn, Y.-P. P. Hsieh, T. D. Nguyễn, and Y.-P. P. Hsieh, “Controlling the properties of graphene produced by electrochemical exfoliation,” *Nanotechnology*, vol. 26, no. 33, p. 335607, Aug. 2015.
- [4] K. Parvez *et al.*, “Electrochemically exfoliated graphene as solution-processable, highly conductive electrodes for organic electronics,” *ACS Nano*, vol. 7, no. 4, pp. 3598–606, Apr. 2013.
- [5] L.-S. Lin, “Characterisation Protocol for Liquid- Phase-Synthesised Graphene,” 2018.
- [6] J. H. Warner *et al.*, *Graphene : fundamentals and emergent applications*, 1st ed., vol. 17. Elsevier, 2013.
- [7] K. S. Novoselov *et al.*, “Two-dimensional gas of massless Dirac fermions in graphene.,” *Nature*, vol. 438, no. 7065, pp. 197–200, Nov. 2005.

- [8] B. Partoens and F. M. Peeters, "From graphene to graphite: Electronic structure around the K point," *Phys. Rev. B - Condens. Matter Mater. Phys.*, vol. 74, no. 7, p. 075404, Aug. 2006.
- [9] P. Bøggild, "The war on fake graphene," *Nature*, vol. 562, no. 7728, pp. 502–503, Oct. 2018.
- [10] A. P. Kauling *et al.*, "The Worldwide Graphene Flake Production," *Adv. Mater.*, vol. 30, no. 44, p. 1803784, Nov. 2018.
- [11] M. Bruna and S. Borini, "Optical constants of graphene layers in the visible range," *Appl. Phys. Lett.*, vol. 94, no. 3, pp. 1–4, 2009.
- [12] K. F. Mak, M. Y. Sfeir, J. A. Misewich, and T. F. Heinz, "The evolution of electronic structure in few-layer graphene revealed by optical spectroscopy.," *Proc. Natl. Acad. Sci. U. S. A.*, vol. 107, no. 34, pp. 14999–5004, Aug. 2010.
- [13] J.-S. S. Kim *et al.*, "Between scylla and charybdis: Hydrophobic graphene-guided water diffusion on hydrophilic substrates," *Sci. Rep.*, vol. 3, no. 1, p. 2309, Dec. 2013.
- [14] C. J. Shearer, A. D. Slattery, A. J. Stapleton, J. G. Shapter, and C. T. Gibson, "Accurate thickness measurement of graphene," *Nanotechnology*, vol. 27, no. 12, p. 125704, Mar. 2016.