

Improving the accuracy and reproducibility of Raman spectroscopy for graphitic particles

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Raman spectroscopy has long been a 'go-to' technique for measuring carbon materials, as the resulting Raman spectra reveal many of the material properties, such as the level of disorder, strain, doping, particle lateral size and thickness. However, although this technique has been used for many decades, there are still glaring issues with reproducibility, as well as the quantification of the Raman peak metrics in relation to the material properties. As a non-destructive and rapid technique, these issues become a crucial barrier, when trying to use this technique for both academic research and as an industrial quality control tool.

Towards this effort, firstly we report the results of an international interlaboratory study (ILC) conducted under VAMAS [1]. By comparing reported results with the measurements of the same regions made by the lead participant, variations in the reported peak intensity ratios and peak fits could be explored for both instrumentation and data analysis. This study has shown that by adopting a relative intensity calibration and consistent peak fitting and data analysis methodologies, these large, and previously unquantified, variations can be significantly reduced, allowing more reproducible and comparable measurements of graphitic materials using Raman spectroscopy.

Secondly, we assess how Raman metrics measured for graphitic particles in powder form can be used to determine impurities and the limits of detection. This form of material is widely produced by industry, and Raman spectroscopy is a common technique to assess the quality of the products. While several metrics have been presented in the literature, these are frequently developed for ideal samples, where unwanted unexfoliated material has been removed. However the effectiveness of these metrics is not known when applied to industrially produced that may not have such ideal separation. We show that when unexfoliated material is deliberately added to samples of few-layer graphene, Raman spectroscopy has limited ability to identify the presence of this unwanted material. In contrast, we show that when a sufficiently large number of spectra are acquired, it is found that by processing and classifying individual spectra, rather than the averaged spectrum, a reasonable estimate of the fraction of unexfoliated material can be obtained. These results highlight the care that must be taken when interpreting results of Raman spectroscopy measurements.

References

[1] Turner, P; Paton, K. R.; Legge, E. J. *et al.* 2D Materials (2022) 9, 035010

Figures

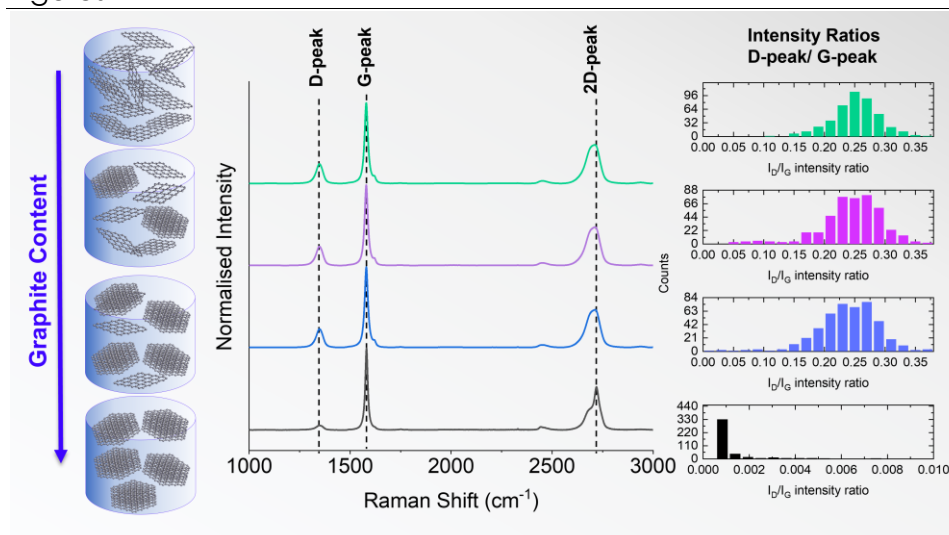


Figure 1: Raman spectroscopy measured from samples of few-layer graphene flakes with increasing amounts of unexfoliated material added. Very little difference in the spectra and extracted metrics could be seen.