

NPL REPORT AS 101

VAMAS TWA 41 - GRAPHENE AND RELATED 2D MATERIALS

**PROJECT 1 - STRUCTURAL CHARACTERISATION OF CVD-GROWN
GRAPHENE:
COVERAGE ON SUBSTRATE, NUMBER OF LAYERS, LEVEL OF
DISORDER**

RAMAN SPECTROSCOPY MEASUREMENT PROTOCOL

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VAMAS TWA 41 - Graphene and Related 2D Materials

Project 1 - Structural characterisation of CVD-grown graphene:
Coverage on substrate, number of layers, level of disorder.

Raman Spectroscopy Measurement Protocol

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ABSTRACT

This report details the measurement and data analysis protocol for Project 1 “*Structural characterisation of CVD-grown graphene: Coverage on substrate, number of layers, level of disorder*” of Versailles Project on Advanced Materials and Standards (VAMAS) Technical Working Area (TWA) 41 “*Graphene and Related 2D Materials*”. This project is an interlaboratory comparison of the measurement of structural properties of a graphene sheet grown through chemical vapour deposition (CVD) and transferred to a silicon dioxide surface. The protocol for Raman spectroscopy measurements and data analysis are described, with an aim to understand the uncertainty in this type of measurement, between laboratories spread across the globe.

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1 INTRODUCTION AND AIM

Graphene is predicted to impact many different application areas such as solar cells, biosensors, displays, composites, flexible electronics and energy storage due to its exceptional properties. One of graphene's many achievements is that it is the first truly two-dimensional material, being only 1 atom thick, and has opened up an area of research into a whole new family of other 2D materials. This has already shown that these new materials have exciting and complementary properties to graphene, revealing potential for many other industry applications.

As industry uptake on this material increases, international standardization is critical to enable commercialization. Reliable, accurate, and reproducible measurements are important due to the multiple production routes and producers of the material in order to maintain quality in manufacture.

Several standards are under development within the International Organization of Standardization (ISO) TC229 'Nanotechnologies', jointly with the International Electrotechnical Commission (IEC), including ISO/Preliminary Work Item (PWI) 21356 'Structural characterisation of graphene' standards, which focuses on determining the physical properties of graphene flakes and chemical vapour deposition (CVD) grown graphene. This will require interlaboratory comparisons (ILCs) to help develop best practice and understand the associated uncertainties.

Within this ILC project of TWA 41: 'Graphene and Related 2D Materials', Raman spectroscopy measurements will be performed on CVD-grown graphene, with the aim to understand the uncertainties between investigations performed in laboratories around the world due to instrumentation, measurement and data analysis. This understanding for the coverage of a substrate, the number of layers and level of disorder will then be used within ISO/PWI 21356-2: 'Structural characterisation of CVD-grown graphene'.

In Raman spectroscopy and microscopy, a laser is used to induce Raman scattering within a material so that a spectrometer can collect and record the amount of scattered light and determine the subsequent Raman shift (cm^{-1}). This technique is particularly powerful for studying carbon nanostructures such as graphene. For CVD-grown graphene, the coverage of graphene, number of layers and level of disorder can be determined and are investigated within this project.

CVD-grown graphene transferred onto Si/SiO₂ will be used for Raman spectroscopy measurements. The CVD-grown graphene has been sourced from an industrial collaborator. Separate samples will be prepared and initially measured by the lead participant, then a sample will be provided to each ILC participant, to allow a degree of normalisation between the Raman spectroscopy results.

1.1 PROJECT CONTACTS

The Project Contacts are Andrew Pollard (andrew.pollard@npl.co.uk) and Piers Turner (piers.turner@npl.co.uk). Any questions regarding this project can be directed to them, and all correspondence described herein should be directed to the Project Contacts.

2 INTERLABORATORY COMPARISON DETAILS

2.1 CONFIDENTIALITY

The samples supplied in this ILC are not certified reference materials, but have been selected for this VAMAS comparison as the best available type of sample for this study. They are sent to the participants in confidence, and if there are any problems with the received sample please contact the lead participant immediately so that it can be determined whether the problem is generic or restricted to a single sample. Please do not publish individual results, or any further or different analysis of these samples without first consulting the Project Contacts (see Section 1.1). Individual results will not be identified with a particular laboratory.

2.2 TIME SCHEDULE

Upon receipt of the samples, please email the Project Contacts (see Section 1.1) to confirm receipt.

Results and samples should be returned to the Project Contacts by **June 3rd 2019**.

The additional Measurement Report (attached separately) should be completed electronically and emailed to the Project Contacts, along with the data requested in Section 6.

Raw data should be uploaded using the information provided in Section 6.1.

Samples should be returned to:

Piers Turner
National Physical Laboratory
Hampton Road
Teddington
Middlesex
TW11 0LW
United Kingdom

2.3 ILC SAMPLES DESCRIPTION

A 10 mm × 10 mm sample of CVD-grown graphene transferred onto a Si/SiO₂ substrate (300 nm oxide layer) from the copper growth substrate, will be provided to each participant. This sample has had one area measured using Raman spectroscopy by the lead participant (NPL) and the location of this measurement position (including optical images) will be provided with the sample along with a Safety Data Sheet (SDS).

2.4 PACKAGING AND HANDLING

These samples are not deemed hazardous, but every care should be taken to avoid contact with the skin and eyes, and the sample should not be consumed. Good laboratory practice should be observed, and participants should refer to the SDS for further/specific details.

Samples are provided in a Fluoroware container as shown in Figure 1, where the surface with the graphene sheet is facing downwards and a small mark has been made on the reverse side of the sample.

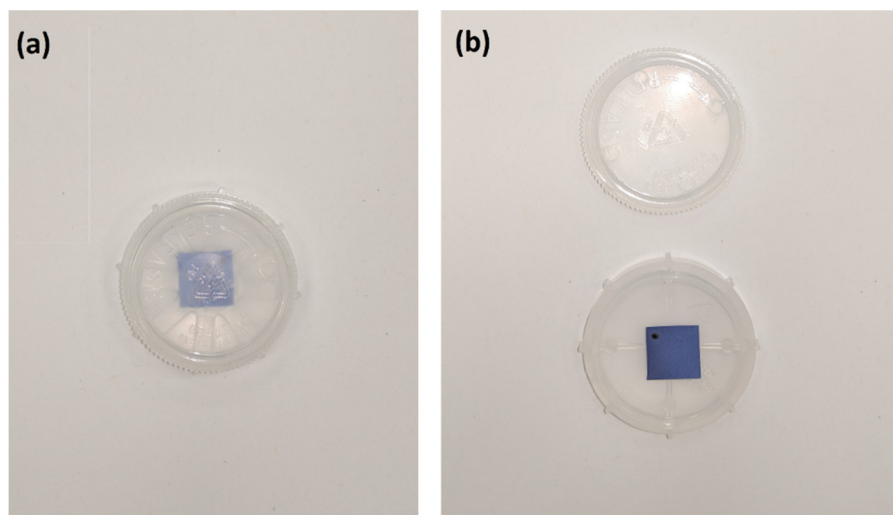


Figure 1: Photographs of a sample when (a) sealed and (b) open with 'spider' removed.

To handle the sample, first open the Fluoroware sample container and remove the plastic 'spider' that secures the substrate within the sample container. After removing the spider, the substrate will be orientated with the backside facing up and the graphene side facing down. The backside of the sample has been clearly marked with a black dot to show the corner of interest (Figure 2a). As the graphene side of the wafer is facing down, the wafer will need to be flipped 180° along either the vertical or horizontal axis, to reveal the graphene. The most reliable way to flip the wafer is to put the lid back on the Fluoroware sample container (without the spider in place), flip the container with the wafer inside it, and then open the container upside down such that the graphene (shiny side) is facing upwards (Figure 2 b-d).

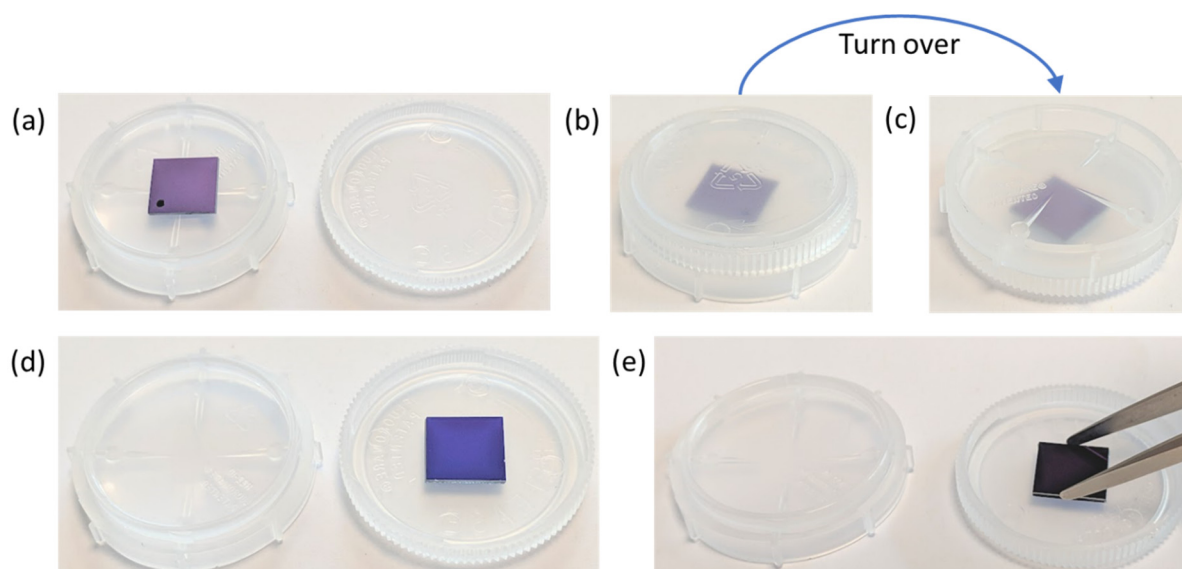


Figure 2: (a) The substrate in the base of Fluoroware sample container, with the back (matte) side facing up and 'spider' removed. The black dot on the back side of the substrate shows the corner of interest. To remove the substrate from the Fluoroware sample container, (b) close the lid then (c) turn the Fluoroware sample container upside down with the substrate still inside it. (d) Open the Fluoroware sample container upside down such that the substrate is in the lid of the Fluoroware sample container with the graphene (shiny) side facing up. (e) Remove the substrate by holding the edges of the substrate with the edge of the metal tweezers.

When the wafer has been flipped such that the graphene (shiny side) is facing upwards, hold the edges of the wafer using metal tweezers and place it onto the sample stage within your Raman spectroscopy system.

The area of interest is approximately above the dot on the backside of the wafer. The coordinate system used to locate the first Raman spectroscopy imaging area is shown in Figure 3.

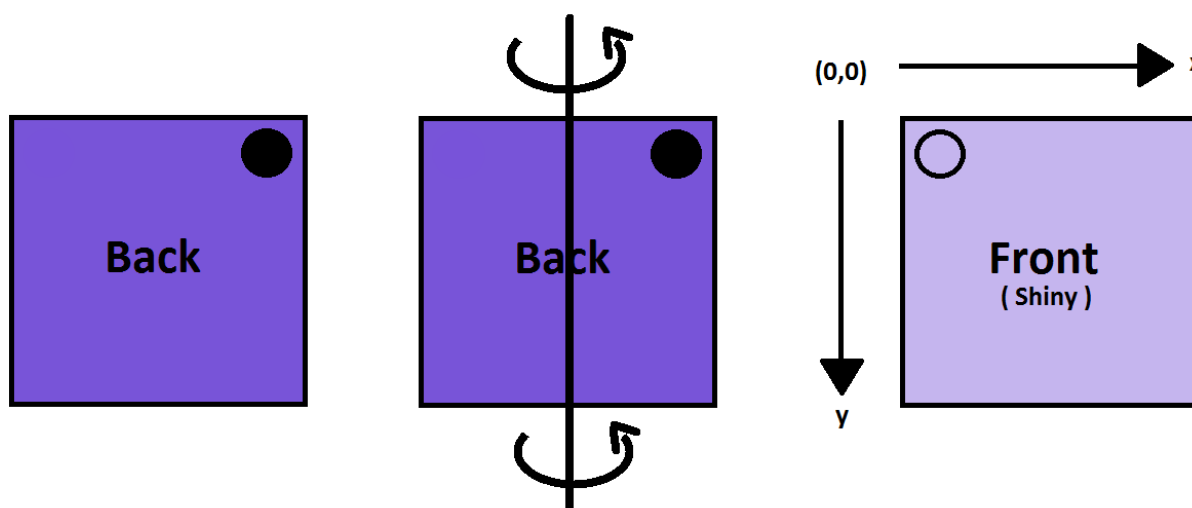


Figure 3: A schematic of the wafer coordinate system and how it relates to the position of the dot on the backside of the wafer. The first area of interest is in close proximity to the (0,0) coordinates.

The optical images in Figure S1, S2 and S3, supplied with the sample, along with the coordinate system, will help locate the first measurement position. Figure S1 shows the whole 10 mm × 10 mm sample and its coordinate system, Figure S2 shows the location and coordinates of the first measurement position relative to the top-left-hand corner of the sample, and Figure S3 shows the coordinates used to capture the two-dimensional Raman spectroscopy image. Care has been taken to ensure the measurement position has user friendly coordinates and is located close to a recognisable feature(s).

If there are any problems with the received sample, or you cannot find the area of interest due to damage that may have occurred during shipping, contact the Project Contacts (see Section 1.1) so that it can be determined whether the problem is restricted to a single sample or more general across all participants.

3 INSTRUMENTATION

A Raman spectroscopy instrument capable of measurement in a 180° backscattering geometry configuration, to provide the best signal-to-noise ratio, should be used, with a $50\times$ or $100\times$ objective lens ($NA \geq 0.75$). The details of the geometry and objective lens **must be recorded in the accompanying Measurement Report**. For more details on the Measurement Report, refer to Section 6.

Typically, a full spectral calibration of the system should be regularly performed using a neon lamp (or another gas lamp with traceable features), and a spectral calibration should be performed daily before use, using for example the 1st order Si peak of a silicon sample. It is expected that at least a daily calibration using the 1st order Si peak of a silicon sample is performed before measurements take place. To aid the understanding of sources of uncertainty in this ILC, the calibration procedure and resulting data **must be recorded**.

All raw data must be corrected in terms of spectral position according to the daily calibration step. All measurements should be collected at room temperature, both the laboratory temperature and the humidity during the experiments **must be recorded**. Importantly, the temperature should remain constant ($\pm 2^\circ\text{C}$) during the measurement procedure.

A green laser with a wavelength of 532 nm (2.33 eV) should be used for the graphene on a Si/SiO₂ substrate. If this is not possible, then a laser with a wavelength of 514 nm shall be used. It should be noted that Raman spectroscopy measurements using different laser wavelengths cannot be directly compared for graphene and so the wavelength of the laser **must be recorded** along with the Raman spectra.

To avoid damaging the graphene itself through localised heating effects, the incident laser power shall be less than 1 mW for a laser spot diameter of ~ 500 nm or greater. Acquisition times must be less than 30 seconds for each measurement position. The laser spot power must be measured using a power meter at the point where the sample surface would be, i.e. at the focus, relative to the objective lens, and the laser power **must be recorded**.

To fulfill the requirements of this ILC, it must be possible to measure a spectral range such that the relevant Raman bands: D band ($\sim 1350\text{ cm}^{-1}$), G band ($\sim 1580\text{ cm}^{-1}$), 2D band (also called the G' band and at $\sim 2700\text{ cm}^{-1}$) and associated widths are included, either in the same spectrum or in separate spectra. Therefore, a Raman spectrum for the **range from 1200 cm^{-1} to 2900 cm^{-1}** must be obtained as a minimum. The details of the grating grooves (in lines/mm) **must be reported**. An example Raman spectrum is included in Figure 4.

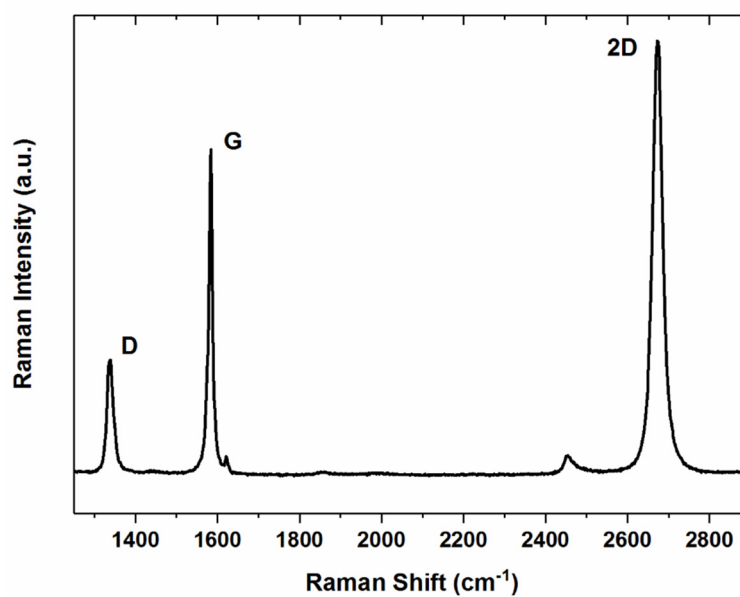


Figure 4: Raman spectrum for single layer graphene, showing the D-peak, G-peak and 2D-peak.

Two Raman spectroscopy images of different areas must be obtained for each sample, for different areas of the surface, the instrumentation must be able to produce Raman spectroscopy images of $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$, with a step size of $1\text{ }\mu\text{m}$, i.e. a minimum of 121 pixels. Note that the step size is not related to the laser spot size of individual instruments.

For other operations not described in this section or Section 4, follow the manufacturer guidelines for the instrument.

4 MEASUREMENT PROTOCOL

1. Making sure to note where the mark on the back of the sample is, secure the sample so that the substrate is horizontal with respect to the excitation laser beam and is fixed with respect to the stage, when the stage is moved during sample scanning.
2. Locate the first measurement position for Raman spectroscopy imaging using optical microscopy, comparing with the measurement position details (optical images and dimensional position) provided with the sample in Figures S1, S2 and S3.
3. After locating the measurement position with optical microscopy, the signal strength should be optimised by adjusting the focus. This is accomplished by adjusting the Z-focus position and performing Raman spectroscopy measurements to determine the position of the highest Raman peak intensity and area.
 - a. Approximate the focus position using optical microscopy, such that the substrate surface/graphene layer is in focus.
 - b. Depending on the capability of the instrument, an example method for this step would be to measure Raman spectra for at least 11 positions in the Z-axis, over a total travel distance of at least 2 μm .
 - c. A measurement time of 5 seconds should be used for each spectra, unless this does not provide a peak intensity (S) 10 times greater than the peak-to-peak intensity of the background signal (N) for the G- and 2D-peaks for the spectra obtained at the position of highest Raman scattering intensity. An example of this S/N ratio of greater than 10 for the G-peak is shown in Figure 5. If a S/N ratio of less than 10 is observed, a longer measurement time should be used to achieve $S/N > 10$. Note that as with all the Raman spectroscopy measurements in this ILC, ≤ 1 mW laser power should be incident on the sample.

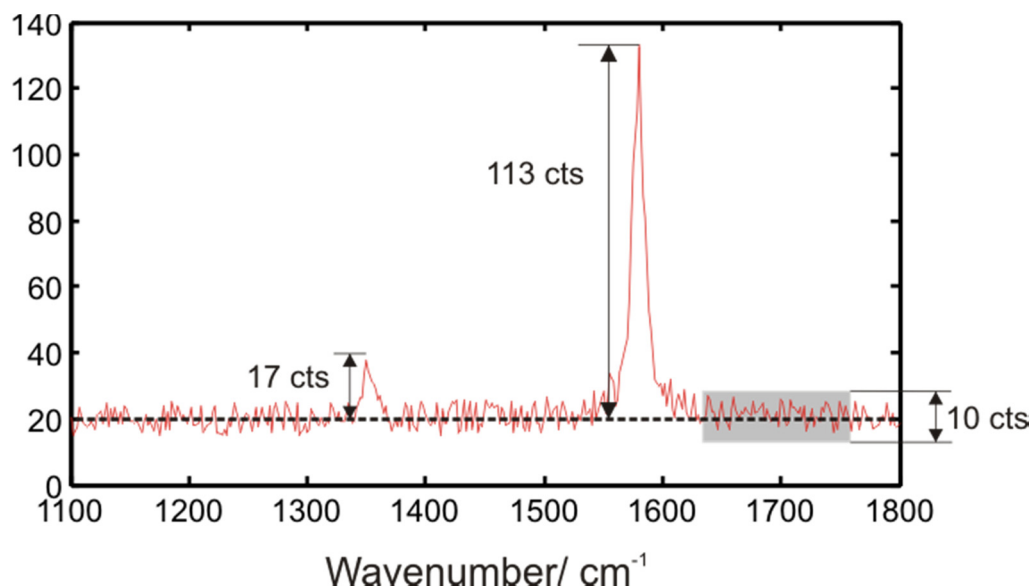


Figure 5: Raman spectra revealing a G-peak with a S/N ratio of 11.3. Image kindly provided by Dr. Erlon Henrique Martins Ferreira, Instituto Nacional de Metrologia, Brazil.

4. Microscale Raman spectroscopy imaging must be performed over the same area as the two-dimensional area shown in Figure S3. An image of $10 \times 10 \mu\text{m}^2$ must be obtained consisting of at least 121 (11×11) Raman spectra, with $1 \mu\text{m}$ steps between positions.
 - a. The same laser power as in Step 3c should be used, with an acquisition time of twice that used in Step 3c to achieve a S/N ratio of at least 20
5. Repeat Step 3 and 4 for a measurement position at roughly the centre of the sample, so that a total of two Raman spectroscopy images have been recorded for the sample.
6. After the images have been acquired, the data should be analysed to determine the sample coverage, percentage of single layer graphene and level of disorder, as described in Section 5.

5 DATA ANALYSIS

5.1 CURVE FITTING

Firstly, for each of the three peaks of interest (D-peak ($\sim 1350\text{ cm}^{-1}$), G-peak ($\sim 1580\text{ cm}^{-1}$) and 2D-peak ($\sim 2700\text{ cm}^{-1}$)) an area of interest must be determined. This area of interest must be 200 cm^{-1} wide and centred on the peak. For each area of interest a linear baseline should be determined and subtracted for each spectrum, before peak fitting is performed. If a linear baseline is not used, the details of how the baseline is calculated **must be recorded**. The three peaks of interest (D-peak ($\sim 1350\text{ cm}^{-1}$), G-peak ($\sim 1580\text{ cm}^{-1}$) and 2D-peak ($\sim 2700\text{ cm}^{-1}$)) must be curve fit using a Lorentzian function for the whole area of interest. This should be performed using Raman spectroscopy software, or other software such as Origin® or MATLAB®.

If a significant D-peak is observed, the D'-peak at $\sim 1620\text{ cm}^{-1}$ may need to be fitted in addition to the G-peak. These Raman peaks for an example single layer graphene sample with varying levels of defect density are shown in Figure 6.

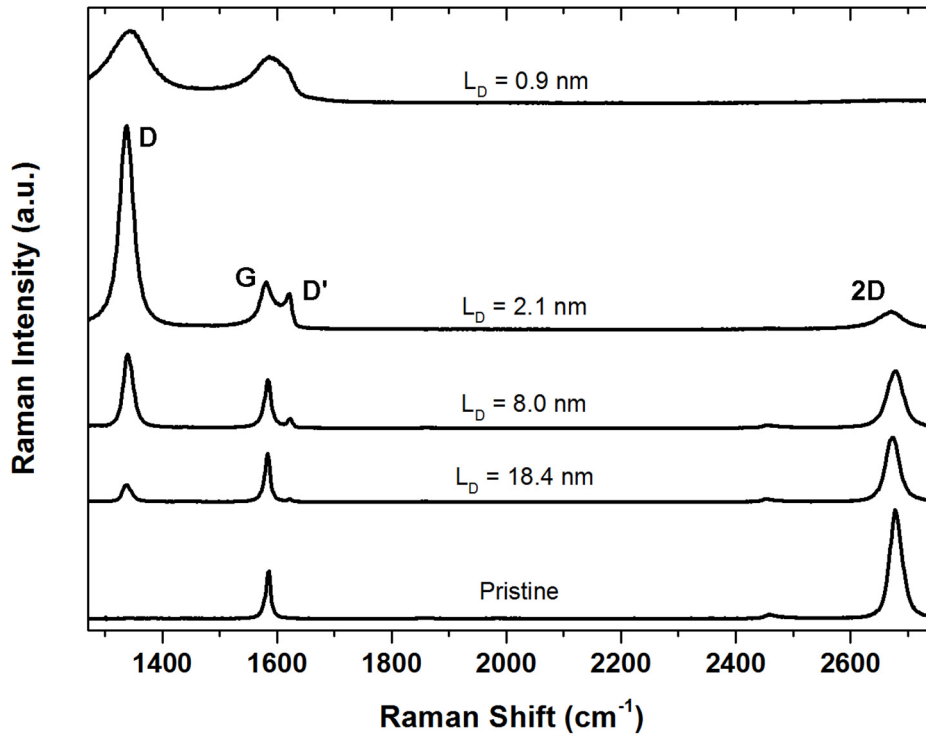


Figure 6: Raman spectra for single layer graphene samples with different inter-defect distances (L_D) with labelled Raman peaks, from Pollard *et al.*, Appl. Phys. Lett. 105, 253107, 2014.

The five parameters listed below must be calculated for each spectra using the fitted peaks previously described and these parameters must be recorded as histograms (minimum of 10 bins) and two-dimensional images, as shown in Figure 6, for each of the two measurement areas of the sample:

- Peak area for the G-peak, A_G
- Peak intensity ratio for the G- and 2D-peaks, I_{2D}/I_G
- Peak intensity ratio for the D- and G-peaks, I_D/I_G
- Full-width at half-maximum (FWHM) of the G-peak (FWHM[G])
- Full-width at half-maximum (FWHM) of the 2D-peak (FWHM[2D])

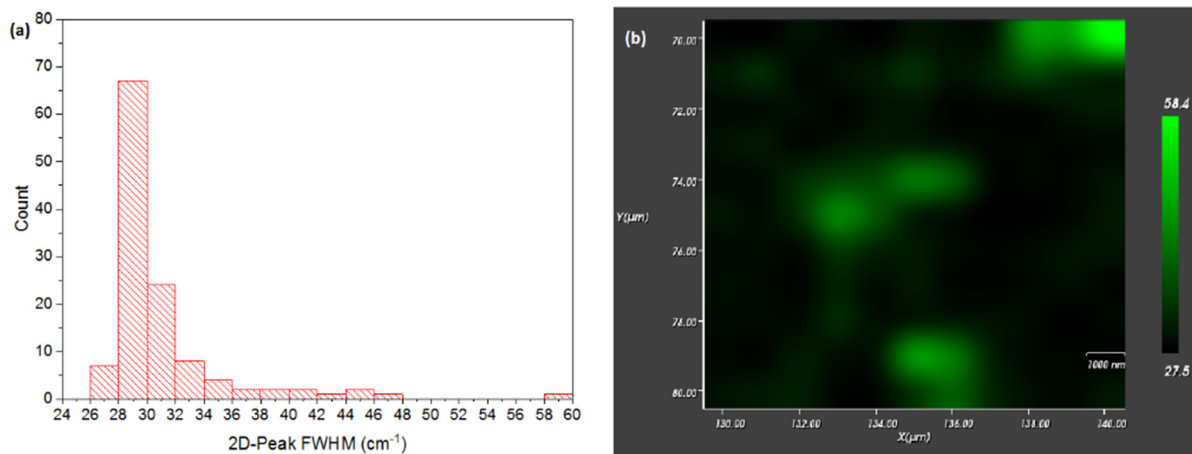


Figure 4: Examples of (a) histogram and (b) 2D image of 2D-peak width data.

The peak intensity is the maximum value of intensity for a Raman peak after a baseline has been determined/subtracted and peak fitting has been performed. The peak area is the area under the curve after peak fitting.

5.2 DETERMINATION OF PROPERTIES

5.2.1 Calculation of Coverage

The percentage of the substrate covered by single layer graphene (1LG), >1LG (i.e. bilayer graphene (2LG) or few-layer graphene (FLG)), and areas with no coverage, must be calculated. This is done through the number of measurement points in the Raman spectroscopy images that reveal a G-peak (shown by the G-peak area), to determine the microscale variations in coverage of graphene.

Pixels of the image where a G-peak is less than 10% of the average G-peak area, are recorded as having no 1LG/2LG/FLG present. The percentage of the pixels with no coverage observed, using the 242 (121+121) pixels measured, **must be recorded in Table 1.**

5.2.2 Calculation of Numbers of Layers

In this study, the percentage of coverage of the substrate of 1LG and the percentage of coverage of 2LG and FLG (i.e., greater than 1 layer of graphene) will be calculated and recorded in Table 1. Note that if there is not 100% coverage of the sample (determined in Section 5.2.1), then the percentage of 1LG and >1LG must take this into account when recorded in Table 1.

The FWHM[2D] is used to determine whether the graphene is 1LG or greater than one layer and thus the percentage of single layer graphene present in the sample. Turbostratic stacking of the graphene layers is assumed for more than one layer of graphene for this sample, based on previous transmission electron microscopy (TEM) studies.

Pixels with a Raman 2D-peak that is a single Lorentzian peak with FWHM[2D] of $\leq 35 \text{ cm}^{-1}$, are determined to be 1LG and count towards the percentage of '1 layer' areas in Table 1. However, it is understood that if pixels with $I_D/I_G > 0.2$ are observed, these areas may still be 1LG even though this FWHM[2D] criteria is not met, even if this is not taken into account in this study.

The percentage of pixels with FWHM[2D] $\leq 35 \text{ cm}^{-1}$ for both images should be calculated and used to determine the total coverage of 1LG and >1 layer (i.e. from 121+121=242 pixels).

Table can then be completed using the data from both measurement positions where imaging was performed.

Table 1: Reporting scheme for characterisation of CVD-grown graphene on a Si/SiO₂ substrate

Number of graphene layers	Area (%)
1 layer	
>1 layer	
No graphene present	

5.2.3 Calculation of Level of Disorder

The values of I_D/I_G and FWHM[G] should be recorded as the measurands for the level of disorder of the graphene. The level of disorder for the sample must be expressed as the average of I_D/I_G as well as the average of the FWHM[G], but only for each spectra acquired for areas where 1LG or >1LG are present (as determined in the previous Sections). Thus two individual values of I_D/I_G and FWHM[G] will be recorded for the sample, as described in Section 6.

5.3 RECORDING DATA

The raw data for each of the two Raman spectroscopy images must be supplied to the lead participant in both a .SPC and .TXT file format. An example spectra must be included for each measurement area with curve fitting included.

The analysed data (that was used to plot the final images, as detailed in Section 5.1) must also be provided in a .TXT format, for the five images and five histograms associated with each image produced. The image number and the parameter recorded must be noted in the file name.

6 MEASUREMENT REPORT

The participants should report the following data by email to the Project Contacts (Section 1.1), using the Measurement Report in the attached document and the OneDrive details. Note that an example Measurement Report is also included on the OneDrive as a guide.

Measurement Report:

- Instrument make and model
- Instrument calibration procedure
- Instrument laser wavelength
- Instrument grating details
- Laser power incident on the sample
- Laboratory temperature and humidity
- Dates (DD/MM/YYYY)
- Details of the baseline calculation
- Central position of Area 2 (centre of sample) using the same coordinate system as the supplementary figures.
- Completed Table 1 (this table includes data for both measurement positions)
- An average I_D/I_G value for both measurement areas
- An average FWHM[G] value for both measurement areas

Sent in Email:

- Area 1:
 - An image (.JPG) of an example spectra, with curve fitting shown
 - An optical image (.JPG) of the area measured
 - Five Raman spectroscopy images (.JPG)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM[G], FWHM[2D]
 - Five histograms (.JPG)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM[G], FWHM[2D]
- Area 2:
 - An image (.JPG) of an example spectra, with curve fitting shown
 - An optical image (.JPG) of the area measured
 - Five Raman spectroscopy images (.JPG)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM[G], FWHM[2D]
 - Five histograms (.JPG)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM[G], FWHM[2D]

Uploaded to OneDrive:

- Area 1:
 - Raw data in .SPC and .TXT format (see Section 6.1 and 6.2).
 - Analysed data in .TXT format of 5 images (see Section 6.1 and 6.3)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM[G], FWHM[2D]
- Area 2:
 - Raw data in .SPC and .TXT format (see Section 6.1 and 6.2).
 - Analysed data in .TXT format of 5 images (see Section 6.1 and 6.3)
 - G-peak area, I_{2D}/I_G , I_D/I_G , FWHM [G], FWHM[2D]

6.1 UPLOADING FILES

Data files that must be uploaded, as previously described, should be uploaded to Microsoft OneDrive, as described in an email from the participant before samples have been dispatched. Any questions or issues should be directed to the Project Contacts (see Section 1.1).

6.2 RAW DATA IN .TXT FORMAT:

Where possible raw data in .TXT format should be provided in the preferred file format below, otherwise the format should be specified in the Measurement Report:

```
- - R1    R2    R3    .... RN
x1 y1 I1_1_1 I1_1_2 I1_1_3 .... I1_1_N
x1 y2 I1_2_1 I1_2_2 I1_2_3 .... I1_2_N
  ::   :       :       :       .... :
x1 yq I1_q_1 I1_q_2 I1_q_3 .... I1_q_N
x2 y1 I2_1_1 I2_1_2 I2_1_3 .... I2_1_N
  ::   :       :       :       .... :
xp yq Ip_q_1 Ip_q_2 Ip_q_3 .... Ip_q_N
```

Where x and y are measurement positions (lateral), R is the Raman shift, and I is the intensity, for a system with N CCD pixels and for $p \times q$ number of lateral positions.

6.3 ANALYSED DATA IN .TXT FORMAT

Analysed data should be provided in .TXT format, as below:

```
x1 y1, A[D]1_1, I[D]1_1, FWHM[D]1_1, A[G]1_1, I[G]1_1, FWHM[G]1_1, A[2D]1_1, I[2D]1_1,
FWHM[2D]1_1
x1 y2, A[D]1_2, I[D]1_2, FWHM[D]1_2, A[G]1_2, I[G]1_2, FWHM[G]1_2, A[2D]1_2, I[2D]1_2,
FWHM[2D]1_2
....
x1 yq, A[D]1_q, I[D]1_q, FWHM[D]1_q, A[G]1_q, I[G]1_q, FWHM[G]1_q, A[2D]1_q, I[2D]1_q,
FWHM[2D]1_q
x2 y1, A[D]2_1, I[D]2_1, FWHM[D]2_1, A[G]2_1, I[G]2_1, FWHM[G]2_1, A[2D]2_1, I[2D]2_1,
FWHM[2D]2_1
....
xp yq, A[D]p_q, I[D]p_q, FWHM[D]p_q, A[G]p_q, I[G]p_q, FWHM[G]p_q, A[2D]p_q, I[2D]p_q,
FWHM[2D]p_q
```

Where A is the peak area and I is the peak intensity.

7 ACKNOWLEDGEMENTS

The authors are grateful to Keith Paton, Alex Shard, Steve Spencer, Charles Clifford and Caterina Minelli at NPL and Erlon Henrique Martins Ferreira (Instituto Nacional de Metrologia, Brazil) and Angela Hight-Walker (National Institute of Standards and Technology, USA) for detailed discussions related to this document. The authors would also like to thank Amy Paterson for her aid in the logistics of this ILC.

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