Extended Abstract of PSA-19

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Standardized Estimation of Spatial Resolution of Confocal Raman Imaging

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The confocal Raman microscope is a very useful tool that spatially visualizes the Raman activity of a sample with diffraction-limited high spatial resolution. Spatial resolution is a key specification of the performance of a manufactured Raman microscope but is often characterized differently. Here, test specimens made using dispersed carbon nanotubes and suspended graphene were prepared and measured to provide standardized estimation of the lateral and axial spatial resolution of the Raman microscope. General information of the standard specimen for lateral or axial resolution and case studies of actual measurements are also provided.

1. Introduction

The confocal Raman microscope is a strong technology for studying material properties such as material structure, strain and molecular bonding with good spatial resolution. This spatial resolution is important to study of material characteristics. There have been many attempts to increase spatial resolution [1]. However, when determining the spatial resolution, it requires many optimized conditions such as the pinhole size, the objective lens and the alignment of the optical lens. As the size of the material to be measured continues to decrease, it is very important to know the exact spatial resolution value. A suitable test samples are demanded to determine the precise spatial resolution value. The carbon nanotubes (CNTs) [2] and suspended monolayer graphene (SMG) [3] are good materials to obtain the lateral and axial resolution respectively.

We extracted the line profile from CNTs Raman image and depth profile from the Z-scan of the SMG. Measured result of lateral and axial resolution is 344 nm and 1.21 μm, respectively

2. Experimental methods

2.1 Sample preparation

0.01 g of Multi-walled CNT (MWCNTs) powder was placed in 10 mL of dichloroethane (DCE) to make 1 μg/mL solution. And then bath sonication for a long time to be well-dispersed in the solvent. Before spin coating on substrate, we dilute 10 times in the same solvent (0.1 mL of 1 μg/mL CNT in DCE solution + 0.9 mL of DCE). A few drops of a 0.1 μg / mL CNT solution were dropped onto the cleaned substrate. And spin-coated for good dispersion throughout the glass substrate. Dispersed CNTs on glass substrate was used for measuring the lateral resolution.

We use monolayer graphene suspended on TEM grids for measuring the axial resolution.

2.2 Optical measurement

We used an optical microscope (Alpha-300S, WITec Instrument GmbH) system using a ND-YAG (532 nm) laser as a light source. Samples were fixed on the microscope stage. The laser output was less than 1 mW to avoid sample damage. The collimated laser light was guided to a beam splitter and focused on the sample through a 100x, 0.9 NA objective lens (OL, Zeiss). The long-pass filter placed in front of the spectrometer was used to block the laser light and only the Raman scattering from the sample was collected by the same OL and an optical fiber with a diameter of 100 μm was acted as a confocal pinhole and guided to a length of 30-long spectrometer. It has a 600 grooves/mm grating and a cooled CCD. For lateral resolution measurement,

the laser focus was moved by pixel to pixel by scanning the sample and Raman image was acquired. It was acquired by 30 nm step size. For axial resolution measurement, the z-position was set piezoelectric positioning table by +5 μm to -5 μm from the Graphene's focus and measured to 100 nm step size.
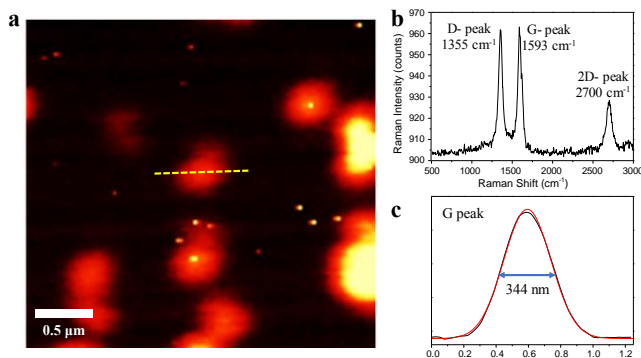
3. Result

3.1 Lateral resolution

Fig. 1 a) CNT Raman image. b) CNT Raman Spectra.
c) Lateral line (yellow line) profile of G-band.

For measurement of lateral resolution, we used carbon nanotubes (CNTs) on cover glass. CNT has tens of nm in width and strong Raman signal. If the actual diameter of the CNT is within a few tens of nanometers, the width of the CNT in the Raman image is reasonable.

In Fig. 1a, we showed the result of Raman mapping of dispersed CNTs prepared on glass substrate. Fig. 1b displayed the representative Raman spectrum of the sample and we used the G-peak to produce the Raman image. One of the bright spots that correspond to the locations of CNTs, was chosen and a cross-sectional line profile was extracted (indicated as the dotted line in Fig. 1a) as the result is shown in Fig. 1c. The line profile was fitted with the Voight function and 344 nm of full width at half maximum (FWHM) was estimated, which is regarded as the lateral resolution of our Raman system.
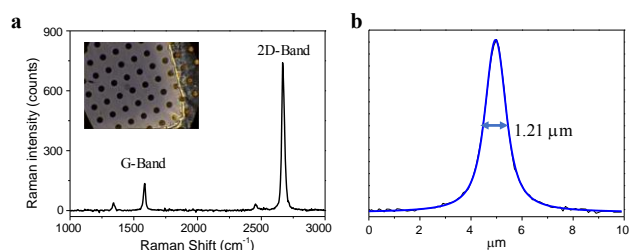
For measurement of axial resolution, we use SMG on TEM grids, because SMG has a sub-nm thickness and strong Raman signal. SMG can be good candidate an ideal sample for axial resolution.

In Fig. 2a, we displayed the spectra of SMG on TEM grids which is G-band and 2D-band. Whole areas were covered by MG and the other areas were Au coated TEM grids as shown in inset image of Fig 2a. Fig. 2b represented the depth profile of SMG. It was fitted with 2D band of SMG and 1.21 μm of FWHM was estimated, which is regarded as the axial resolution of our Raman system.

4. Conclusion

We described the methods to estimate the lateral and axial resolution of confocal Raman imaging by using dispersed CNTs and suspended graphene layer, respectively. We found that the CNT and suspended graphene are suitable as test specimens for the measurement of sub-micron optical spatial resolution of Raman imaging. In both of lateral or axial resolution measurements, we found there was some variation in measured FWHMs mostly within 10 %, therefore average of multiple measurements is recommended as the final result.

5. References

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3.2 Axial resolution

Fig. 2 a) SMG Raman Spectra. Inset: Optical image of SMG
b) Axial line profile of 2D-band