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Simplest method of measuring nanoparticles' size and size distribution on the surface and on sub-surface layers

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Recently there has been an emphasis for characterizing nanostructured materials such as two-dimensional (2D) and zero-dimensional (0D) materials. For example, graphene, carbon nanotubes, etc. are two-dimensional (2D) materials. Quantum dots (QDs) [1] is an example of a zero-dimensional (0D) material because the nano particles of the QDs do not form a network; instead they remain by themselves. See ref [2] for a review of 0D, 1D, etc. materials.

While synthesis of these nanomaterials has enjoyed success, however, characterizing various structural properties of these materials still face significant challenge. In this review, we describe a method for measuring the size and size distribution of nanoparticles via terahertz imaging technique. Reconstructive imaging via terahertz reflectometry has been described [1] where sub-nanometer resolution was demonstrated by a nanoscanner and a gridding algorithm.

ARP's terahertz nanoscanner offers a straightforward means for characterizing nanoparticles for their size parameter and size distribution. A step-by-step outline is given herein.

1. Mount the sample on the nanoscanner. Here an 8″ wafer is mounted (see Fig. 1); however, samples may be of any size or shape.

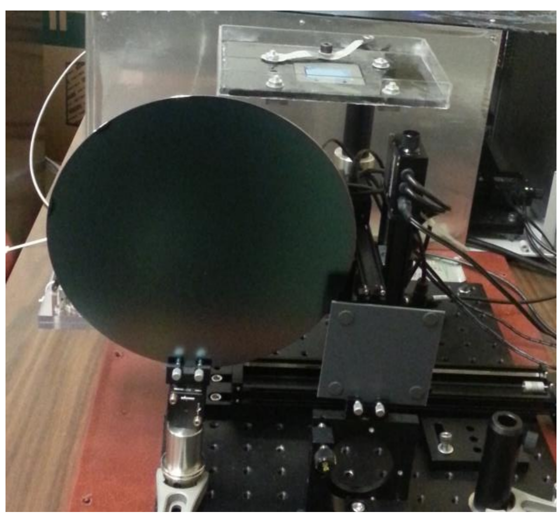

Fig. 1. An 8″ wafer is mounted on the nanoscanner. Rough side is visible; sample is on the opposite side, facing the scanning beam. Sample remains stationary.

2. Conduct the scanning over a suitable area or volume. Minimum scan able area for the current setup is 1 μm^2 or a volume of 1 μm^3. The scanning resolution ranges from a few hundred microns down to ~25 nm, in all three orthogonal directions (see Fig. 2). Appropriate scanning routine should be chosen either for Cartesian coordinates or for imaging curved surfaces and/or cylindrical volumes.

3. Convert the rasterized data to image via reconstructive imaging algorithm with provided software program; see Fig. 3 (a) for rasterized data and 3 (b) for a reconstructive image of the same data. The inverse gridding algorithm is described in details in ref. [1]. In Fig. 3 (b), only the top surface is shown.

4. View a 3D image (if applicable) and also you may want to examine different layers one at a time. This is outlined in Fig. 4.

5. Extract a single layer of interest from the 3D image as shown in Fig. 5. You may want to render the image in greyscale for digitizing; however, greyscale is not mandatory for simple particle size analysis.

6. Analyze the image for individual particle's size measurement or for particle size distribution. An example is shown in Fig. 7 for the image in Fig. 6.

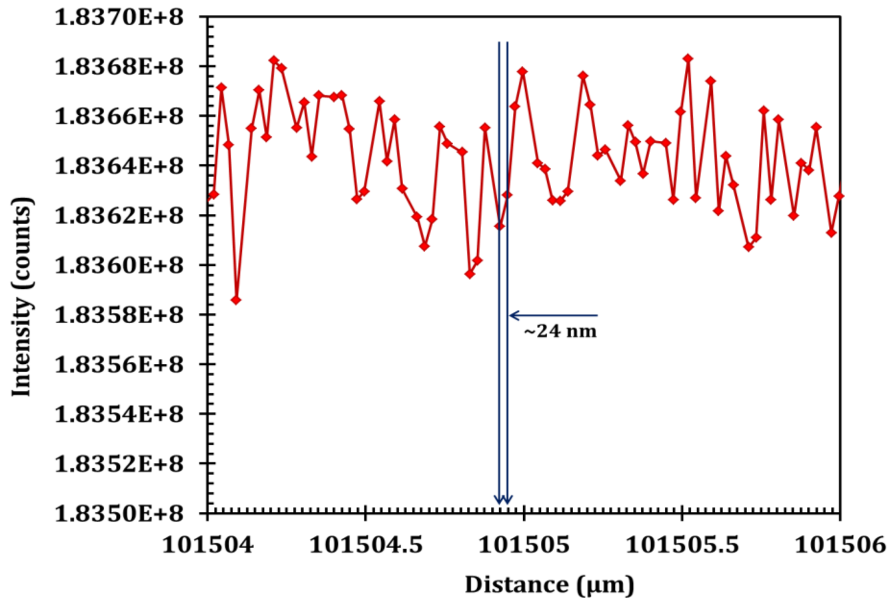

Fig. 2. Linear scanning resolution is ~24 nm.

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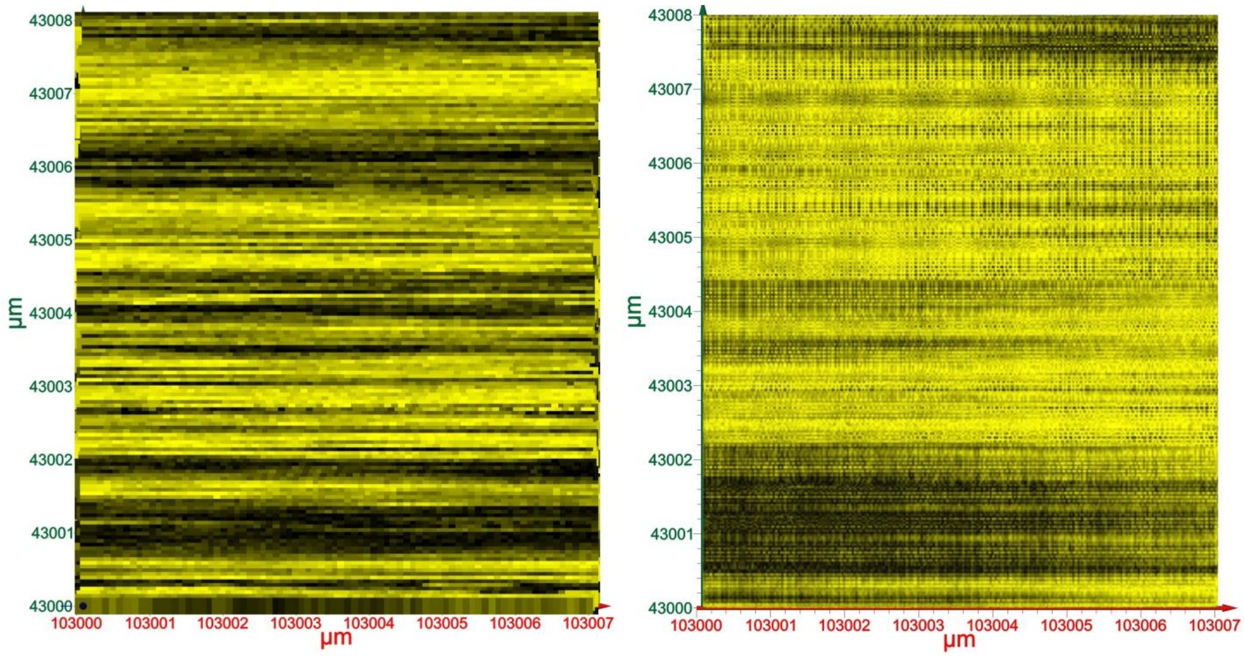

Fig. 3. Example of imaging of carbon nanotube (CNT) samples obtained by personal correspondence with Prof. Junichiro Kono of Rice University [3]. (a) As measured (rasterized) data, (b) reconstructive image formed from the rasterized data via inverse gridding algorithm.

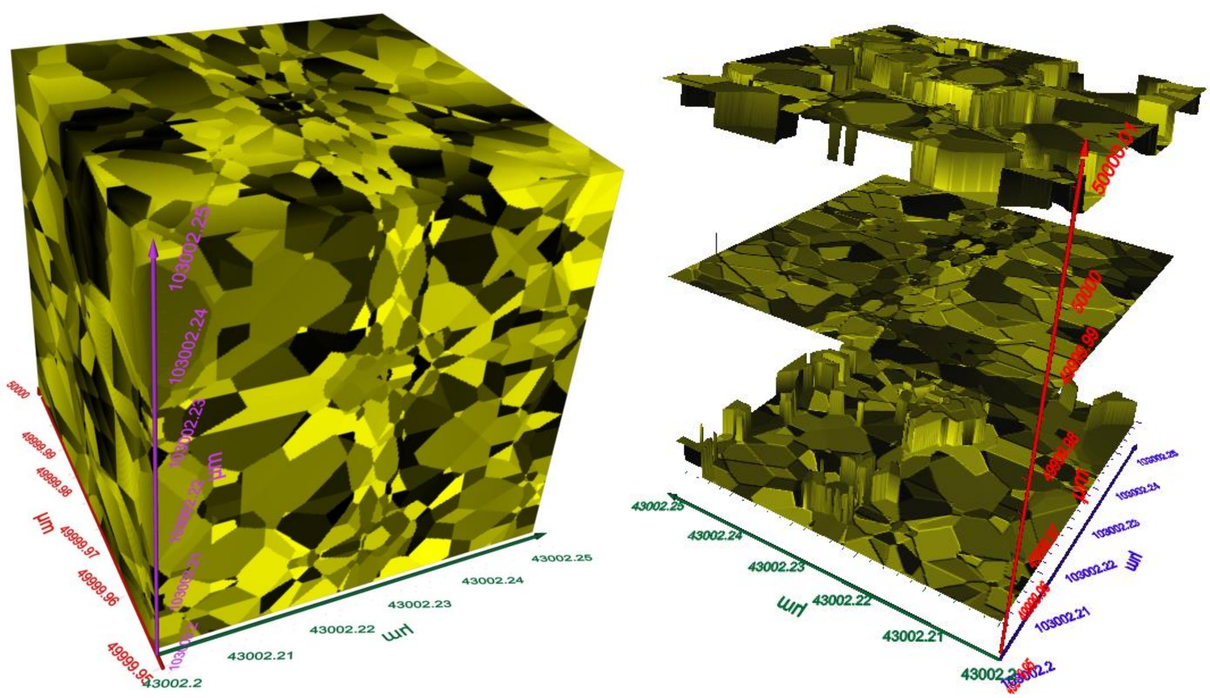

Fig. 4. (a) 3D image of 50 nm x 50 nm x 50 nm volume of the CNT sample. (b) Three different layers of the volume in (a) are shown. Each layer may be analyzed independently.

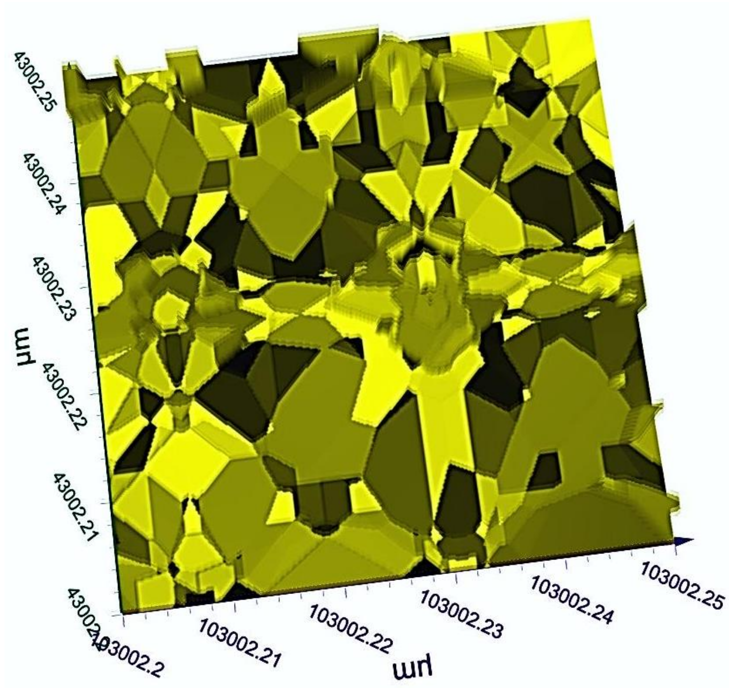

Fig. 5. A 50 nm x 50 nm area extracted from Fig. 4 (a).

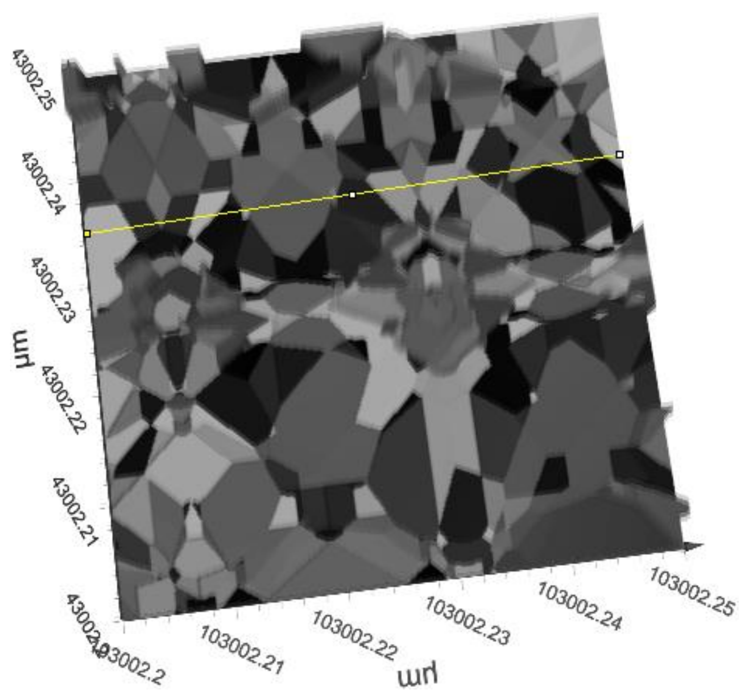

Fig. 6. A slice of the surface image rendered in greyscale for digitizing.

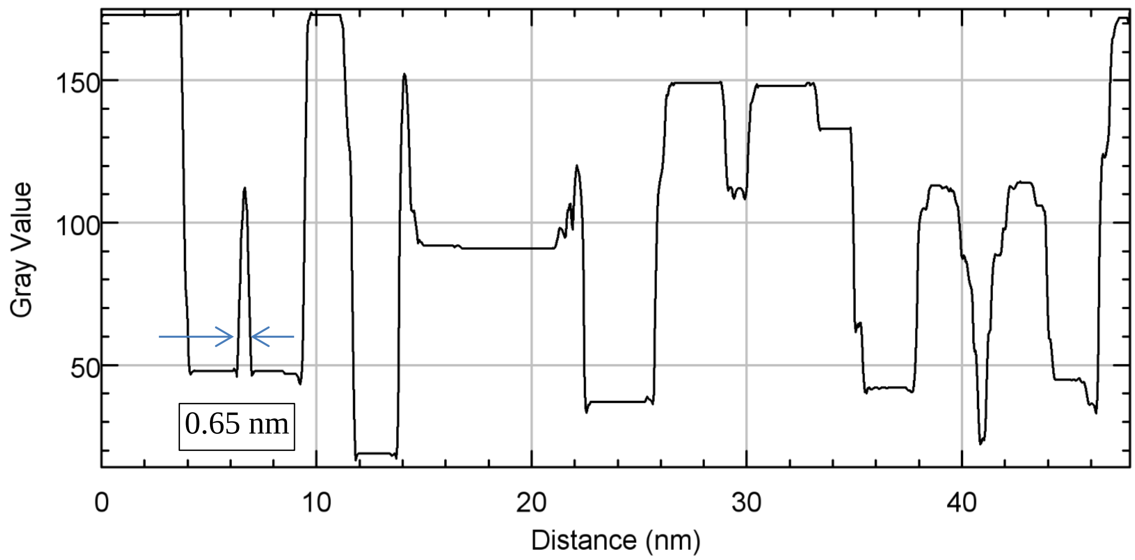

Fig. 7. Individual particles and their size distribution along the line in Fig. 6. Smallest particle size is ~0.65 nm.

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References

1. Rahman, A. et al. (2016). "Terahertz Sub-Nanometer Sub-Surface Imaging of 2D Materials." Journal of Biosensors & Bioelectronics **7**(3): 1-8. doi: 10.4172/2155-6210.1000221

2. Tiwari, J. N., et al. (2012). "Zero-dimensional, one-dimensional, two-dimensional and three-dimensional nanostructured materials for advanced electrochemical energy devices." Progress in Materials Science **57**: 724-803.

3. These samples were received from Prof. Junichiro Kono of Rice University. We are thankful to Prof. Kono for the samples.