Application Note

Producing superior quality carbon films for EM sample preparation with the Q150V Plus

Dr. Anna E Walkiewicz (2018)

In carbon thin film applications the quality of the carbon film production is key to successfully conducting complex experiments.

Commonly, carbon coatings are used in sample preparation for TEM (ambient and cryo) imaging, EDS analysis and creating replicas.

The advantage of using carbon films is that they are transparent to the electron beam. The thin carbon films are conductive and easy to produce. They are free from contamination, smooth and strong, and most of all ,they can be prepared thin enough that they do not attenuate the contrast when imaging the structural elements of specimen. On the one hand, in TEM imaging – the most demanding of high carbon film quality applications – the support layer has to be as thin as possible because the thickness and density of its material influence the image resolution and contrast.

On the other hand, the layers have to be dense and sturdy to withstand complex sample preparation processes. Another advantage of using carbon is that its surface properties can be altered in processes like glow discharge, UV irradiation or chemical treatment. This provides an excellent solution to issues caused by different affinities of molecules to carbon.

1. Required characteristics

- To be as neat and amorphous as possible with no contamination and a low amount of hydrocarbons
- To be sturdy and not to break in floating or replica preparation processes
- In a form of thin film that can be easily floated from mica sheets and placed on TEM grids
- Withstand freezing when used as support for cryo-TEM samples

2. Carbon film quality

The parameters that mostly influence the quality of produced carbon films are the base vacuum during carbon deposition and the purity of the carbon source.

To achieve superior carbon film quality it is necessary to use ultra-pure carbon sources and to ensure a base vacuum of 10^{-7} mbar or better.

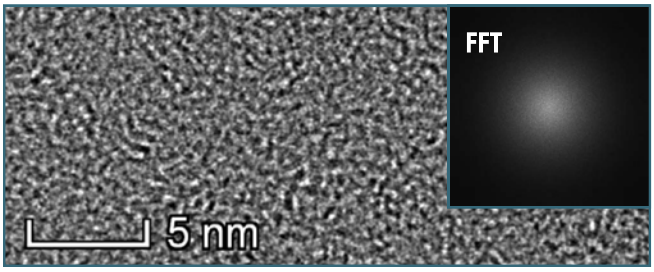

Fig. 1: HRTEM image of 10 nm carbon layer deposited on 50 nm SiN membrane in the base pressure of $4x10^{-5}$ mbar, and the corresponding FFT

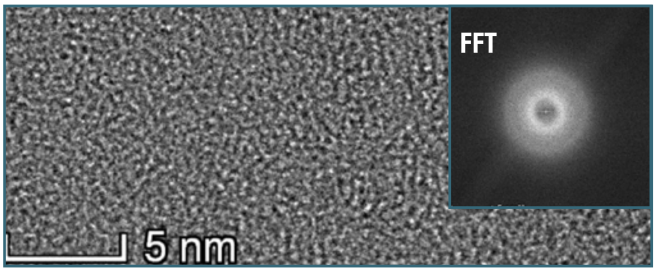

Fig. 2: HRTEM image of 10 nm carbon layer deposited on 50 nm SiN membrane in the base pressure of $8x10^{-7}$ mbar, and the corresponding FFT

The Q150V Plus coater uses the thermal carbon rod evaporation (Bradley method) along with a thickness monitor to produce the high-quality carbon films which are required for a variety of EM applications. Employing high-purity carbon rods allows the deposition of a set thickness and overcomes the source outgassing problem that usually influences purity of the formed layers.

The influence of the deposition base pressure is depicted in figures 1 and 2.
The simplest and most straight-forward test for the mechanical stability of a carbon film is floating the layer from a mica and setting it onto TEM grids. This method is used as one of the steps in sample preparation for TEM imaging. The quality of carbon layers has a direct impact on the final process result.

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3. Base pressure influence on carbon stability

Influence of poor base vacuum on mechanical and electron stability (5 nm carbon film, base pressure $4x10^{-5}$ mbar)

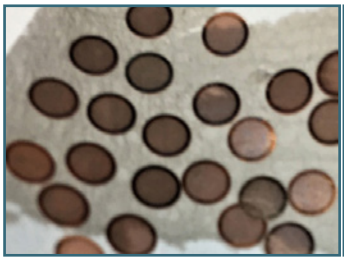

Fig. 3: Layer floated from mica onto TEM grids, visible cracks and flaws of the carbon layer

Fig. 4: Layer floated from mica onto 200 mesh TEM grids, visible cracks and wrinkles, carbon not covering the whole grid

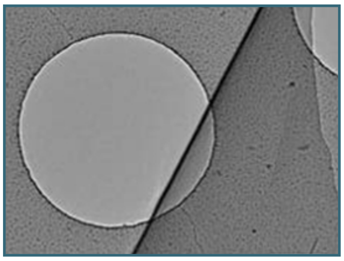

Fig. 5: HRTEM image of layer, floated from mica onto 200 mesh TEM grid, break in carbon layer and an overlap of the layer caused by poor mechanical stability

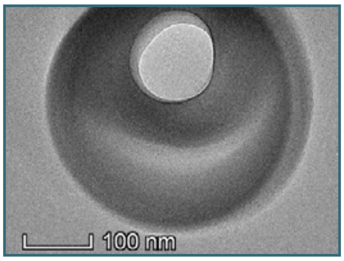

Fig. 6: HRTEM image layer floated from mica onto 200 mesh TEM grid, the amount of hydrocarbons influences the layer stability under the beam – visible burn out

Influence of high base vacuum on mechanical and electron stability (5 nm carbon film, base pressure $8x10^{-7}$ mbar)

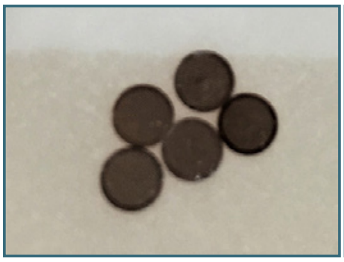

Fig. 7: Layer floated from mica onto TEM grids, neat, sturdy and dense layer

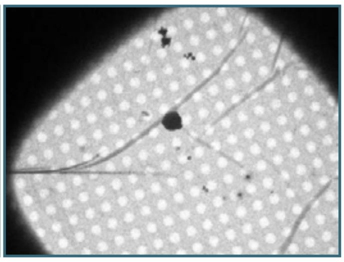

Fig. 8: Layer floated from mica onto 200 mesh TEM grids, with holey carbon TEM grid, neat, sturdy and dense layer covering the whole grid

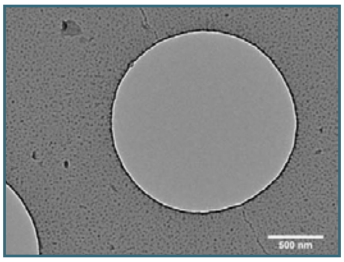

Fig. 9: HRTEM image of layer floated from mica onto 200 mesh TEM grid, sturdy and even layer

A base vacuum of $1x10^{-6}$ mbar or better allows to produce pure, dense and sturdy films that can easily be floated from mica and set onto TEM grids giving very good coverage.
Low number of hydrocarbons and high film purity ensure good electron stability and prevent additional signals in EDS analysis.

4. Influence of high base vacuum on thermal and electron stability

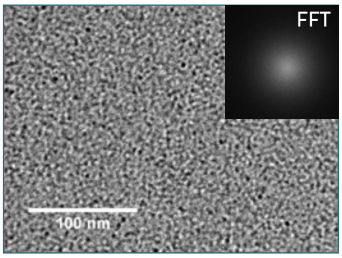

Fig. 10: HRTEM image and corresponding FFT of 200 mesh TEM grid not covered with carbon

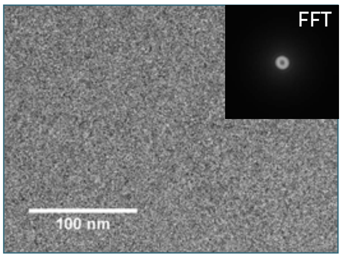

Fig. 11: HRTEM image and corresponding FFT of 5 nm C film produced with base pressure of $8x10^{-7}$ mbar, floated from mica onto 200 mesh TEM grid showing the film quality

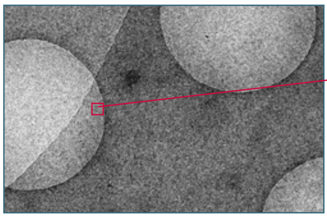

Fig. 13: Cryo-HRTEM image of DNA replication complex placed onto 200 mesh TEM grid with holey carbon and 5 nm additional carbon layer (produced at $8x10^{-7}$ mbar, floated from mica); image showing no flaws of floated carbon film

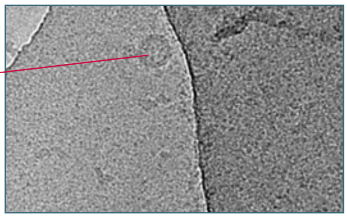

Fig. 14: Zoom-in of cryo-HRTEM image showing DNA replication complex, mag x 92 k

The quality of produced carbon thin films for ambient and cryo-TEM significantly influences sample preparation. Dense, sturdy and pure films made with the Q150V Plus coater will make sample preparation much easier, giving certainty and confidence along the process – from transferring carbon onto TEM grids, post treatment, sample application and finally during imaging.