APPLICATION NOTE

Model 1020 Plasma Cleaner and Model 1070 NanoClean

Plasma cleaning of carbon films for transmission electron microscopy

Transmission electron microscopy of nanoscale materials requires the use of support films deposited on standard specimen grids. These films are often amorphous carbon, and are either free standing or backed by formvar, a polymer composed of hydrocarbons. Plasma cleaning of a specimen grid and side-entry holder eliminates mobile carbonaceous contaminants that may migrate to the vicinity of the electron beam. These contaminants obscure the area of interest during imaging and subsequent analysis.

To slow the degradation rate within the Ar–25% O_2 inductively coupled plasma of the Model 1020 Plasma Cleaner, Fischione Instruments designed a shielded holder port. The shield reduces the plasma density by reducing the cross-sectional area available to the plasma near the holder tip. The shield provides an electrically grounded surface that is concentric with the holder geometry, Figure 1.

To assess the reduction in degradation rate, Quantfoil® holey carbon films backed by formvar were plasma cleaned with and without the protective shield.[1] Without the shield, the mean rate for complete removal of a film was

FIGURE 1.

Shielded specimen holder port.

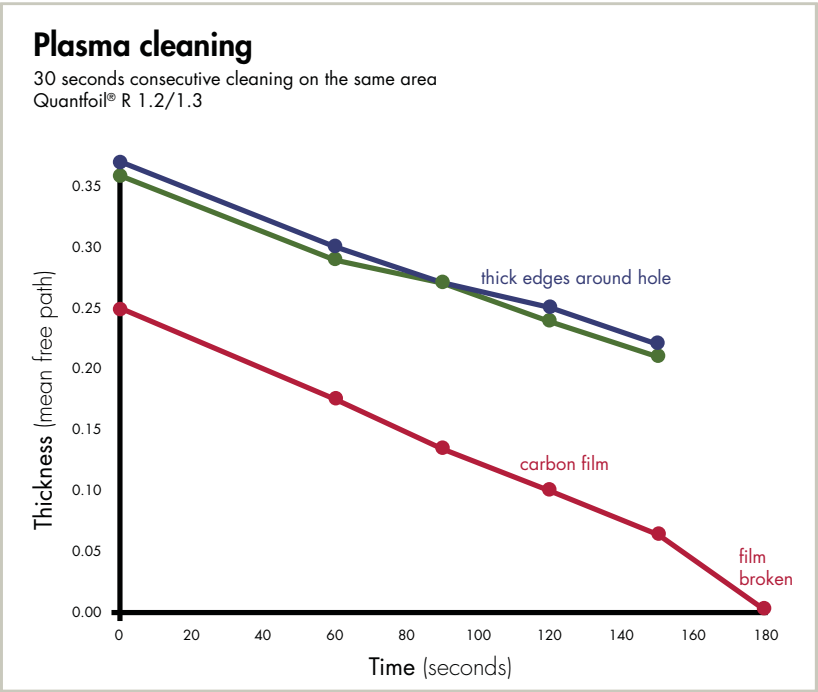

FIGURE 2.

Film thickness (multiplied by mean free path) as a function of plasma cleaning time.

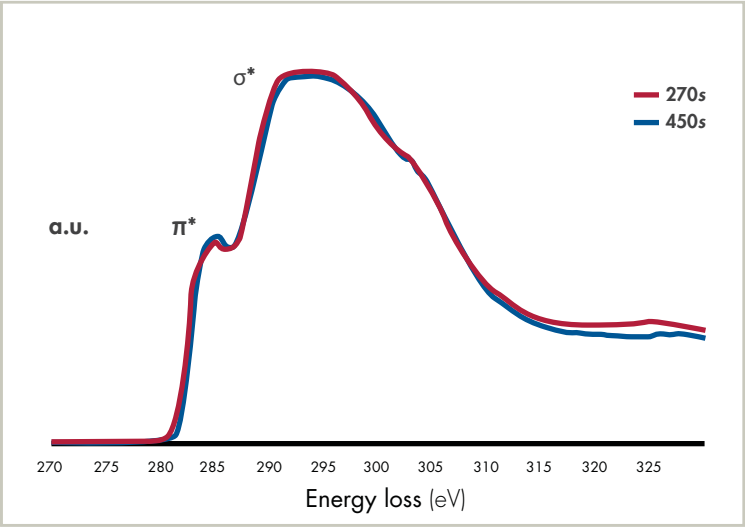

FIGURE 3.

Amorphous film, carbon K-edge spectra after 270*s* and 450*s* of plasma cleaning with the shield. The relative heights of the peaks are constant.

determined as 15.7 nm/min. With the shield, this rate decreased to 2.6 nm/min. In practical terms, the shield expanded the time window for plasma cleaning from less than 2 to more than 10 minutes.

When a support film was cleaned for 30 seconds in consecutive steps and analyzed, electron energy loss spectroscopy (EELS) data was generated as shown in Figure 2. The experiment was ended when the support film was observed to rupture in the FEI CM30 transmission electron microscope. Using a value of 110 nm for the inelastic mean free path (MFP) for carbon at 200 kV, the degradation rate for the carbon film is 7.9 nm/min. After 6 minutes (360 seconds) of plasma cleaning, the film could still be imaged and without recontamination.

EELS was performed on an amorphous carbon film at selected times of plasma cleaning with the shield to determine any change of structure of the film as it was slowly thinned. The carbon K-edge spectrum (above 285 eV of energy loss) shows the transition from the 1*s* occupied electronic state to the unoccupied states above the Fermi level.

The first two peaks represent the transitions to the σ* and π* unoccupied states. These proved constant as Figure 3 shows. The structure of the film is unchanged when plasma cleaned, while no contamination during imaging indicates effective plasma cleaning.

[1]Courtesy of A. L. Hamon, B. Willems, D. Schryvers, EMAT, University of Antwerp, RUCA.

FISCHIONE INSTRUMENTS

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