

CHARACTERIZATION AND APPLICATIONS OF  
ULTRA-FLAT CARBON FILM ELECTRODES  
PREPARED BY ELECTRON-BEAM EVAPORATION

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Carbon materials are commonly employed in electroanalysis, electrocatalysis and electrosynthesis due to a number of advantageous properties. In recent years, attention has been drawn to the fabrication of low-cost, disposable, carbon film electrodes for mass-scale use as electrochemical sensors. Several methods have been reported for the fabrication of thin film ( $\leq 400$  nm) electrodes including chemical vapor deposition (CVD) of carbon containing gases, sputtering, and the pyrolysis of polymeric thin films. In general, these thin film carbon electrodes are highly pure, contain some graphitic microstructure, are easily mass-produced and yield electrochemical reactivity comparable to glassy carbon (GC).

A method will be presented for fabricating high purity carbon films using e-beam evaporation directly onto flat, highly doped silicon wafers. Due to the nature of the deposition and the use of a high-purity carbon source, these e-beam deposited carbon films (ECF) are highly pure. The microstructure of ECFs were examined with Raman spectroscopy before and after pyrolysis at  $1000^{\circ}\text{C}$ . Importantly, non-pyrolyzed ECFs possess near-atomic-scale flatness. To illustrate the topography of the ECF surfaces, Figure 1 presents an assembly of contact-mode scanning force microscopy (SFM) images for 7 nm and 200 nm thick films at image sizes of  $100 \times 100 \text{ nm}^2$  and  $1 \times 1 \mu\text{m}^2$ . Note that the black-to-white vertical scale on these images is only 5 nm. The left column displays images of non-pyrolyzed samples (A, C, E, G), whereas the right column displays the respective images for the post-deposition-pyrolyzed samples (B, D, F, H). The SFM determined root-mean-square (RMS) roughness values of all the films 7 nm and thicker are between 0.7 Å and 1.1 Å. These values are very close to the 0.6 Å RMS roughness measured over both length scales for the underlying silicon  $\langle 100 \rangle$  surface. Pyrolysis of the films results in a considerable increase in roughness at both length scales. This increase in roughness may be correlated with the Raman results demonstrating increased graphitization with pyrolysis; the formation of independently oriented graphitic domains in the films can be expected to disrupt the originally flat surface. Despite the increased roughness, however, the actual values of between 3 Å and 6 Å still represent an exceedingly flat surface.

The electrochemical reactivity of ECFs was also thoroughly characterized and compared to GC and other types of carbon thin films. For example, the cyclic voltammetry in Figure 2 shows that electron transfer rates are similar for ECFs and GC for the  $\text{Ru}(\text{NH}_3)_6^{3+}$  redox system. These easily prepared, ultra-flat films exhibit excellent electrochemical properties and should find uses in a wide variety of applications.

An exciting area of recent interest that requires extremely flat conductive surfaces is solid-state molecular electronics. For example, a common approach to measure

the electrical properties of molecules is to create a *molecular junction* by sandwiching a molecular layer between two conducting surfaces. We are now examining electron transport through aryl layers covalently bound to ECFs with conducting probe atomic force microscopy (CAFM). A junction is made with CSFM by placing a conductive SFM tip (i.e., metal coated) in contact with the carbon-supported layer. Our preliminary results from these experiments will be presented.

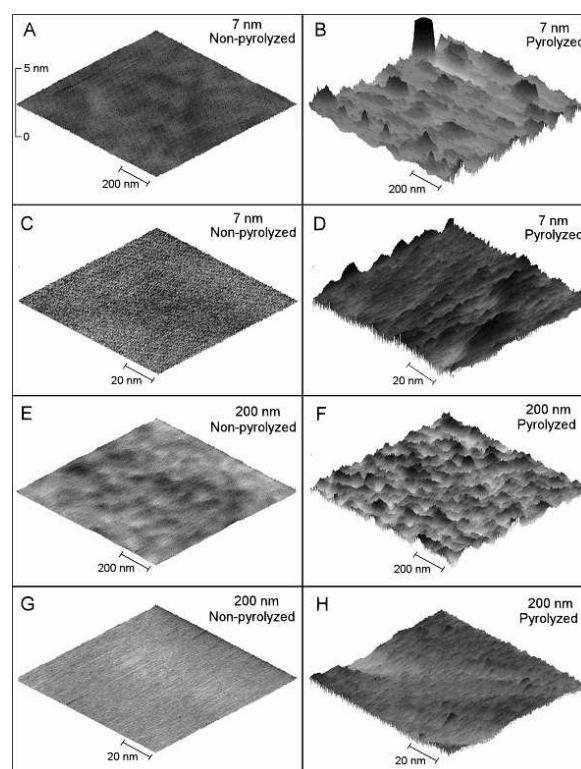


Figure 1: Surface plot presentation of contact mode SFM imaged of ECFs. The z-scale bar in A applies to all images. A.  $1 \times 1 \mu\text{m}^2$  image of a non-pyrolyzed 7 nm film. B.  $1 \times 1 \mu\text{m}^2$  image of a pyrolyzed 7 nm film. C.  $100 \times 100 \text{ nm}^2$  image of non-pyrolyzed 7 nm film. D.  $100 \times 100 \text{ nm}^2$  image of a pyrolyzed 7 nm film. E.  $1 \times 1 \mu\text{m}^2$  image of a non-pyrolyzed 200 nm film. F.  $1 \times 1 \mu\text{m}^2$  image of a pyrolyzed 200 nm film. G.  $100 \times 100 \text{ nm}^2$  image of a non-pyrolyzed 200 nm film. H.  $100 \times 100 \text{ nm}^2$  image of a pyrolyzed 200 nm film.

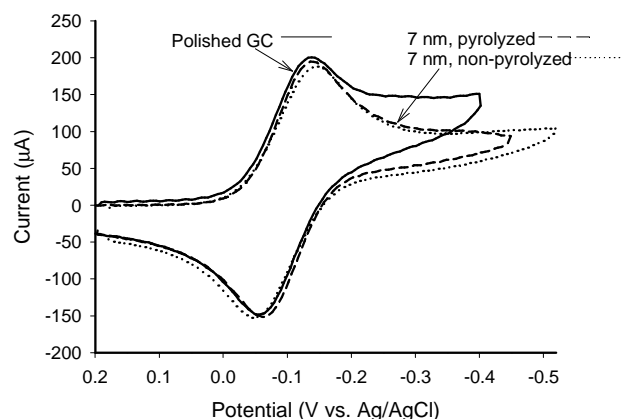


Figure 2: Cyclic voltammetric results for 1 mM  $\text{Ru}(\text{NH}_3)_6^{3+}$  (1 M KCl), on ECFs and GC.  $v = 500 \text{ mV/s}$ .