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## Secondary Electron Emission Study of Annealed Graphitic Amorphous Carbon

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“Secondary Electron Emission Study of Annealed  
Graphitic Amorphous Carbon”

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May 21, 2003

## **INTRODUCTION**

In the 1880s a curious phenomena was observed: when a ray of light, no matter how weak, hit certain metals; electrons were emitted from the surface. Called the “photoelectric effect”, this puzzle was never explained until much later. In 1905, Albert Einstein put forth one possible explanation, which is currently accepted as correct. Einstein proposed that light propagated in discrete energy packets rather than as a continuous wave. While most scientists disbelieved Einstein theory, it was later proved in detail by Robert Milikan.<sup>1</sup> Rays of light traveling in discrete packets hit metal surfaces, depositing energy. If the energy is high enough, electrons will be emitted. Since emission is only dependant on incident energy, electrons and even ions may also be used to deposit energy. Electrons emitted as a result of this energy deposition are called secondary electrons (SE).

Today this process of secondary electron emission (SEE) is well understood, as well as the key factors affecting the magnitude of electron emission. In metals, the work function of the material is the biggest factor. In insulators, it is a combination of bandgap and the threshold energy at the surface of the material. However, in between these two extremes lie semiconductors. Previous work by Neil Nickles at Utah State University sought to establish the role of bandgap in SE yield magnitude in small bandgap semiconductors, specifically graphitic amorphous carbon (g-C).<sup>2</sup> As a standard method of measuring structural disorder<sup>3</sup>, one of the main reasons g-C was chosen was because of the extensive work done with Raman spectroscopy on annealed g-C by Tim Dallas at Texas Tech<sup>4</sup>, providing a check for our annealed samples. The major problem Neil ran into was in the annealing process. Samples of g-C were bound to stainless steel pieces

and then heated with a vacuum oven. However, because of the large thermal expansion coefficient difference of graphitic carbon (g-C) and stainless steel, the samples curled<sup>2</sup> (Fig. 1) making it impossible to



Fig. 1. Curling of annealed g-C.<sup>2</sup>

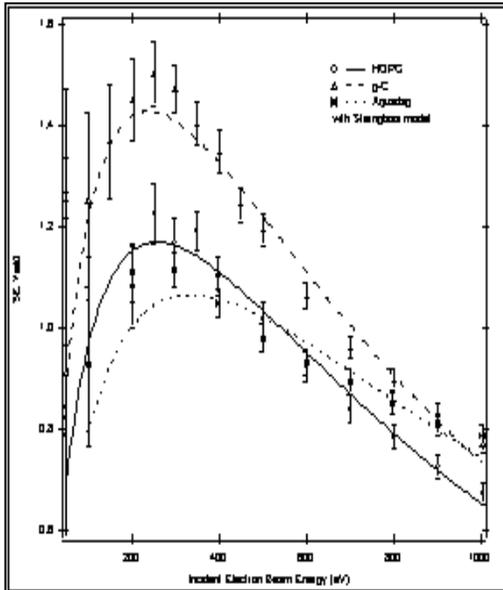


Fig. 2. SE yields of HOPG, g-C, and Aquadag, showing a 30% difference in yields magnitudes.<sup>2</sup>

take data.

Neil was able to

take data on a nonannealed g-C sample (bandgap of  $\approx 0.6\text{eV}$ ); Aquadag, a colloidal graphite (bandgap of  $\approx 0.1\text{eV}$ ); and highly ordered pyrolytic graphite or HOPG (bandgap of zero). A 30% difference (Fig. 2) was shown to exist in the SE yields of g-C and HOPG or Aquadag, which has a similar peak magnitude to HOPG.<sup>2</sup> My

project has been to extend this research: to prepare

and anneal g-C samples, to characterize them, and then to take data on them.

## THEORY

Since metals obviously do not have bandgaps, theory for semiconductor behavior

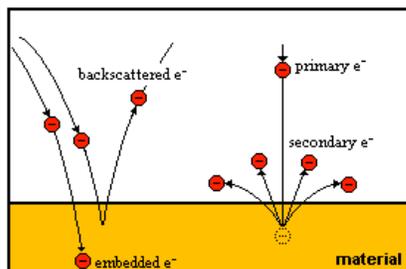


Fig. 3. Possible actions of incident electrons.<sup>5,6,7</sup>

must be derived from insulator theory. To facilitate

explanation, assume an incident beam of electrons.

One of three things may happen to these electrons (Fig.

3). One, they are reflected from the material. Two,

they are imbedded within the material. Or three, they

deposit energy in the material, which in turn excites other electrons.

Since electrons cannot be labeled, a definition cutoff energy of 50eV was used to distinguish those electrons reflected from the material (backscattered electrons) from those emitted as secondaries.

These secondary electrons then travel to the surface of the material, losing energy along the way through various processes, one of which is electron-electron scattering near the conduction band. At the surface these electrons can be emitted if their energy is enough to overcome the threshold energy at the surface. If these secondaries do not have sufficient energy, they continue to travel around within the material. In insulators, enough energy must be deposited for the electrons to be excited beyond the bandgap into the conduction band. However, because of this bandgap in insulators, conducting electrons are effectively shielded from a lot of the electron-electron scattering, thereby retaining more energy so that more electrons are able to escape the material.

## **PROCEDURE**

The first part of this project involved sample preparation. With funding from an undergraduate research and creative opportunities (URCO) grant, thin sheets of graphitic amorphous carbon were purchased from ACF-metals in Arizona. For this project, one sheet of g-C was used, thereby keeping the thickness of each sample uniform. This sheet was carefully sliced with a razor blade into samples of size  $\approx 1\text{cm}^2$ . Instead of using stainless steel sample mounts; Molybdenum was used because of the similarity of its thermal expansion coefficient with g-C. Also, instead of binding the samples to the surface, small copper clips were used to attach the sample to the sample mount. Each sample was then annealed at varying temperatures of 0°C-1050°C in a vacuum oven

pumped with a common mechanical pump. A problem occurred in annealing at 1050°C, however. Attempts to anneal at that temperature resulted in disintegration of the sample. A turbo pump was introduced into the system bringing the pressure down to  $\approx 10^{-5}$  Torr range and allowing annealing at 1050°C.

Once the samples were prepared, they were taken to the Biology Department at USU for scanning electron microscope (SEM) images and contamination measurements using x-ray spectra (Fig. 4-6).

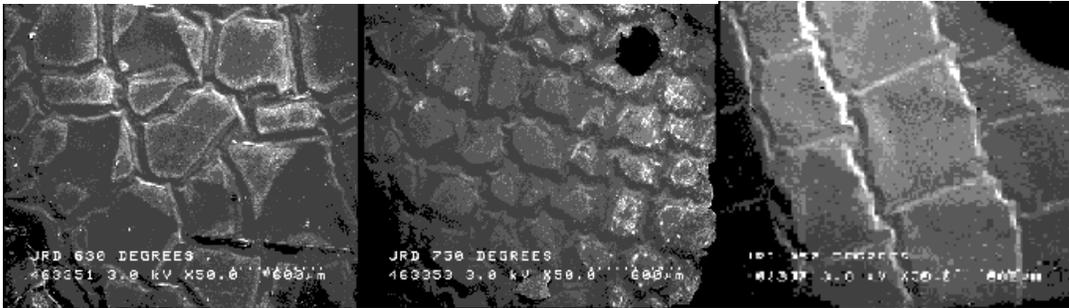


Fig. 4 (left). SEM image of annealed g-C at 630°C.  
Fig. 5 (middle). SEM image of annealed g-C at 750°C.  
Fig. 6 (right). SEM image of annealed g-C at 850°C.

Raman Spectroscopy was then used as a check for annealing. Images were compared to those taken at Texas Tech (Fig. 7,8).

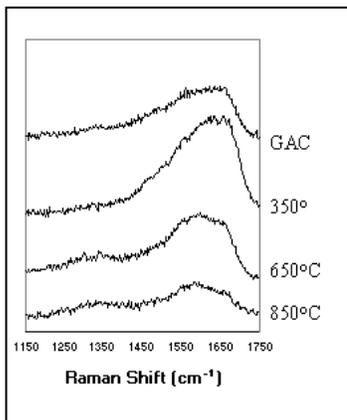
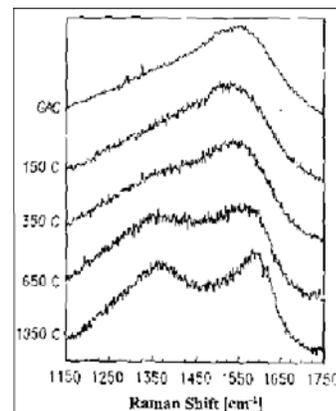


Fig. 7 (left). My Raman spectra.

Fig. 8 (right). Raman data from Texas Tech.<sup>4</sup>



After these preliminary characterization measures were taken, the samples were then prepared for ultra-high vacuum (UHV) measurements. Each sample was mounted on a standard cylindrical copper sample mount with a conducting silver glue made by mixing adhesive with silver paint.

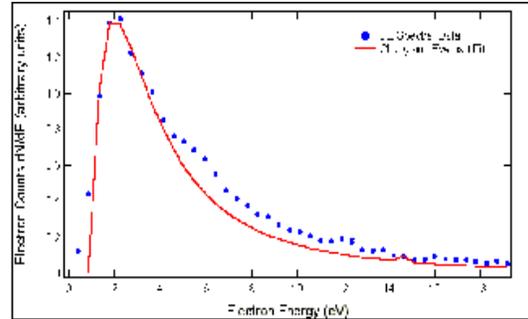


Fig. 9. SE yield of 1050 °C g-C sample

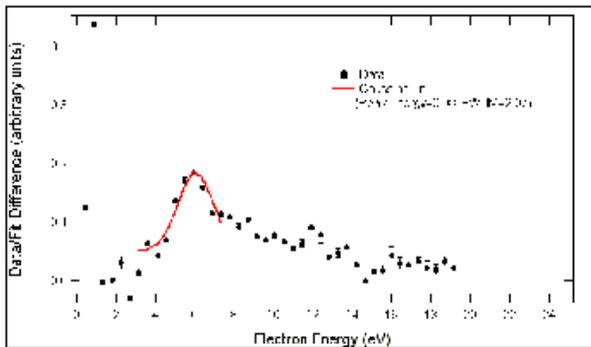


Fig. 10. Difference between SE spectra and fitted curve for 1050°C annealed sample, showing a peak at a few eV below the bandgap.

done by taking an SE yield spectra (Fig. 9), fitting a basic curve to the data, and taking the difference between each point on the two curves (Fig. 10). Spectra from g-C, the 1050°C annealed sample, and HOPG have shown a peak in an energy range just below the bandgap.

Bandgap was determined for each sample using photoyield spectroscopy.

Materials will begin to emit secondary electrons when enough energy is deposited for the electrons to be excited above the bandgap and when those excited electrons have energies

First, contamination was again checked for using Auger spectroscopy. Next, one of the questions that first needed to be answered was whether the annealing process affected the energy density of states below a few eV for each sample, or below the bandgap. This was

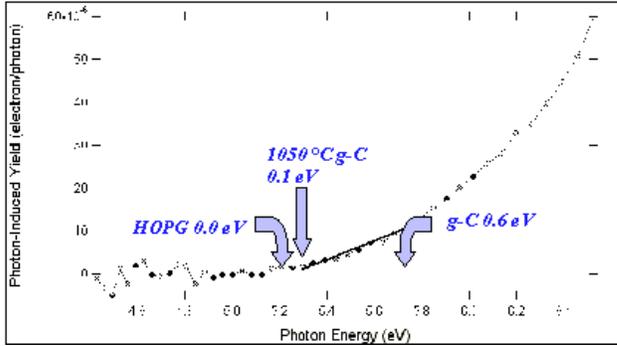


Fig. 11. Photonyield Spectra of annealed 1050°C g-C sample also showing the relative energies of HOPG and g-C.

higher than the threshold energy at the surface. By increasing the energy in the incident photon beam while

measuring for secondary electrons, the energy at which the material begins to emit can be found (Fig. 11). This energy is a sum of the threshold energy and the bandgap. Since each sample is of the same material, the difference in these measured energies is the difference in bandgap. HOPG is conducting and so has a bandgap of approximately zero. The bandgap of the each sample was then calculated from that point (Fig. 12).

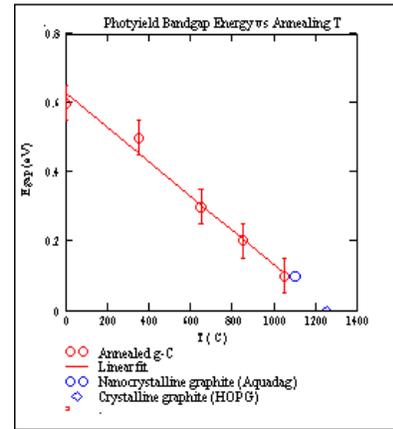


Fig. 12. Plot of calculated bandgap energies of each sample.

Finally, SEE curves were taken on the 1050°C annealed g-C sample and compared to the previous work done by Neil Nickles (Fig. 13).

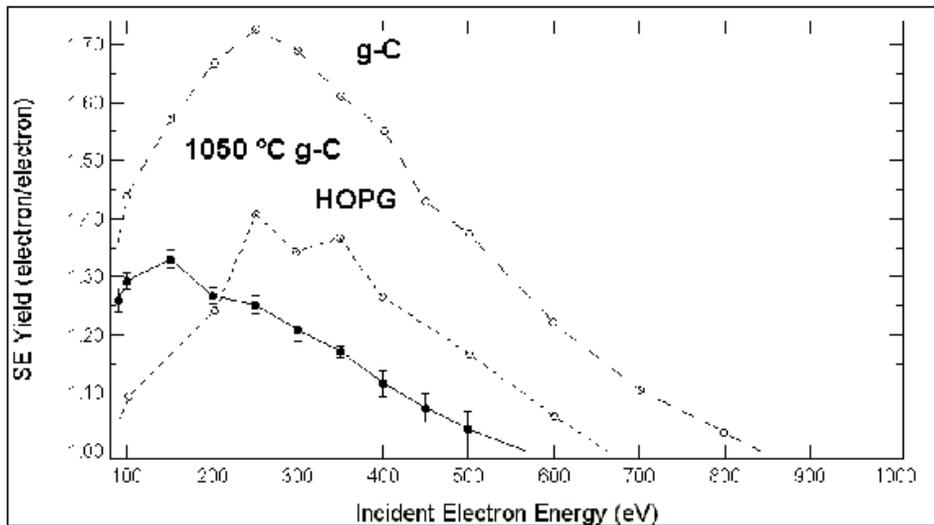


Fig. 13. SE Yield curves for 1050°C, g-C, and HOPG. Peak magnitude of 1050°C sample comparable to that of HOPG.

## **CONCLUSIONS**

Contamination measurements from the Biology Department and Auger spectroscopy showed only minimal contamination. SEM images and optical images showed no serious defects in the sample surfaces. The similarity shown by Neil in SE yield peak magnitudes between HOPG and Aquadag (nanocrystalline colloidal graphite) imply that surface roughness does not play a significant factor in SE yields. SE Spectra show similar density of states for the samples below bandgap energies. The conclusion of these characterizations is that each sample holds other factors constant while only varying bandgap energies.

Bandgap energies were calculated from photoyield spectra and showed a nearly linear trend from nonannealed g-C to the most annealed sample at 1050°C, Aquadag, and HOPG. Calculations also showed that the bandgap energies of the 1050°C sample and Aquadag were identical within  $\pm 0.05\text{eV}$ , thereby implying that the 1050°C sample completely annealed to nanocrystalline graphite.

Unfortunately, initially the samples were not large enough to cover the copper sample mounts. Besides photoyield spectra, the only sample I was able to take data on was the most annealed sample at 1050°C. SE yields for this sample showed a similar peak magnitude to Aquadag, further implying that it completely annealed.

Future work on this project includes annealing new, larger samples; taking data on them, and comparing the results to data already taken. A trend in peak magnitude from the 1050°C sample to the nonannealed sample is expected to be found and awaits further investigation.



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