

# Survey of Post Last Glacial Maximum Environment: Unusual Soil Constituents in Rocky Hock Bay Stratigraphy

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**Abstract-** Throughout North America's eastern coastal plain are found a variety of features attributed to ice age climate. These include many elliptical, shallow depressions collectively called Carolina Bays, hypothesized to have been formed by the strong, sustained winds and arid, cold climate characteristic of glacial epochs (Raisz, 1934, Johnson, 1942 and Kaczorowski, 1977). This view eclipsed the 1933 proposition by Melton and Schriever, and expanded by Prouty (1934, 1953), that extraterrestrial debris produced by an aerial meteorite or comet explosion in the vicinity of the Great Lakes during the late Pleistocene formed the bays. Recent discovery that a number of the bays were found to contain material associated with extraterrestrial impacts including carbon and magnetic spherules, glass-like carbon, charcoal and nanodiamonds reinvigorated the debate over the bay's origins [4].

To confirm the bays were receptacles for impact material; soil samples were previously taken from Rocky Hock Bay in Chowan County, northeastern North Carolina at (36°10'N, 76°41'W). Sequential soil samples were excavated near the bay's center and core samples extracted near the bay's rim. The samples were examined to determine the presence of carbon-associated markers and to measure the density of magnetic grains and grain-size distribution. Magnetic spherules were found among the smaller size portions of the magnetic grains and spherule density estimated. The geochemistry of a magnetic spherule was determined using scanning electron microscopic energy dispersive x-ray spectroscopy (SEM-EDS).

**Keywords:** last glacial maximum, pleistocene-end, Carolina Bays, slurry, stratigraphy, microtektite, scanning electron microscope

## I. Introduction

Evidence for the harsh climate prevalent during the Last Glacial Maximum (LGM) are seen in topographical features visible south of the ice sheet margin in the uplands and coastal regions of the eastern United States.

Among these are many elliptical, shallow depressions called collectively Carolina Bays, hypothesized to have been formed by "blow outs" of loose sediment by the strong, sustained winds and arid, cold climate

characteristic of glacial epochs (Raisz, 1934, Johnson, 1942 and Kaczorowski, 1977).

12,900 years ago, post-LGM warming was interrupted by a return to a glacial climate that persisted for over 1,000 years. The events precipitating the cooling, known as the Younger Dryas (YD), are the subject of debate. Recently Firestone proposed that an impact in the Laurentide ice sheet by a fragmented comet might have simultaneously initiated the YD and formed the Carolina Bays [4]. Carbon 14 dating and pollen analysis of core samples taken from Rocky Hock Bay (RHB, in Chowan County, NC, by Whitehead [5] indicate a pre-YD genesis. However, a number of the bays have been found to contain material associated with extraterrestrial impacts including carbon and magnetic spherules, glass-like carbon, charcoal and nanodiamonds [4]). The discovery reinvigorated the debate over the bay's origins.

If created before the YD, the bays would have experienced episodic post-formation modification due to cold, dry, windy periods alternating with warm, moist and calmer climatic conditions. Carolina Bays would thus episodically fill with wind-blown or water-borne sediment or water. Some evidence of bay history should be evident in their stratigraphy. Whitehead's correlation of depth to date at Rocky Hock Bay, shown in figure 1, makes it possible to establish a chrono-stratigraphic context for potential impact markers found in bay sediments and also provide an opportunity to confirm Whitehead's inferred bay-structure and age.

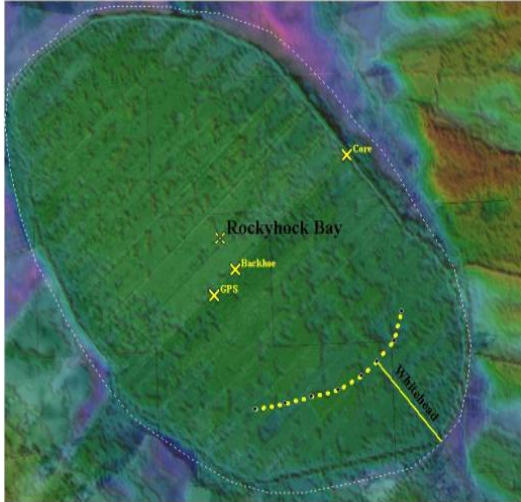


Figure 1. Shows where Whitehead took samples to correlate depth to date in Rocky Hock Bay. Also shows where the URE OMPS 2009 Gambit Team collected their coring and backhoe samples [1].

## II. Procedures

### A. Materials

The following items were used for analyzing and examining the soil samples (Fig. 2).

- Hefty qt-capacity Storage Bags
- NdFeB-N50 Magnet
- Soil Samples
- Artist's Brushes
- 12 L & 2.5 L Containers
- 1.8 mL Glass Vials
- 20  $\mu$ m Paper & Gold- Screen Filters
- AWS DIA-10 1 mg scale
- Digital 1 gram Scale
- Keck Sand-Shaker Sieve
- AmScope SM-2BZ 7-180 x trinocular Microscope



Figure 2. Some of the materials used for the analysis of the soil samples.

### B. Soil Samples

Soil samples from Rocky Hock Bay (RHB) located in Chowan County, NC at (36°10'N, 76°41'W) were previously collected by the URE OMPS 2009 Gambit Team [1]. This was done by using a backhoe and coring to take samples from the center and rim of the bay respectively. The bay rim-core samples that were studied were collected in 15.2 centimeter (cm) intervals (only with the core samples taken from near the bay rim).

The same team performed a Ground Penetrating RADAR (GPR) survey of the semi-minor axis of the bay. GPR results indicated the core sample was actually taken in a swale between concentric bay-rims. The sediment from bay center were taken in 5 cm increments.

The Bay rim-Core samples examined were stored in plastic bags, and mixed for homogeneity with a spoon. 300-gram aliquots of each sample were extracted from the total one to two kilogram sample and mixed with water in a 12 L containers to create a slurry.



Figure 3. Shows the weighing of a sample to 300 grams.

### C. Extraction of Carbon Spherules, Glass-like Carbon, and Charcoal

Charcoal, glass-like (or vitreous) carbon, and carbon spherules are all considered to be evidence of biomass burning. These markers have low-densities, so they are able to float. The slurry was stirred around to free any of the floating fraction. Then the water from the slurry was

repeatedly exchanged through a 20 micron-mesh filter between two buckets (Fig. 4). This process was completed when no more of the floating fraction was visible on the surface of the slurry. The filter contents were then placed on a plate to dry to later be examined using an optical microscope (Fig. 5).



Figure 4. Shows the slurry being filtered through a 20-micron mesh filter.



Figure 5. Shows two filters containing the floating material extracted.

#### *D. Extraction of Magnetic Grains*

Magnetic material is also present within these soil samples. Unlike the carbon-associated markers, they tend to sink to the bottom since they are denser. To extract the magnetic grains, a super magnet was placed into a one quart plastic bag. The bag was then inserted into the slurry and moved slowly around to catch any of the magnetic grains (Fig. 6). Magnetic grains attracted to the magnet were then transferred to a smaller bucket of water. This was done by removing the magnet from the

immersed bag, so that the magnetic grains would be released from the bag's outer surface and fall into the container. This process was repeated 14 times (each consisting of a 45 sec. immersion periods) for consistency. The magnetic extraction process was repeated using a second small bucket containing clear water to perform a cleansing rinse of the grains (Fig. 7). The water from the rinsed magnetic fraction was poured through a 20  $\mu$ m coffee filter to capture the smallest grains. The larger heavier magnetic grains rested at the bottom of the bucket, while the lighter grains supported by surface tension were caught in the filter as the water was decanted. The heavier magnetic grains were set aside to dry, and the filter was placed on a plate to dry also. After they dried, they were combined and put into a glass vial and the mass was measured. Density of the magnetic grains in grams per kilogram in each aliquot was calculated. Stratigraphic profiles were also constructed to determine any peaks in the density.



Figure 6. Shows the magnet being slowly moved around the slurry.



Figure 7. Demonstrates the process of rinsing the magnetic grains.



### *E. Size Sorting of Magnetic Grains*

When a peak in bulk magnetic grains was found, the magnetic grains were divided into size portions based on their diameter ( $d$ ):  $d > 250 \mu\text{m}$ ,  $53 \mu\text{m} < d < 250 \mu\text{m}$ , and  $d < 53 \mu\text{m}$ . This process was also done on those sample depths above and below the peaks. The size sorting was done using a Keck Sand-Shaker Sieve with appropriate American Society for Testing and Materials (ASTM) standard grid sizes (Fig. 8). The three size portions were then put into separate glass vials and measured for mass.



Figure 8. Size sorting of magnetic grains.

### *F. Detection, Photography, and Extraction of Magnetic Spherules*

Petri dishes containing 10-30 milligrams (mg) of magnetic grains were scanned under an optical microscope. The scanning magnification was at 100-130x. When a suspected spherule was found, the magnification was zoomed to its maximum at 180x. A photograph was taken of the possible spherules with a 3 megapixels digital camera (Fig 9). After the photo was taken, a one-stranded artist's brush was used to retrieve the spherule from the Petri dish. To extract the spherule, saliva was applied to the brush's tip. Once the spherule was obtained, it was affixed to a double-sided adhesive surface that was attached to a scanning electron microscope mount (stub) (Fig. 10).

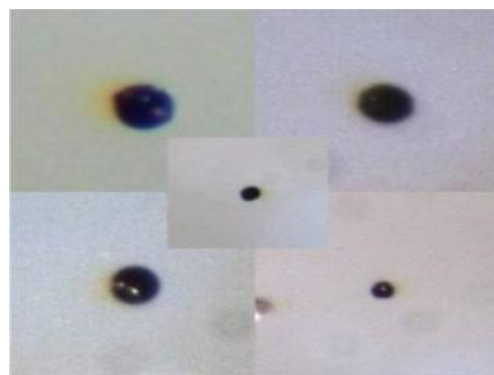


Figure 9. Photos of possible magnetic spherules



Figure 10. SEM stubs containing possible magnetic spherules.

### *G. Chemical Analysis of Magnetic Spherules*

For chemical analysis of the magnetic spherules, the stubs were taken to the Analytical Instrumentation Facility at NC State University. The spherules were examined by a Hitachi S-3200N Scanning Electron Microscope (SEM). The chemical composition of the spherules was determined by the energy-dispersive X-ray spectroscopy (EDS) (Fig. 11).

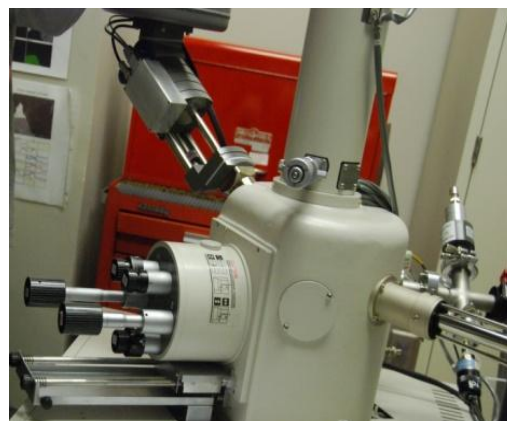


Figure 11. Hitachi S-3200N SEM at the NC State's Analytical Instrumentation Facility

### III. Results

There were four peaks in the bulk magnetic material: (1) 61 cm, (2) 91.5 cm, (3) 121.9 cm, and (4) 152.4 cm depths (Fig. 12 & Fig. 13). Each of the peaks were ~ 30.5 cm apart. Each peak was size sorted and its mass was measured and density calculated. The ratio of the smallest size fraction's density to that of the sum of the three size portions was calculated and tabulated as a percent of the total (Fig. 14). The largest bulk peak was found in sample 61 cm depth. The smallest grains ( $d < \mu\text{m}$  53) were most abundant in the core-sample from 121.9 cm depth. The largest bulk peak was found in sample 61 cm depth. Suspected spherules were founded in 61 cm and 121.9 cm depths, but not in any of the depths surrounding them. However, only the sample at 121.9cm depth revealed the presence of magnetic spherules at an abundance of about 60 spherules/kilogram.

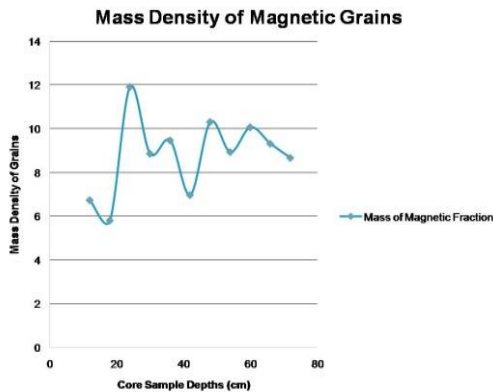


Figure 12. Mass density of all soil depths analyzed.  
\*Note: There are four bulk grain peaks.

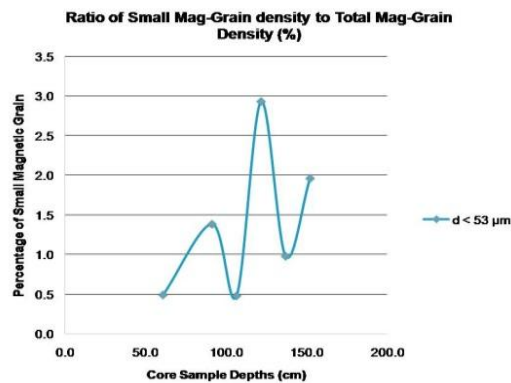


Figure 13. Mass density percent of small magnetic grains from the samples containing peaks and those surrounding them.

Peak Depths (cm)	Mass of Magnetic Grains (g/kg)	Mass of Smallest Magnetic Grains ( $d < 53 \mu\text{m}$ ) (g/kg)	Percent of Smallest Magnetic Grains
61	11.914	0.057	0.48%
91.4	9.473	0.127	1.34%
121.9	10.313	0.297	2.87%
152.4	10.070	0.197	1.96%

Figure 14. Table of the bulk magnetic grain peaks and their percentages of the smallest grain size.

The EDS showed that the geochemical composition of the 32  $\mu\text{m}$  magnetic spherule found in the 121.9 cm depth: oxygen (29.9%), silicon (16.54%), aluminum (14.83%), iron (3.05%), magnesium (0.42%), calcium (1.02%), and potassium (0.52%), as shown in figure 15. There were trace amounts of other elements found (Fig. 16).

Geo-chemistry of Magnetic Spherule (48")	
Elements	Percent (%)
Oxygen	29.9
Silicon	16.54
Aluminum	14.83
Iron	3.05
Magnesium	0.42
Calcium	1.02
Potassium	0.57

Figure 15. Table of the composition of the magnetic spherule from 121.9 cm sample depth.

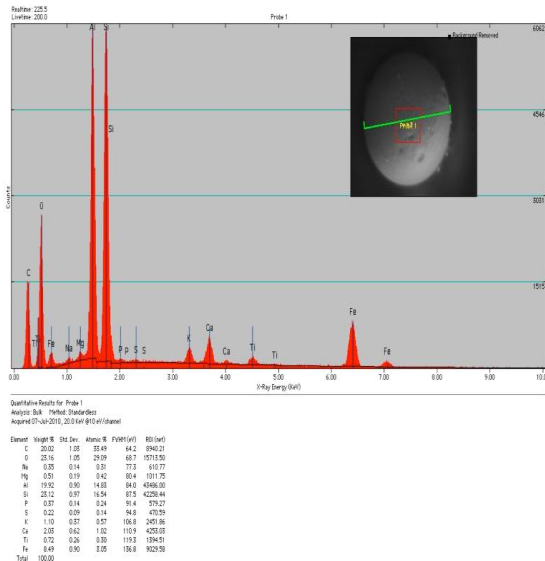


Figure 16. EDS of magnetic spherule found from 121.9 cm depth.

The Rockyhock bay spherule was found to possess similar soil constituents to spherules found in other Carolina Bays, (Fig. 17). There were small amounts of all the markers found in RHB, except for carbon spherules. Since no carbon spherules were found, no tests were run for the presence of nanodiamonds.

Markers	RHB	Other Bays
Amber Spherules	small amounts	varying amounts
Carbon Spherules	none	varying amounts
Charcoal	small amounts	varying amounts
Glass-like Carbon	small amounts	varying amounts
Nano-diamonds	untested	varying amounts
Magnetic Grains (Peak)	multiple peaks	single & multi-peaked
Magnetic Spherules	small amounts	varying amounts

Figure 17. Comparison of the soil constituents found in RHB to other bays.

#### IV. Conclusion

There was no significant indication of biomass burning beyond small fragments of charcoal and glass-like carbon. This can be attributed to the geography of Rocky

Hock Bay, which was probably a lake during the late-Pleistocene. Therefore, there was mostly water present and no biomass-forest or trees. Also, multiple bulk magnetic grain peaks were found in 30.5 cm intervals beginning at 61 cm. There is only one significant small grain diameter that produced magnetic spherules, which was at 121.9 cm. Firestone concluded that peaks found in bulk grain materials indicated the YD layer [4]. However, it was discovered that the peaks in size  $d < 53 \mu\text{m}$  spherules showed the true layer.

The SEM confirmed the grain's spherulitic nature and the EDS results indicated similar geo-chemistry to other spherules taken from distant sites. Spherule chemical composition appears to be very similar to that of the Earth's crust but not of either volcanic or anthropogenic or biogenic origin. The chemistry appears to be very similar to microtektites found around the world and attributed to extraterrestrial impact (Glass, 1974 and Koeberl, 1986).

#### V. Future Work

Soil samples analyzed from Rock Hock Bay contained soil constituents that correlate with an extraterrestrial impact. Further research should be done by taking new core samples from just inside of the bay. It is also suggested that core samples at 7.6 cm intervals beginning at 61 cm to 167.6 cm to confirm the YD impact layer and obtain more accurate results.

The URE OMPS 2009 Gambit Team previously took and analyzed soil samples from Sandra Kimbel Bay (SKB) [1]. It is recommended that processed samples from (SKB) with peaks in bulk magnetic grain density are size sorted ( $d > 250 \mu\text{m}$ ,  $53 \mu\text{m} < d < 250 \mu\text{m}$ , and  $d < 53 \mu\text{m}$ ) and calculate the percentages of each. Afterwards, analyze the sample with the peak in  $d < 53 \mu\text{m}$ . Any possible spherules found should have the same SEM-EDS work that was done on the spherules found in RHB. In doing so, the spherulitic nature and chemical composition of the possible magnetic spherules can be determined. Afterwards, those magnetic spherules should

be compared to spherules from other impact sites.

Future Lacustrine-history bays should be surveyed and soil analyzed for impact markers including: charcoal, carbon spherules, glass-like carbon, and magnetic spherules. If magnetic spherules are found, SEM-EDS should be performed. If carbon spherules are found, Transmission Electron Microscopic (TEM) analysis should be performed to reveal the presence of nanodiamonds should be performed.

## VI. Acknowledgments

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## VII. References

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