Using Nanoindentation to Investigate the Effect of Manufacturing Pressure on the Microstructure and Stiffness of Pyrolytic Carbon

Nicholas Landry
University of New Hampshire

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research article

Using Nanoindentation to Investigate the Effect of Manufacturing Pressure on the Microstructure and Stiffness of Pyrolytic Carbon

—Nicholas Landry (Editors: Michael Pope and Clia Goodwin)

During my first semester at the University of New Hampshire in the fall of 2010, several people I knew were starting to get involved in undergraduate research, and this piqued my interest. At the urging of a faculty member, I visited the Hamel Center for Undergraduate Research and met the director, Dr. Paul Tsang, who informed me of the Research Experience Apprenticeship Program (REAP) for first-year honors students interested in research. As a mechanical engineering major, my primary interest was the field of materials science; and I knew that the chair of the Department of Mechanical Engineering, Dr. Todd Gross, specialized in materials science. Professor Gross was willing to mentor me as part of a three-year project he was performing in collaboration with the Karlsruhe Institute of Technology (KIT), a top university in Germany.

With Dr. Gross' help, I was awarded the REAP grant. Thus, in the summer of 2011, following my freshman year, I started working in UNH's Scanning Probe Microscopy Lab with Nikolay Timoshchuk, a graduate student who was nearly finished with his master's research.

The author and his mentor, Professor Todd Gross, inspecting the nanoindenter in the Scanning Probe Microscopy lab at UNH.

UNH and KIT had a jointly sponsored project to model the mechanical behavior of carbon-carbon composites. Dr. Gross' portion of that project was to characterize the mechanical behavior of the different components in these composites. In specific, Dr. Gross was investigating the behavior of the composite composed of a carbon fiber felt and pyrolytic carbon (Pyro-C). My work was the final part of his investigation: I was testing the stiffness of three different samples of this composite, using a technique known as nanoindentation, in order to correlate stiffness with the conditions under which the samples were manufactured.

Microstructure and Manufacturing of the Carbon-Carbon Composite

Carbon-carbon composites have high heat resistance, durability, and high strength-to-weight ratio, which have led to numerous high performance applications in the aerospace industry (Timoshchuk, 2011). Carbon-carbon composites appear in various applications: heat shields on the underbelly of space shuttles, the exhaust nozzles of space shuttles, and the brake pads and rotors for high performance aircraft. (Fig.1)

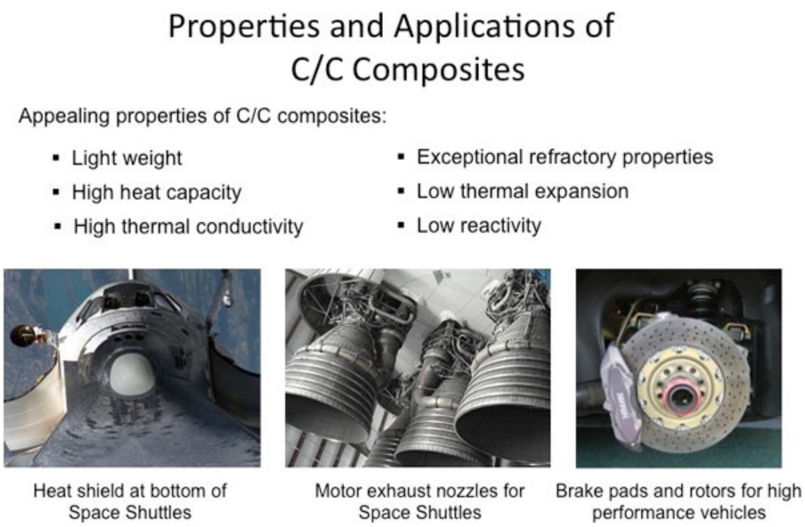

Figure 1: Current properties and applications of carbon-carbon (C/C) composites. (Courtesy of Todd Gross)

The carbon-carbon composite I tested is made of a carbon fiber felt and a matrix material made of Pyro-C, which fills the spaces between the fibers. (The carbon fibers are about twelve microns in diameter, roughly seven times smaller than an average human hair.) These composites are manufactured by placing a block of carbon fiber felt in a furnace, heating it to about 1200-1500 degrees Celsius, and pumping either propane or methane gas into the furnace. The high heat breaks down the gas into carbon and hydrogen atoms, its constituent elements, and the free-floating carbon atoms bond to each other and build up in ordered sheets around the randomly oriented felt fibers. These sheets are graphene, a layer of carbon one atom thick with the constituent atoms arranged in a hexagonal pattern. These layers change in orientation to one another as they build up on the carbon fibers.

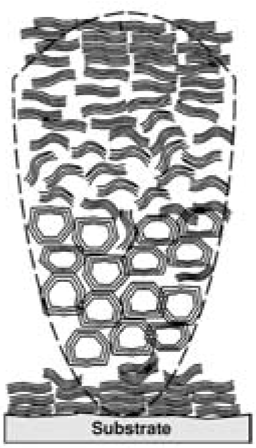

Figure 2: Variations of the microstructure in growth cones. Highly ordered layers at the top of the structure become more disordered closer to the carbon fiber (substrate) before resuming the highly ordered structure at the fiber surface. (Courtesy of Boris Reznik)

During the deposition of the graphene sheets in layers around the fibers, the microstructure of the Pyro-C matrix varies. The schematic of Figure 2 shows a highly ordered layer on the surface; however, as the graphene sheets continue to deposit, onion-like structures, or growth cones, form before transitioning to a less orderly layered structure. These growth cones are a repeated structural phenomenon visible throughout the pyrolytic carbon. Pores, or voids, in the carbon-carbon composite are formed when small parts of the composite are sealed off before the gas has finished filling up all the spaces with Pyro-C. The pores have different shapes and comprise about 30% of the volume of the composite. (Fig. 3)

The stiffness of the pyrolytic carbon depends on the resulting structure, specifically on how highly ordered the graphene layers are. The three samples of the composite I tested were manufactured at KIT under differing pressures. We measured the stiffness of each sample using nanoindentation.

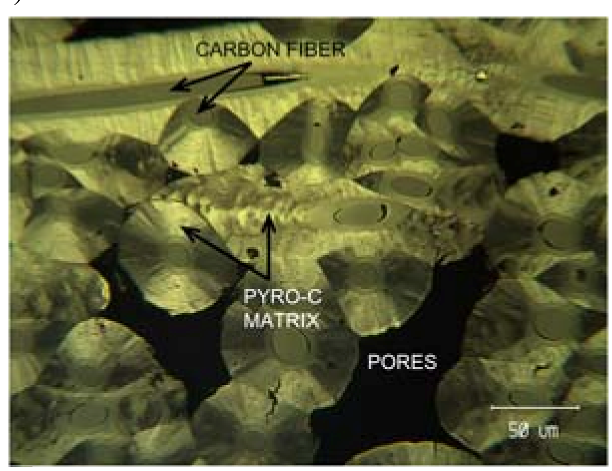

Figure 3: A micrograph of the structure of the carbon-carbon composite tested, showing the Pyro-C matrix surrounding the carbon fibers and the pores that are formed during deposition.

Testing Stiffness with Nanoindentation

Indentation is conducted by pressing a pyramid-shaped diamond tip into a material. Similarly, nanoindentation is indentation on the nanoscale, a billionth of a meter. Nanoindentation is useful for making measurements of the material properties in very precise locations on the composite (within 1/1000th of a human hair).

We tested the three carbon-carbon samples, each manufactured at a different pressure. Because different pressures during manufacture change the microstructure of the Pyro-C, we expected to see changes in stiffness among the samples. We used two different indenter tips to measure the stiffness as a function of distance from the edge of the fiber and also from two directions relative to the carbon fiber. The first direction was longitudinal (parallel to the axis of the fiber) and the second direction was radial (perpendicular to the axis of the fiber).

The stiffness of the sample is measured by how far the tip penetrates the sample for a given force and how fast the material springs back (Cornell, 2011). Because of the scale on which this test is conducted, two computers control the indenter tip: one controls the force exerted, the other controls the location. The information is combined to correlate the stiffness with the location. The table that the material sample sits on can move back and forth, enabling the tip to scan the surface and generate a topographical map that covers at most the area of a human hair, or about 100 microns. This map of the sample allowed me to precisely position indents. This is helpful for testing how stiffness changes across the material. Most materials have a uniform stiffness, so differing stiffness in a material is relatively unusual.

When a material has identical stiffness in all directions, the stiffness measured by indentation is constant regardless of indenter tip. This is not the case if a material is anisotropic, which means that its characteristics, such as stiffness, are dependent on the orientation of the material. Anisotropic materials have different stiffnesses when measured from different directions, and the degree of anisotropy reflects the difference in stiffness for two different directions. Pyrolytic carbon, unusually, is anisotropic, and its degree of anisotropy depends on its manufacturing pressure.

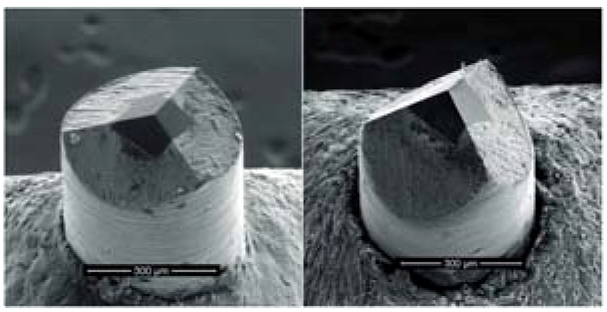

Figure 4: The Berkovich indenter tip (left) and the Cube Corner indenter tip (right). (University of Nebraska-Lincoln, College of Engineering)

I used two different indenter tips to make the stiffness measurements on the samples: Berkovich and Cube Corner. (Figure 4) The Berkovich tip has a shallow pyramidal shape similar to a sledgehammer and measures the stiffness of only one direction because its blunt shape is able to measure only the vertical stiffness in reference to the sample of the composite being tested. In contrast, the Cube Corner, exactly the shape of a corner of a cube, is very sharp and tends to push laterally also when it pushes down, in the way a wedge pushes apart a log. Because the Cube Corner's sharper profile exerts force both laterally and vertically on the composite sample, an anisotropic material will respond to it differently than when force is applied by the Berkovich tip. This difference can offer additional information about the behavior of the material's microstructure. We compared stiffness measurements from the two tips to observe the materials' responses to the differing shapes and sharpness of the tips.

Results

We noticed a discrepancy between the two stiffness measurements from Cube Corner and Berkovich when measuring in the longitudinal direction. We observed that the pyrolytic carbon manufactured at low pressures exhibited behavior that can be explained by the buckling of the highly oriented sheets of graphene. We did not observe this effect with the materials manufactured at high pressures because the graphene sheets were not highly oriented.

The biggest achievement of my research was my indentation of pyrolytic carbon in the radial direction. No other researchers have made this measurement. This accomplishment helps the scientific community better understand this carbon-carbon composite. This knowledge will, we hope, be applicable to other materials as well.

Throughout the summer, Professor Gross and Nikolay gave me guidance about what the research process was like. They taught me to focus on interpreting the results, not on achieving a perfect data collection, because materials don't behave with perfect consistency. I also learned how to use academic papers to inform and reinforce my research. Lastly, I learned the importance of patience because research always takes longer than you think it will.

I would like to thank Dr. Todd Gross for his willingness to mentor me and let me conduct research under his supervision. Nikolay Timoshchuk was invaluable in showing me how to conduct the experimentation and operate the instruments. Thanks also to the Hamel Center for Undergraduate Research for their generous grant, which made my summer research possible.

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Author Bio

*As a curious freshman, **Nicholas Landry**, from Barrington, New Hampshire, sought out the Hamel Center for Undergraduate Research hoping to become involved in a research project in his mechanical engineering major. His initiative paid off, and he was awarded a Research Experience and Apprenticeship Program (REAP) for the 2011 summer. For Nicholas, the opportunity to write an article for Inquiry has not only helped him advance his own knowledge of materials science, but has allowed him to share it with Inquiry's wide range of readers. In the future, Nicholas plans to pursue a Ph.D. in materials science before teaching at the university level or leading research at a national laboratory. In the meantime, aside from studies and lab work, he lifeguards, plays squash, and tutors math.*

Mentor Bio

*Professor **Todd S. Gross** is currently completing his twenty-fourth year at the University of New Hampshire, where he is professor and chair of the Department of Mechanical Engineering. His special area of research is the mechanical behavior of materials, with a focus on experimental measurements. While Professor Gross has worked with many undergraduate students on research, it is his first experience as a Research Experience and Apprenticeship Program (REAP) mentor. He enjoyed working with Nicholas, he said, who "was an exceptionally motivated student." Professor Gross likes being able to balance both teaching and research, and views writing in general as a useful exercise for helping students organize their thoughts and critically evaluate the concepts and results they are trying to explain.*