NANOINDENTATION DETERMINATION OF ELASTIC MODULUS AND HARDNESS OF PYC SAMPLES

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Introduction

Pyrolytic carbon (PyC) is used as the principle material in the fabrication of the majority of mechanical prosthetic heart valves. The heart valve components are fabricated by depositing carbon onto graphite mandrels or substrates within a fluidized bed CVD reactor, and subsequently machining the parts to their desired specifications.

In an effort to explore the relationships between characteristics of PyC and positions within the reaction chamber, samples of PyC were fabricated at specific locations within a PyC coater and subsequently analyzed. The samples were examined visually; densities, modulus, and hardnesses were also determined.

Experimental

The CVD reactor and the process employed has been described previously [1]. Graphite rods were suspended parallel to the CVD coater's central axis at the coater center (R1) and at a radial position near the coater wall (R2). The rods were coated using propane as the source gas. Six rods were coated in separate runs; three rods at each radial position.

The coated rods where cut at ½" intervals and these samples were prepared metallographically so as to expose a cross section of the coating corresponding to positions within the coater; samples were also retained for density analysis.

Photographs of the samples were obtained using a Nikon Epiphot inverted micrograph under bright field conditions, using the polarizer and Nomarski filter; typical magnifications were 50X and 500X. Density measurements were made using a sink-float method. The elastic moduli and hardnesses were determined using the NANO Indenter® II; the nanoindentation method has been described previously [2].

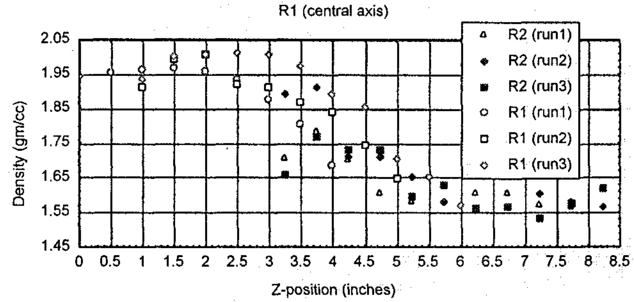
Results

The micrographs revealed the presence of a smooth and homogeneous material (similar in appearance to that described as isotropic) and a rough, dark, and porous material (granular) [1]. The homogeneous material is present on samples fabricated at R1, at low Z-positions (1.0 to 2.0 inches above the source gas inlet). At higher Z-positions, granular material begins to appear as rings, which are concentric with the graphite core. Above about 4.5 inches, the samples are comprised mostly of granular PyC.

Samples prepared at R2 were comprised of both homogeneous and granular PyC. In general, higher magnification micrographs revealed that homogeneous PyC contains only a small amount of porosity and granular PyC contains considerable porosity.

The density determinations are shown in Figure 1.

FIGURE 1
Densities and Z-positions
R2 (radial position near wall)
R1 (central axis)

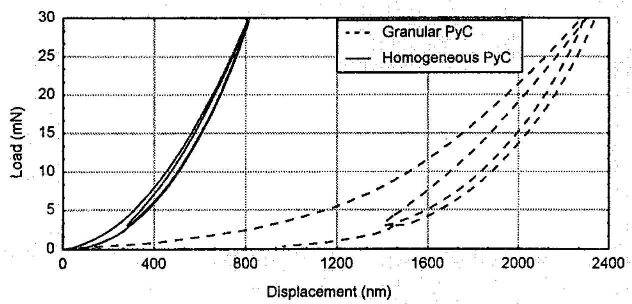


When performing the modulus and hardness determinations on the R1 samples, indentations were made only in regions of homogeneous PyC[1], which is some cases, were narrow (roughly 100 μm) so as to warrant the use of the nanoindentation technique. Similarly, for R2 samples, indentations were made only in regions of granular PyC.

[1] Modulus and hardness determinations for R1 (run1) were made at NΔNO® Instruments, using the Continuous Stiffness™ method; see reference [2].

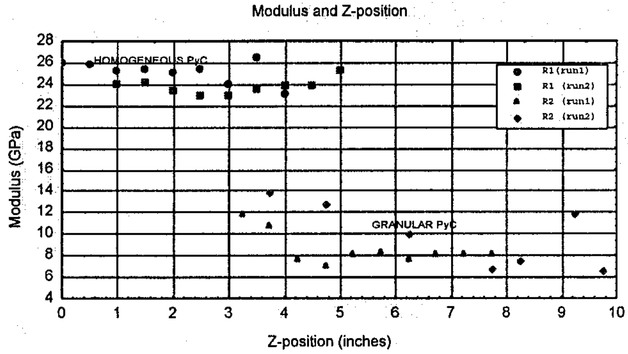
In general, the load and displacement curves associated with the modulus and hardness determinations showed that homogeneous PyC is more elastic than granular PyC. Plots of load and displacement, which are representative, are given in Figure 2.

Figure 2
Load and Displacement
Homogeneous and Granular PyC



The modulus and hardness results are plotted in Figures 3 and 4. The modulus data corresponding to homogeneous PyC are distributed about 24.4 GPa ± 0.8 GPa, and the modulus of granular PyC is seen to be about 8.8 GPa ± 1.9 GPa. For both types of PyC, the error has been set to the pooled standard deviation of each data set. Similarly, the hardness data for homogeneous PyC is scattered about 3.6 GPa ± 0.2 GPa; the hardness data for granular PyC is distributed about 0.9 GPa ± 0.2 GPa.

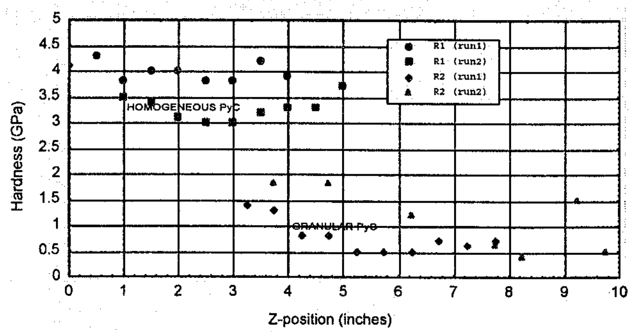
Figure 3
Modulus and Z-position



Discussion

The model of the deposition process that is believed to apply here is the droplet, or agglomerate, model [3]. In this model, the PyC coating is comprised of somewhat spherical agglomerates of carbon atoms, which were nucleated and grown in the gas phase, with molecular species deposited at the agglomerate interstitial sites.

Figure 4
Hardness and Z-position



It is postulated that the occurrence of porosity is influenced by the size distributions of the agglomerates; granular PyC is more porous than isotropic PyC, and it is believed that this results from granular PyC being constituted of larger agglomerates.

It is further hypothesized that the size distribution of the agglomerates is controlled by the residence time, which is influenced by gas flow rates and bed particle densities. Lastly, it is believed that the visual appearance of a sample, and the sample's density, modulus, and hardness are affected by porosity.

Conclusion

Two types of PyC can be produced in the CVD reactor. These two types of PyC can be distinguished qualitatively and quantitatively, and are connected with the position within the coater.

Acknowledgments

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